



BURN PIT TREATABILITY STUDIES SAMPLING AND ANALYSIS PLAN

ASCEND/FORMER SOLUTIA FACILITY
GREENWOOD, SOUTH CAROLINA

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August 2024

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Acronyms

Ascend	Ascend Performance Materials Operations, LLC
BP	Burn Pit
CSIA	compound-specific isotope analysis
DHEC	South Carolina Department of Health and Environmental Control
DO	dissolved oxygen
EPA	U.S. Environmental Protection Agency
Eurofins	Eurofins Environment Testing, LLC
Montrose	Montrose Environmental Solutions, Inc.
ORP	oxidation-reduction potential
PDB	passive diffusion bag
SAP	Sampling and Analysis Plan
SC	specific conductivity
SOP	standard operating procedure
VOC	volatile organic compound

1 INTRODUCTION

In June 2024, Montrose Environmental Solutions, Inc. (“Montrose”) on behalf of Ascend Performance Materials Operations, LLC (“Ascend”) submitted a Treatability Studies Work Plan (herein referred to as “the Work Plan”) (Montrose, 2024) for the former Burn Pit (“BP”) Area groundwater condition at Ascend’s facility located in Greenwood, South Carolina (herein referred to as “the Site”). The Work Plan presented a scope of work to evaluate monitored natural attenuation and *in situ* treatment for volatile organic compounds (“VOCs”) in groundwater, historically addressed through groundwater extraction. The prescribed scope of work involves sampling direct push sampling (groundwater and saturated solid matrix), monitoring well sampling, surface water and pore water monitoring, and an *in situ* microcosm study. This Sampling and Analysis Plan (“SAP”) specifies the procedural and analytical requirements for executing these work elements.

The remainder of the Work Plan is organized as follows:

- Section 2 describes the sample collection procedures and methods as well as the analytical testing regimen that will be followed in execution of the work scope;
- Section 3 provides supporting protocols for sample collection including sample custody, equipment decontamination, and quality assurance/quality control; and
- Section 4 is a list of references cited.

2 FIELD PROCEDURES

2.1 Overview

As previously noted, the work scope outlined in the Work Plan includes sampling of various environmental media (groundwater, surface water, pore water, and saturated aquifer matrix) and an *in situ* microcosm study. Field samples will be collected per the procedures outlined in this SAP. The field sampling procedures presented in this section are generally consistent with standard operation procedures (“SOPs”) developed by the U.S. Environmental Protection Agency (“EPA”) Region 4 or with vendor specifications. Relevant SOPs are presented in the Appendix A.

2.2 Monitoring Well Sampling

Monitoring well sampling prescribed by the Work Plan includes the first routine semi-annual sampling event of the 2024 monitoring regimen described in September 2023 Semi-Annual Water Quality Monitoring Report (HRP, 2023) and targeted treatability study testing. The routine sampling focuses on characterization of volatile organic compounds (“VOCs”; by Method 8260D), while the feasibility study analytical testing regimen is comprised of alkalinity (by SM 2320B-2011); total organic carbon (by Method 9060A); nitrate, sulfate, and nitrite (by Method 9056A); sulfide (by Method 9034); ferrous iron (by SM 3500-Fe B-2011); dissolved gases ethene, ethane, and methane (by Method RSK-175); and compound-specific isotope analysis (“CSIA”). A summary of the monitoring well sampling and analysis regimen is provided in Table 2-1. Characterization of VOCs and MNA parameters will be completed by Eurofins Environment Testing, LLC (“Eurofins”) (in Atlanta, Georgia), and CSIA will be completed by the environmental isotope laboratory at the University of Waterloo (in Ontario, Canada).

Previous routine monitoring has involved sampling with passive diffusion bags (“PDBs”) and sampling from a spigot, as opposed to conventional purge-and-sample methods that allow for collection accurate measurement and of the treatability study parameters. Where PDBs are deployed or where samples were previously collected via spigot, VOC samples will be collected in the same manner to remain consistent with previous routine monitoring events. Samples for treatability studies analyses will be collected using a peristaltic or downhole pump following low-flow purging techniques in accordance with EPA SOPs (EPA, 2023a). Standard field parameters temperature, pH, oxidation-reduction potential (“ORP”), dissolved oxygen (“DO”), specific conductivity (“SC”), and turbidity will be measured using a water quality meter and recorded during purging. Groundwater samples will be collected when pH and specific conductivity stabilize, defined as follows: pH \pm 0.1 units and conductivity \pm 5% for three consecutive measurements (minimum of 30 minutes of purge time).

2.3 Direct Push Sampling

Sample collection for the pH buffering assessment and abiotic panel testing involves direct push technology. The pH buffer assessment will be conducted by Terra Systems, Inc. and requires paired

samples of the groundwater and solid aquifer matrix. Abiotic panel testing will be completed by Microbial Insights, Inc. and requires a sample of the solid aquifer matrix.

Continuous soil borings will be completed and sampled according to EPA SOPs for Macro-Core® (i.e., piston) system (EPA, 2023b). The piston sampler is advanced incrementally to the target depth with dual tube technology and utilizes an internal direct push extension attached to a drive tip to displace the aquifer matrix, eliminating the potential for the aquifer matrix to enter the piston sampler prior to achieving the desired sampling depth. Once the target depth is achieved, the inner extension and drive tip are removed, and the piston sampler is advanced to collect a sample of the aquifer matrix. The outer casing and piston sampler are removed from the borehole and the sample is retrieved. Groundwater is collected by retooling the rig with a discrete (split-screen) sampler; this sampling device is simply pushed or driven to the desired depth and opened, whereupon the groundwater sample is collected and retrieved (purged) via a peristaltic pump.

2.4 Surface Water and Pore Water Monitoring

The Work Plan prescribes monitoring of groundwater-surface water interaction via pore water and surface water testing within North Creek and South Creek.

Surface water samples will be collected according to the SOPs developed by EPA (EPA, 2023c) using a 1-liter amber glass container. The amber will be carefully submerged 4-6 inches (where possible) below the water surface with the cap left secure on the bottle. The sampler will face upstream (into the current) when collecting the sample to prevent/limit any re-suspended sediments from entering the amber. Once the neck of the amber is submerged, the cap will be removed to fill the bottle, re-secured, and its contents transferred to the appropriate sample containers. Care will be taken not to flush any preservative out of sample containers during the fill process. The surface water analytical testing regimen will consist of VOCs by Method 8260D. Montrose field technicians will collect standard water quality parameters (i.e., temperature, ORP, DO, SC, turbidity) and document visual observations of surface water conditions during sample collection, including presence/absence of a sheen, odor, and aquatic wildlife.

Sediment interstitial (pore) water samples will be collected co-located to surface water samples using PushPoint™ samplers from the top 6 inches of the sediment column, in accordance with EPA SOPs (EPA, 2023d). Pore water samples will also be characterized for VOCs by Method 8260D. Montrose field technicians will collect standard water quality parameters during the purging process.

2.5 In Situ Microcosm Study

Montrose is conducting an *in situ* microcosm study to evaluate the capacity of the aquifer to support a biological-based treatment. The study involves deploying Bio-Trap® samplers from Microbial Insights in select well points, to provide microbial, chemical, and geochemical lines of evidence of biodegradation

potential. The Bio-Traps utilize a passive sampling approach where the sampling unit remains suspended in the well screened interval for at least 30 days prior to retrieval. The Bio-Trap units will be sampled in accordance with vendor specifications and procedures.

3 SUPPORTING PROTOCOLS

3.1 Sample Handling and Storage

Environmental samples collected for laboratory analysis will be placed in containers provided by the analytical laboratory. Disposable gloves will be worn during sampling and discarded after each sample is collected to minimize the potential for cross-contamination. Field samples will be labeled with the Julian date and a unique identification number indicating environmental media (groundwater, surface water, pore water) and location/site where the sample was collected. Samples will be preserved at 4°C until prepared for shipment to the analytical laboratory.

3.2 Sample Shipping and Custody

Sample labeling, shipping, and chain of custody will be performed in accordance with EPA procedures (EPA, 2023e; EPA, 2024). Samples will be packed for shipping in coolers and shipped on wet ice to maintain a 4°C condition. Chain of custody records will be sealed in a waterproof plastic bag and taped inside the cooler lids. After packing, two custody seals will be signed, dated and affixed from the cooler lid to the cooler body to ensure that any tampering with the cooler contents would be immediately evident to sample custodians on the receiving end of the shipment. The cooler lids will be sealed with strapping tape aligned overtop of the custody seals, and the coolers will be shipped overnight to the analytical laboratory.

3.3 Logbooks

Sampling personnel will use a bound field logbook with moisture-resistant pages to record pertinent sampling information with an indelible pen or marker. Logbooks will be maintained according to the SOP developed by the EPA (EPA, 2021). Each logbook will identify the project name, project number, and geographic location of the site. Daily field activities and sampling information will be entered in the log book on serially-numbered pages. At the end of each day's entries, sample-collection personnel will sign and date the entry. Corrections will be made to entries by initialed and dated line-out deletions. A diagonal line will be drawn across the remaining blank space of the last page of each day's entry.

3.4 Equipment Calibration

Water quality meters will be calibrated at a daily frequency according to manufacturer specifications to ensure collection of accurate measurements. Field personnel will maintain calibration records in the logbook.

3.5 Equipment Decontamination

Field equipment used to collect samples for laboratory analysis will require decontamination between samples to assure sample integrity, except in instances where the equipment is dedicated to collecting

a single sample. Decontamination will be performed according to EPA guidance (EPA, 2020). In general, sampling equipment will be decontaminated according to the following procedure:

1. Rinse with tap water and scrub with brush to remove particulate matter and surface films.
2. Wash with phosphate-free detergent (e.g., Liquinox).
3. Rinse with organic-free water.
4. Allow to air dry.
5. Wrap dry equipment in aluminum foil or bag in clean plastic.

Field equipment requiring decontamination are described below categorized by environmental media.

Groundwater. Water level meters will be decontaminated between each well. Tubing will be dedicated to a single well and therefore not require decontamination.

Direct Push Sampling. Samples of the solid aquifer matrix will be collected in dedicated acetate liner. Discrete samplers used for groundwater sampling will be decontaminated between boreholes.

Surface Water. A clean amber will be used to collect surface water at each sampling location; thus, no decontamination is required for surface water sampling.

Pore Water. A fresh PushPoint sampler and dedicated tubing will be used to collect pore water at each sampling location; thus, no decontamination is required for pore water sampling.

In Situ Microcosm Study. Bio-Traps are single-use and dedicated to an individual well. Decontamination is not necessary.

3.6 Quality Assurance/Quality Control

Quality Assurance/quality control samples will include field duplicates, that is co-located samples for assessing the precision of the sampling process and integrity of the analytical result. Field duplicates will be tested at a frequency of 5% or more frequently if required by the analytical laboratory (but no more than 10%), in accordance with EPA guidance (EPA, 2023f).

3.7 Investigation-Derived Waste Handling

Investigation-derived wastes include decontamination fluids and purge water. These waste streams will be placed through the groundwater extraction system, which conveys wastewater through underground piping to the Ascend facility wastewater system prior to discharge to the Greenwood Metropolitan Sewer District Publicly Owned Treatment Works. The groundwater extraction system process is operated under an Industrial User Permit maintained by Ascend.

4 REFERENCES

- EPA (2020). Field Equipment Cleaning and Decontamination, Laboratory Services and Applied Science Division LSASDPROC-205-R4. Dated June 2020.
- EPA (2021). Procedure for Documenting Environmental Information in Logbooks, Laboratory Services and Applied Science Division LSASDPROC-1002-R1. Dated May 2021.
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- EPA (2023c). Surface Water Sampling, Laboratory Services and Applied Science Division LSASDPROC-201-R6. Dated April 2023.
- EPA (2023d). Pore Water Sampling, Laboratory Services and Applied Science Division LSASDPROC-513-R5. Dated April 2023.
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- HRP Associates, Inc. (2023). September 2023 Semi-Annual Water Quality Monitoring Report. December.
- Montrose Environmental Solutions, Inc. (2024). Burn Pit Treatability Studies Work Plan – Revision 1. Ascend/Former Solutia Facility, Greenwood, South Carolina. April.

Figures

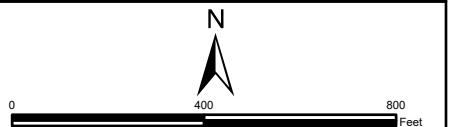


Well Location Map

Ascend/Former Solutia Site
Greenwood, SC

Legend

- Extraction Well
- Monitoring Well
- Creek
- Roads



DRN: XXX	APR: XXX
DATE: 08.14.2024	PRN: PROJ-042952

Figure No. 2-1

Document Path: C:\Users\jlabstott\Documents\Ascend_SCI\GIS\Basel\Basel.aprx

Tables

Table 2-1. Monitoring Well Testing Regimen

Well	COCs	Indicator Parameters*				MNA Parameters*								Compound-Specific Isotope Analysis (CSIA)*							
	VOCs	Temperature	pH	Oxidation-Reduction Potential	Dissolved Oxygen	Nitrate	Nitrite	Sulfate	Sulfide	Ferrous Iron	Alkalinity	Ethene & Ethane	Total Organic Carbon	δ13C on CxHyClz, PCE	δ37Cl on CxHyClz, PCE	δ13C on CxHyClz, TCE	δ37Cl on CxHyClz, TCE	δ13C on CxHyClz, 111-TCA	δ37Cl on CxHyClz, 111-TCA	δ13C on CxHyClz, 11-DCE	δ37Cl on CxHyClz, 11-DCE
	8260D	Water Quality Meter				9056A	9056A	9056A	9034	3500-Fe B-2011	2320B-2011	RSK-175	9060A	Isotope Lab							
<i>Burn Pit Area</i>																					
OW-055	LF	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X				
OW-10D	PDB	X	X	X	X	X	X	X	X	X	X	X	X								
OW-24S	LF	X	X	X	X																
OW-25S	PDB	X	X	X	X	X	X	X	X	X	X	X	X								
OW-26S	PDB	X	X	X	X	X	X	X	X	X	X	X	X								
OW-27D	PDB	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X				
OW-28D	PDB	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X				
OW-33S	PDB																				
OW-34S	PDB																				
OW-35D	PDB	X	X	X	X	X	X	X	X	X	X	X	X								
OW-36S	PDB	X	X	X	X	X	X	X	X	X	X	X	X								
OW-37D	PDB	X	X	X	X	X	X	X	X	X	X	X	X								
EW-04D	Spigot	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X				
EW-32S	Spigot	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X				
PZ-04S	PDB	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X				
PZ-29S	PDB	X	X	X	X	X	X	X	X	X	X	X	X								
PZ-31S	PDB																				
<i>Construction Debris Site</i>																					
EW-01C	Spigot	X	X	X	X	X	X	X	X	X	X	X	X					X	X	X	X
EW-02A	Spigot	X	X	X	X	X	X	X	X	X	X	X	X					X	X	X	X
EW-03B	Spigot	X	X	X	X	X	X	X	X	X	X	X	X					X	X	X	X
OW-01	PDB	X	X	X	X	X	X	X	X	X	X	X	X								
OW-02D	PDB	X	X	X	X	X	X	X	X	X	X	X	X					X	X	X	X
OW-13S	PDB																				
OW-13D	PDB	X	X	X	X	X	X	X	X	X	X	X	X					X	X	X	X
OW-15D	LF	X	X	X	X																
OW-16	PDB	X	X	X	X	X	X	X	X	X	X	X	X					X	X	X	X
OW-17	PDB	X	X	X	X	X	X	X	X	X	X	X	X								
OW-18D	LF	X	X	X	X																
OW-19D	LF	X	X	X	X																
OW-38	PDB																				
OW-39	PDB																				

Notes:
 * All indicator parameters and samples tested for MNA parameters and CSIA collected via low-flow purging
 PDB VOCs sampled via PDB
 LF VOCs sampled via low-flow purging
 Spigot VOCs sampled via spigot

Appendix A – Standard Operating Procedures

Region 4 U.S. Environmental Protection Agency Laboratory Services & Applied Science Division Athens, Georgia	
Operating Procedure	
Title: Groundwater Sampling	ID: LSASDPROC-301-R6
Issuing Authority: Field Services Branch Supervisor	
Effective Date: April 22, 2023	Review Due Date: November 23, 2025
Method Reference: N/A	SOP Author: Doug Peters

Purpose

The purpose of this procedure is to document both general and specific procedures, methods, and considerations to be used and observed when performing groundwater sampling for field screening and laboratory analysis. This Standard Operating Procedure (SOP) is specific to the Field Services Branch (FSB) to maintain conformance to technical and quality system requirements. While this SOP may be informative for other businesses, it is not intended for and may not be directly applicable to operations in other organizations. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

Scope/Application

This document describes specific methods to be used by field personnel when collecting and handling groundwater samples in the field. On the occasion that Laboratory Services and Applied Science Division (LSASD) field personnel determine that any of the procedures described in this SOP are inappropriate, inadequate, or impractical and that another procedure may be used to obtain a groundwater sample, the variant procedure will be documented in the field logbook, in accordance with the LSASD Operating Procedure for Logbooks (LSASDPROC-1002), along with a description of the circumstances requiring its use.

Note: LSASD is currently migrating to a paperless organization. As a result, this SOP will allow for the use of electronic logbooks, checklists, signatures, SOPs, and forms as they are developed, which will also be housed on the Local Area Network (LAN) and traceable to each project.

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1.0 General Information

1.1 Documentation/Verification

This procedure was prepared by persons deemed technically competent by LSASD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.2 General Precautions

1.2.1 Safety

Proper safety precautions must be observed when collecting groundwater samples. Refer to the LSASD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. The reader should address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.2.2 Procedural Precautions

The following precautions should be considered when collecting groundwater samples.

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Always sample from the anticipated cleanest, i.e., least contaminated location, to the most contaminated location, if possible. This minimizes the opportunity for cross-contamination to occur during sampling.
- Collected samples must remain in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous

materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.

- Documentation of field sampling is done legibly, completely, and neatly in a bound logbook.

2.0 Groundwater Purging and Sampling

2.1 Overview of Purging and Sampling Methods

Purging is the process of removing stagnant water from a well, immediately prior to sampling, causing its replacement by groundwater from the adjacent formation that is representative of aquifer conditions. Sampling is the process of obtaining, containerizing, and preserving (when required) a ground water sample after the purging process is complete. There are several approaches to well purging and sampling that may be appropriate in various circumstances or for various combinations of available equipment. They are briefly summarized below and in *Table 1, Purge and Sample Strategies with Equipment Considerations*.

The static water level should always be measured first, prior to inserting any tubing or pumping equipment into the well casing and after the well has been uncapped long enough for the water level to adjust to atmospheric pressure. The water level should also be monitored and documented during pumping, especially for the low-flow method. A clean, decontaminated water level probe should always be used and should be cleaned and dried off in the field after use in a well, prior to being inserted in a different well.

The **Multiple-Volume Purge** method involves removing a minimum of three well volumes of water from the top of the water column and then sampling when the well has achieved stability of water quality parameters and adequately low turbidity. This is a traditional method and consistent results are generally obtained with samplers of varying skill. A drawback is that large volumes of contaminated purge water may be produced for large diameter or deep wells.

The **Low-Flow** method involves purging the well at a relatively low flow rate that minimizes drawdown, with the pump or tubing inlet located within the screened interval of the well. The well is sampled when water quality parameters are stable, adequately low turbidity is achieved, and the water level has achieved a stable drawdown (an unchanging water level). This method is often faster than Multiple-Volume Purge and generates less purge water. The method requires more skill and judgment on the part of the samplers.

The **Multiple-Volume Purge** method and the **Low-Flow** method can be considered equivalent for conventionally screened and filter-packed wells in that they both sample a flow-weighted average of water entering the well during pumping. However, other variables can result in differences between results with the two methods. In repeat sampling events, the sampling design should not change from one method to the other without appropriate cause. The transition should be noted in the report.

Minimum-Purge and **No-Purge** methods are based on the assumption that water within the screened interval of the well is at equilibrium with the water in the surrounding aquifer.

This assumption should be carefully considered in the use of these methods and various cautions are discussed in the sections below. The **Minimal-Purge** and **No-purge** methods are most useful for long-term monitoring and are generally inappropriate for the early stages of investigation. In some cases, the methods might be used to gather screening-level data from wells that are too large to practically purge or have other sampling complications.

Table 1 Purge and Sample Strategies with Equipment Considerations

Purging Strategy	Purge Equipment	Sample Equipment	Comments
Multi-Volume Purge	-----	-----	Overall Method Comments- Advantages: Consistent results can be achieved with minimal skill level required. Common, simple equipment can be used. Disadvantages: Can result in large volumes of purge water/ more IDW. Can take extended periods of time with large diameter wells or long water columns.
In this traditional method, 3-5 well volumes of water are removed from the top of the water column while verifying the stability of water quality parameters. Following the well purge, the well is sampled from the top of the water column.	Bailer	Bailer	Bailers are rarely used for purging due to the effort required, the difficulty of lowering turbidity adequately, and the possibility of aerating the upper water column.
	Electric Submersible Pump	Bailer	Common multiple-volume setup when depth to water exceeds 25 feet. Abbreviated pump decontamination procedure can be used between wells.
	Electric Submersible Pump	Electric Submersible Pump	Requires full pump decontamination and new tubing at each well. In most cases the pump would be deployed to the screened interval instead to perform Low-Flow sampling.
	Peristaltic Pump	Peristaltic Pump	Common, multi-volume setup when depth to water is less than 25 feet. Special sampling techniques are required for the collection of SVOCs and VOCs.
Low-Flow methods	-----	-----	Overall Method Comments- Advantages: Lower volumes of purge water. May be faster, especially with longer water columns. Disadvantages: Requires greater skill for consistent results. Higher tubing costs than multi-volume method.
The pump or tubing inlet is placed within the screened interval and the well is purged to stable water quality parameters while maintaining stable drawdown of the water level.	Electric Submersible Pump	Electric Submersible Pump	Commonly used when depth to water exceeds 25 feet. Pump is cleaned to sample equipment standards prior to sampling each well and new or dedicated tubing used for each well. Concerns have been raised concerning VOC loss from agitation in the turbine section or from sample heating.
	Peristaltic Pump	Peristaltic Pump	Commonly used where depth to water is less than 25 feet. Special sampling techniques required for the collection of SVOCs and VOCs. Concerns have been raised concerning VOC loss from vacuum created in sample tubing.
	Bladder Pump	Bladder Pump	Least danger of VOC loss as entire sample train is under positive pressure and little sample heating occurs. Difficult to remove large volumes of water in reasonable time. Mild surging effect may keep turbidity elevated in sensitive wells.
Minimum-Purge, No-Purge Methods	-----	-----	Overall Method Comments- Advantages: Very little or no waste water. Well suited to repeat sampling events. Likely faster with lower costs. Disadvantages: Not directly equivalent to other methods. Vertical stratification or vertical flow conditions in the screened interval can result in deceptive or non-intuitive analytical results.
Predicated on the assumption that aquifer flow through the well maintains the water in the screened interval in a state equivalent to that in the aquifer. This assumption should be proven or the data qualified. Sampling is conducted with little or no purge, or by equilibrating a sampler in screened interval.	Pumps, various	Pumps, various	In the minimum-purge method, the internal volume of the sample tubing and pump is calculated. One volume of the pump and tubing is purged to flush the equipment and the well is then sampled.
	na	Passive Diffusion Bags	In most common form, a sealed water-filled polyethylene bag is allowed to equilibrate in the water column. Suitable primarily for VOCs. Generally require 2 week minimum in-situ residence time.
	na	Hydrasleeves	Collect a fixed volume of water from a specific interval. Requires duplicate samplers or redeployment for larger volumes. Sorbtion issues may bias results.
	na	Snap sampler	Deploys a sample container in the sampling interval where it is allowed to equilibrate (commonly for two weeks) before being sealed insitu by the sampler mechanism and retrieved. Limited to specific containers.

The **Minimum-Purge** and **No-Purge** methods collect water in the vicinity of the sampling device under near-static conditions and are not equivalent to the **Multiple- Volume Purge** and **Low-Flow** methods. Stratification of horizontal flow or vertical flow conditions within the well can result in non-intuitive and deceptive results. A comparison study should be conducted before transitioning a sampling program to the minimal-purge or no-purge methods.

2.2 Purging

Wells are purged to eliminate stagnant water residing in the casing and/or screen that has undergone geochemical changes or loss of VOCs. At the conclusion of purging, the desired flow-weighted average of water entering the well under pumping conditions will be available for sampling. Turbidity is often elevated during purging by the disturbance of formation materials at the borehole walls. As many contaminants (metals and many organics) will sorb to the formation particles, a sample including these particles will not represent the dissolved concentrations of the contaminants. Thus, a secondary goal of purging is to reduce the turbidity to the point that the sample will be representative of the dissolved contaminant concentration.

In order to determine when a well has been adequately purged, field investigators should monitor, at a minimum, the pH, specific conductance and turbidity of the groundwater removed and the volume of water removed during purging. The measurements should be recorded in a purge table in the field logbook that includes the start time of purging, the parameter measurements at intervals during purging, estimated pumped volumes, depths to water (especially for **Low-Flow** sampling), and any notes of unusual conditions. A typical purge table used for Low-Flow sampling is reproduced below in **Figure 1**.

2.0.1 Parameter Stabilization Criteria

With respect to the ground water chemistry, an adequate purge is achieved when the pH and specific conductance of the ground water have stabilized and the turbidity has either stabilized or is below 10 Nephelometric Turbidity Units (NTUs) (twice the Secondary Drinking Water Standard of 5 NTUs).

Stabilization occurs when, for at least three consecutive measurements, the pH remains constant within 0.1 Standard Unit (SU) and specific conductance varies no more than 5 percent. Other parameters, such as dissolved oxygen (DO) or oxidation-reduction potential (ORP), may also be used as a purge adequacy parameter. Normal stability goals for DO are 0.2 mg/L or 10% change in saturation, whichever is greater. DO and ORP measurements must be conducted using either a flow-through cell or an over-topping cell to minimize oxygenation of the sample during measurement. A reasonable ORP stability goal is a range of 20 mV, although ORP is rarely at equilibrium in environmental media and often will not demonstrate enough stability to be used as a purge stabilization parameter. Determining the frequency of measurements has generally been left to 'Best Professional Judgement'. Measurements recorded at frequent intervals with low flow rates can falsely indicate stability of parameters. Several measurements should be made early in the well purge to establish the direction and magnitude of trends, which can then inform the stability decision. Stability

parameters should either be not trending, or approaching an asymptote, when a stability determination is made. As a matter of practice, parameter measurements are generally made at 5-10 minute intervals.

Sometimes the measurement interval may be greater if turbidity is lessening very slowly.

Because the measured groundwater temperature during purging is subject to changes related to surface ambient conditions, pumping rates and pump temperature, its usefulness is subject to question for the purpose of determining parameter stability. As such, it has been removed from LSASD's list of parameters used for stability determination. Even though temperature is not used to determine stability, it is still advisable to record the temperature of purge water as it is often used in the interpretation of other parameters.

Information on conducting the stability parameter measurements is available in the LSASD Operating Procedures for Field pH Measurement (LSASDPROC-100), Field Specific Conductance Measurement (LSASDPROC-101), Field Temperature Measurement (LSASDPROC-102), Field Turbidity Measurement (LSASDPROC-103), Field Measurement of Dissolved Oxygen (LSASDPROC-106) and Field Measurement of Oxidation-Reduction Potential (LSASDPROC-113).

Figure 1 Purge Table Showing Field Measurements

Continuation of sample GW 65-0713

TIME	pH (S.U.s)	Spec. Cond. (us/cm)	Temp. (Deg. C)	D.O. (mg/L)	D.O. (% sat.)	ORP (mV)	Turbidity (NTUs)	Water Level (Ft.)	Purge Vol. (gallons)
0930									Pump On
0935	5.71	1065	19.6	0.77	8.7	43.9	2.10	24.83	1/4
1004	5.64	988	20.0	0.36	3.9	222.5	17.8	25.24	2
1026	5.63	959	20.5	0.25	2.7	98	9.95	25.18	3 1/2
1038	5.62	950	20.5	0.21	2.4	75	9.85	25.18	4
1046	5.61	946	20.8	0.21	2.4	73	6.07	25.18	4 1/2
1047									Sample Collection Time

INITIAL
23.33ft

2.3 Multiple-Volume Purge

In the traditional Multiple-Volume Purge method, water is removed from the top of the water column, causing water to enter the screen and flush stagnant casing water upward to be subsequently removed. In recognition of the mixing of fresh and stagnant water in the casing section, a minimum of three well volumes is removed, at which time purging can be terminated upon parameter stabilization. Wells can be assumed to be adequately purged when five well volumes have been removed, although further purging may be conducted to meet specific goals, such as further reduction of turbidity.

2.3.1 Purge Volume Determination

Prior to initiating the purge, the amount of water standing in the water column (water inside the well riser and screen) should be calculated. The diameter of the well is determined and the water level, followed by total depth of the well, are measured and recorded prior to inserting anything into the well. The water level is subtracted from the total depth, providing the length of the water column. Specific methodology for obtaining these measurements is found in LSASD Operating Procedure for Groundwater Level and Well Depth Measurement (LSASDPROC-105).

Once this information is obtained, the volume of water to be purged can be determined using one of several methods. The well volume can be calculated using the equation:

$V = \pi r^2 h \times 7.4805$ gal / cu foot Where:

h = length of water column in feet, r = radius of well casing in feet V =
one well volume in gallons

Or, more conveniently:

$$V = 0.041 d^2 h$$

Where:

h = length of water column in feet d =
diameter of well in inches
V = one well volume in gallons

Multiplying the casing volume, V, by 3 provides the usual 3-volume purge in gallons. Alternatively, the volume of standing water in the well and the volume of three water columns may be determined using a casing volume per foot factor for the appropriate diameter well, such as listed in **Table 2, Well Casing Diameter Volume Factors**. The water column length is multiplied by the appropriate factor from **Table 2** to determine the single well volume, three well volumes, or five well volumes for the well in question. Other acceptable methods include the use of nomographs or other equations or formulae.

An adequate purge is normally achieved when three to five well volumes have been removed. The field notes should reflect the single well volume calculations or determinations, according to one of the above methods, and a reference to the appropriate multiplication of that volume, i.e., a minimum three well volumes, clearly identified as an initial purge volume goal.

TABLE 2 WELL CASING DIAMETER VOLUME FACTORS

		Reference	Minimum purge	Maximum purge*
		1 Well Volume (gallons/ft)	3 Well Volumes (gallons/ft)	5 Well Volumes (gallons/ft)
Well Casing Diameter (in)	0.5	0.01	0.03	0.05
	0.75	0.02	0.07	0.11
	1	0.04	0.12	0.20
	2	0.16	0.49	0.82
	3	0.37	1.1	1.8
	4	0.65	2.0	3.3
	5	1.0	3.1	5.1
	6	1.5	4.4	7.3
	7	2.0	6.0	10.0
	8	2.6	7.8	13.1
	9	3.3	9.9	16.5
	10	4.1	12.2	20.4
	11	4.9	14.8	24.7
	12	5.9	17.6	29.4
	13	6.9	20.7	34.5
	14	8.0	24.0	40.0
	15	9.2	27.5	45.9
	16	10.4	31.3	52.2
	18	13.2	39.7	66.1
24	23.5	70.5	118	
36	52.9	159	264	
48	94.0	282	470	

* See text for discussion on terminating purge at five well volumes

2.3.2 Pumping Conditions

The pump or tubing inlet should be located at the top of the water column. If the pump is placed deep into the water column, the water above the pump may not be removed, and the subsequent samples, particularly if collected with a bailer, may not be representative of the aquifer conditions. If the recovery rate of the well is faster than the pump rate and no observable draw down occurs, the pump should be raised until the intake is as close as possible to the top of the water column for the duration of purging. If the pump rate exceeds the recovery rate of the well, the pump or tubing will have to be lowered to accommodate the drawdown.

2.3.3 Stability of Chemical Parameters

In the **Multiple-Volume Purge** method, a stability determination may be made after three well volumes have been removed. If the chemical parameters have not stabilized according to the above criteria, additional well volumes (up to a total of five well volumes) should be removed. If the parameters have not stabilized after the removal of five well volumes, it is at the discretion of the project leader whether or not to collect a sample or to continue purging. If, after five well volumes, pH and conductivity have stabilized and the turbidity is still decreasing and approaching an acceptable level, additional purging should be considered to obtain the best sample possible.

2.3.4 Sample Collection

There are several means by which sampling can proceed after adequate volume has been purged and water quality parameters have stabilized. If a submersible pump and tubing are of suitable material and cleanliness for sample collection, sampling can proceed immediately by directly filling bottles from the tubing outlet. Commonly with the multiple-volume purge method, the pump is set up and cleaned in a manner suitable only for purging. In these cases, the pump is stopped and removed from the well and sampling proceeds with a bailer per the procedure described in **Section 2.6.5**. The pump should have a check valve to prevent water in the pump tubing from discharging back into the well when the pump is stopped. If a peristaltic pump is used, sampling can proceed as described in **Section 2.6.1**.

2.4 Low-Flow Method

The **Low-Flow** method involves placing the pump or tubing inlet within the screened interval of the well (generally mid-screen, discussed in **Section 2.4.2**) and purging at a low enough rate to achieve stable drawdown and minimal depression of the water level (a total drawdown of one foot or less is generally preferred, if possible). The well is sampled with as little interruption in pumping and flow rate as is possible after field parameters are stable and low turbidity is achieved. In general, only water in the screened interval of the well is pumped and the stagnant water in the well casing above the screen is not removed. Wells can generally be sampled in less time with less purge volume than with the multi-volume purge method. More attention is required in the assessment of stability criteria than the multi-volume method.

2.4.1 Nomenclature

A variety of terminology has been used to describe this method by LSASD and others, including: 'low flow', 'low-flow/low-volume', 'tubing-in-screen method', 'low flow/minimal drawdown', and 'micropurge'. The current preferred LSASD terminology for this method is 'Low-Flow'. As the term 'micropurge' is sometimes used to refer to minimal-purge methods and has been trademarked by a vendor, the use of 'micropurge' to describe the **Low-Flow** method generally introduces ambiguity and confusion and thus the use of the term is discouraged.

2.4.2 Placement of Pump Tubing or Intake

The inlet of the pump tubing or intake of the submersible pump is placed in the approximate mid-portion of the screened interval of the well. In cases where the water column does not cover the entire screen length, the midpoint of submerged screen should be determined for intake placement. The pump or tubing can be placed by carefully lowering them to the bottom of the well and then withdrawing half of the screen length, plus the length of any sump sections at the bottom of the well. A measuring tape should be used at the top of casing to accurately determine the length the pump is raised from the bottom. The pump should then be tied off so that it stays at the desired depth. A drawback of this approach is that it may stir up sediment at the well bottom. An alternate approach is to lower the pump or tubing a measured distance to place it at mid-screen without touching the bottom of the well. Special care should be used in lowering pumps slowly, especially in the screened interval, to prevent elevating turbidity needlessly by the surging action of the pump. While it is often thought that particular aquifer zones can be targeted by specific pump or intake placement, for conventionally constructed screened and filter-packed monitoring wells the zone monitored is only weakly dependent on the intake placement (Varljen, Barcelona, Obereiner & Kaminski, 2006).

2.4.2.1.1 Conditions of Pumping

Prior to initiation of pumping, a properly decontaminated well sounder should be lowered into the well to measure the water level prior to and during the purging process. Ideally, there should be only a slight and stable drawdown of the water column after pumping begins. In some cases, because of low recharge rates, it may be necessary for the well to drawdown a considerable distance (10 ft or more in extreme cases) to maintain a minimal usable pumping rate for sampling (100-200 ml/min). Excessive pump rates and drawdown can result in increased turbidity, aeration of the sample if the screen is exposed, or pulling in of stagnant water from the column. Stable drawdown is an essential condition of the Low-Flow method. If the stable drawdown condition cannot be met, then one of the other methods should be employed.

2.4.2.1.2 Stability of Chemical Parameters

As with the **Multiple-Volume Purging** method described, it is important that all chemical parameters be stable prior to sampling. It is common for wells to require the removal of one or more screened-interval volumes (~2 gal for a 10 ft screen in a 2" dia. well) to achieve stability. Although it is possible for wells to achieve stability with lower purge volumes, the sampler should exercise caution in making an early stability determination.

2.4.2.1.3 Sample Collection

Low-Flow sampling is implemented using a pump and tubing suitable for sampling. After making the determination of parameter stability with stable drawdown, sampling can proceed immediately. Where submersible or bladder pumps are used, sampling can proceed by directly filling bottles from the tubing outlet. Where peristaltic pumps are used, sampling can proceed per the procedure described in Section 2.6.1.

2.5 Minimum-Purge and No-Purge Sampling

The **Minimum-Purge** and **No-Purge** sampling methods are employed when it is necessary to keep purge volumes to an absolute minimum, where it is desirable to reduce long-term monitoring costs, or where large wells or other limitations prevent well purging. The underlying assumption when employing these methods is that the water within the well screen is equilibrated with the groundwater in the associated formation. This assumption should be demonstrated prior to use of these methods or the results suitably qualified. These methods are generally impractical for LSASD to implement because of the common lack of hydrogeological information in early investigative phases and the necessity with some methods that the samplers be pre-deployed to allow equilibration.

Vertical flow conditions and stratification of the water column have also been known to result in deceptive and non-intuitive analytical results. The use of these methods in the early phases of investigation can easily result in misinterpretation of site conditions and plume boundaries.

Particular caution is in order in the use of these methods when any of the following conditions exist:

- Low hydraulic conductivity ($K < 10^{-5}$ cm/sec)
- Low groundwater surface gradients
- Fractured bedrock
- Wells with long screened intervals
- Wells screened in materials of varying hydraulic conductivities

If it is desired to transition a long-term monitoring program to **Minimum-Purge** or **No-Purge** sampling, a pilot study should be conducted where the **Minimum-Purge** or **No-Purge** sample results are compared to the conventional methods in use. Multiple samplers may be deployed in the screened interval to help establish appropriate monitoring intervals.

These methods are in common use and for the purposes of the LSASD quality system they can be considered standard, but unaccredited, procedures. Several **Minimum-Purge** or **No-Purge** procedures that might be employed are shown below. It is not the intention to recommend particular equipment or vendors, and other equipment that can accomplish the same goals may be suitable.

2.5.1 Minimum Purge Sampling

The pump or tubing inlet is deployed in the screened interval. A volume of water equal to the internal pump and tubing volume is pumped to flush the equipment. Sampling then proceeds immediately. While superficially similar to Low-Flow sampling, the results obtained in this method will be sensitive to the vertical pump or tubing inlet placement and are subject to the limitations described above.

2.5.2 Passive Diffusion Bags

The no-purge Passive Diffusion Bag (PDB) typically consists of a sealed low-density polyethylene (LDPE) bag containing deionized water. They are deployed in the screened interval of a well and allowed to equilibrate, commonly for two weeks, prior to retrieval and decanting of the water into sample containers. Many volatile organic compounds will reach equilibrium across the LDPE material, including BTEX compounds and many chlorinated solvents. Compounds showing poor equilibration across LDPE include acetone, MTBE, MIBK, and styrene. PDBs have been constructed of other materials for sampling other analytes, but the vast majority of PDB samplers are of the LDPE material. Various vendors and the Interstate Technology and Regulatory Council (ITRC) can provide additional information on these devices.

2.5.3 *HydraSleeves*TM

*HydraSleeves*TM are no-purge grab sampling devices consisting of a closed-bottom sleeve of low-density polyethylene with a reed valve at the top. They are deployed in a collapsed state to the desired interval and fill themselves through the reed valve when pulled upward through the sampling interval. The following is a summary of their operation:

Sampler placement – A reusable weight is attached to the bottom of the sampler or the sampler is clipped to a weighted line. The *HydraSleeve*TM is lowered on the weighted line and placed with the top of the sampler at the bottom of the desired sampling interval. In-situ water pressure keeps the reed valve closed, preventing water from entering the sampler. The well is allowed to return to equilibrium.

Sample collection - The reed valve opens to allow filling when the sampler is moved upward faster than 1 foot per second, either in one continuous upward pull or by cycling the sampler up and down to sample a shorter interval. There is no change in water level and only minimal agitation during collection.

Sample retrieval - When the flexible sleeve is full, the reed valve closes and the sampler can be recovered without entry of extraneous overlying fluids. Samples are removed by puncturing the sleeve with the pointed discharge tube and draining the contents into containers for sampling or field parameter measurements.

Because the *HydraSleeve*TM is retrieved before equilibration can occur and they are constructed of non-Teflon® materials, there may be issues with sorption of contaminants in the use of this sampler.

2.5.4 Snap Samplers

The Snap Sampler is a patented no-purge groundwater sampling device that employs a double-end-opening bottle with “Snap” sealing end caps. The dedicated, device is deployed at the desired position in the screened interval with up to six Snap Samplers and six individual sampling bottles. The device is allowed to equilibrate in the screened interval and retrieved between 3 and 14 days after deployment. Longer deployments are possible to accommodate sampling schedules.

To operate, Snap Samplers are loaded with Snap Sampler bottles and the "Snap" caps are set into an open position. Samplers are deployed downhole with an attachment/trigger line and left to equilibrate downhole. To collect samples, the Snap Sampler bottles seal under the water surface by pulling a mechanical trigger line, or using an electric or pneumatic trigger system. The trigger releases Teflon® "Snap Caps" that seal the double-ended bottles. The end caps are designed to seal the water sample within the bottles with no headspace vapor. After the closed vial is retrieved from the well, the bottles are prepared with standard septa screw caps and labeled for laboratory submittal.

The manufacturer of the Snap Sampler provides considerable additional information on the validation and use of the device.

2.6 Equipment Considerations and Use

Equipment choices are dictated by the purging and sampling method used, the depth to water, the quantity of water to be pumped, and quality considerations. The advantages and disadvantages of various commonly used pumps are discussed in the sections below and summarized in *Table 1, Purge and Sample Strategies with Equipment Considerations*. Additional information on the use of individual pumps is available in LSASD Operating Procedure for Pump Operation, SESDPROC-203.

2.6.1 Peristaltic Pumps

Peristaltic pumps are simple, inexpensive, and reliable equipment for purging and sampling where the limit of suction is not exceeded (approximately 25-28 vertical feet from the groundwater surface to the pump). When used for sampling, they should be equipped with new Teflon® tubing for each well. The flexible peristaltic pump-head tubing should also be changed between wells.

Samples for organic analyses cannot be exposed to the flexible peristaltic pump-head tubing, both due to the risk that the tubing would sorb contaminants and the propensity of this tubing to contribute organic compounds to the sample. Samples can be collected without contact with the pump-head tubing by the use of vacuum transfer caps for analyses requiring 1 liter glass containers and the use of the 'soda-straw' method for the filling of VOC vials.

The sample containers for the more turbidity-sensitive analyses are filled first, as removing tubing to fill the VOC vials (and to a lesser extent the glass bottles) may disturb the well and increase turbidity. The most appropriate order of sampling with a peristaltic pump is generally to fill poly containers for metals and classical analyses, followed by glass bottles for SVOCs and associated analyses, and finally to fill 40 ml VOC vials.

The following step-by-step procedure assumes that the pump has been set up per LSASD Operating Procedure for Pump Operation (LSASDPROC-203) and that containers for a typical full suite of analyses will be filled. The procedure is suitable for use with either **Multi-Volume Purge** or **Low-Flow** methods with minor differences in the collection of VOCs:

- 2.6.1.1 Deploy the lower end of the tubing to the desired point in the well. This would be the top-of-water for the multi-volume purge method or to the mid-screen for the **Low-Flow** method. Connect the well tubing to the flexible pump-head tubing and connect a short piece of tubing from the pump-head tubing to an over-topping cell (where probes can be inserted to measure parameters) in a measuring bucket for purge water collection.
- 2.6.1.2 Turn on the pump and establish a suitable pumping rate. For the multi-volume purge method, the rate will generally be a relatively fast rate that the well will sustain without elevating turbidity. For the **Low-Flow** method the pump rate is established at a slower rate to maintain a minimal and stable drawdown level.
- 2.6.1.3 Proceed with the measurement of water quality parameters and adjust the pump rate as needed to achieve low turbidity and stable drawdown.
- 2.6.1.4 When the well purge has been determined to be sufficient, fill containers for metals and classical analyses directly from the pump outlet. There is no need to interrupt pumping. The tubing should be held at the opening of the container and should not touch the container during filling. Protect caps from dust and debris during filling.
- 2.6.1.5 After filling the containers for metals and classical analyses stop the pump. Make sure that the tubing leading into the well is secured against movement during the following operations.
- 2.6.1.6 Create a crimp in the well tubing approximately one foot from the pump and grasp the crimped tubing in one hand. It is generally most effective to create a double 'Z' crimp.
- 2.6.1.7 Cut the sample tubing between the crimp and the pump. The tightly-held crimped tubing should keep water from running back into the well. In lieu of cutting the tubing, the well tubing can be disconnected from the pump and a short piece of tubing connected in its place.
- 2.6.1.8 Insert both free ends of the tubing into the ferrule-nut fittings of a pre-cleaned Teflon® transfer cap assembly and tighten the nuts. Attach the transfer cap assembly to the first glass container for semi-volatile analysis and securely tighten the threaded ring.
- 2.6.1.9 Turn the pump on. Very slowly release the 'Z' crimp in the sample tubing. As vacuum builds up in the sample container, water should begin to move up the sample tubing instead of back into the well. If after several minutes water has not begun moving up the tubing, check the tightness of fittings and the attachment of the cap to the bottle. Allowing water to rush back down the tubing from the 'Z' crimp can surge the well and elevate turbidity.

- 2.6.1.10 Fill the container to about halfway between the shoulder and the neck. Crimp the well tubing. Move the transfer cap to any additional bottles and repeat the filling process.
- 2.6.1.11 When finished filling bottles with the transfer cap, again crimp the tubing. Remove the well tubing from the transfer cap and reattach it to the pump. Slowly run the pump and release the crimp until water is approaching the flexible peristaltic tubing.
- 2.6.1.12 Make a kink or otherwise mark the tubing at the top of the casing in case the tubing needs to be reinserted (to the same depth) for additional sample volume. Slowly remove the tubing from the well and coil it in one hand in loose coils. With the top end of the tubing blocked, water is retained in the tubing as it is withdrawn, much as in a capped soda straw, hence the name for this method.
- 2.6.1.13 Remove the top from a 40 ml VOC vial and position the end of the sample tubing near the top of the vial. Reverse the pump direction and turn the speed knob to its slowest position. Turn on the pump and slowly increase speed until water slowly fills the vial. Fill the vial with a slow laminar flow that does not agitate the water in the vial or entrain bubbles. Continue to fill the vial until a convex meniscus forms on the top of the vial and turn off the pump.
- 2.6.1.14 Carefully screw the septum-lid to the vial and fasten firmly. Invert the vial and gently tap it on your knuckles or a hard surface to check for bubbles. Carefully add additional volume to the vial if necessary. Small bubbles are undesirable but may be unavoidable with some media, especially when using pre-preserved vials.
- 2.6.1.15 Repeat the filling process for additional vials. Avoid partially filling vials as the available water in the tubing is used. If more volume is required than that contained in the tubing, purge the remaining water from the tubing and reinsert the tubing in the well to the level marked previously. Run the pump to refill the tubing. If performing **Low-Flow** sampling, run additional volume through the pump to purge any water that may have been collected from the stagnant water column, and, preferably, to reestablish drawdown.

Note: During the filling of vials and re-insertion of tubing into the well to collect additional sample volume, the water level will begin to recover or completely recover, and pumping equilibrium will be lost while the pump is temporarily turned off. Therefore, to re-establish equilibrium and to clear out any water collected in the tube while re-inserting it, it may be desirable to let the pump run a few minutes at the established equilibrium purge speed before stopping it and removing the tubing to continue the soda straw method to fill more vials. Re-establishing equilibrium is preferable, if time permits. After re-establishing drawdown, if the pump is slowed slightly, allowing the water level to be rising in the casing, fresh formation water should be coming in from the screen, which should be acceptable for pulling water into the tubing for sample collection.

2.6.1.16 Fill additional vials as needed. Be sure that any water that has contacted the flexible peristaltic tubing is not pumped into a vial.

2.6.2 Submersible Centrifugal Pumps

Submersible centrifugal pumps are used in wells of 2” diameter and larger. They are especially useful where large volumes of water are to be removed or when the groundwater surface is too far below ground surface for peristaltic pumps to function. Commonly used pumps are the Grundfos® Redi-Flo2, the Geotech GeoSub, and the various ‘Monsoon’ style pumps. Other pumps are acceptable if constructed of suitable materials.

When used with the Multiple-Volume Purge method, the pump is generally used only to purge, with sampling performed with a bailer. In this use, the pump can be used with polyethylene or other tubing or hose that will not contribute contaminants to the well. The pump and tubing are decontaminated between wells per the relevant provisions of LSASD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205). When used in this application the pump should be equipped with a check valve to prevent water in the discharge tubing or hose from running back down into the well.

When used for Low-Flow purging and sampling (for contaminants other than PFAS compounds) the pump must be constructed of stainless steel and Teflon®. Pump cleaning at each well follows the more stringent procedures described in LSASD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) for this application. The sample tubing should be either new Teflon® tubing, or tubing dedicated to each well. Dedicated tubing would ideally be cleaned between uses, but tubing stored in the well casing between uses is acceptable, although caution should be exercised where very high concentrations of contaminants have been sampled in a well. --- Note: See Section 3.4 for PFAS sampling requirements.

2.6.3 Bladder Pumps

Bladder pumps use a source of compressed gas to compress and release a bladder straddled by check valves within the pump body. As the bladder is compressed, water is expelled out the upper check valve to the surface. When gas pressure is released, the bladder refills as well water enters the lower pump inlet. A control unit is used to control the pressure and timing of the bladder inflation gas flow.

Bladder pumps are capable of pumping from moderate depths to water, but are not capable of high flow rates. As they operate cyclically, the well is surged slightly on each cycle and it may be difficult to lower turbidity in sensitive or poorly developed wells. As the entire sample train is under positive pressure and the pumps develop little heat, they are ideal for sampling VOCs.

Prior to sampling and between each well the pumps are cleaned internally and externally per the provisions of LSASD Operating Procedure for Field Decontamination

(SESDPROC-205) and a new Teflon® bladder installed. New (or dedicated) Teflon® sample tubing is used at each well, although polyethylene tubing can be used for the compressed gas drive line and cleaned between each well.

2.6.4 Inertial Pumps

Inertial pumps consist of a check valve which is affixed to the lower end of semi-rigid tubing. The tubing and valve are cycled up and down, allowing water to alternately be drawn into the check valve inlet and then pulled up towards the surface. Two commonly used inertial pumps are the Waterra® pump for wells larger than 1" and the Geoprobe® Tubing Check Valve for small diameter wells. The primary use of these pumps is in well development where their near-immunity to silt is an advantage. Inertial pumps should not be used for the final well purge or for sampling as there is a low likelihood of reducing turbidity to appropriate levels and they have the potential to strip volatiles from the water column through agitation.

To set up the pump, the check valve is screwed onto the discharge tubing where it will cut its own threads. In the case of the Waterra® pump, a surge block can also be pressed onto the check valve. The pump is lowered into the well to the screened interval and rapidly cycled up and down a distance of 3" -12". The stroke length and speed are adjusted for pumping effect. Electric actuators can be used to reduce the effort involved. The pump should be moved to different levels in the screen to surge the entire screen.

The pump can occasionally be lowered to the bottom of the well to vacuum out fine sand and silt. Any sediment that clogs the valve is usually quickly rinsed out by the pump cycling and if the clog remains the pump is easily retrieved and redeployed.

The surging activity is usually continued until turbidity is lowered to a measurable range and is not easily lowered further. Further development or purging is then conducted with other pumps. --- Note: Also see Sections 2.8.2 and 3.15 for more on well development.

2.6.5 Bailers

Bailers are a common means of sampling when the Multiple-Volume Purge method is used. They are occasionally used for purging wells with low purge volumes when other equipment is not available or has failed. As bailers surge the well on each withdrawal, it is very difficult to lower turbidity adequately during a well purge, and when used for sampling they can elevate turbidity in a well before all sample volume is collected. If not lowered carefully into the top of the water column, the agitation may strip volatile compounds. Due to the difficulties and limitations inherent in their use, other sampling or purging means should generally be given preference.

Bailers should be closed-top Teflon® bailers with Teflon® coated stainless steel leaders used with new nylon haul rope. They are lowered gently into the top of the water column, allowed to fill, and removed slowly. It is critical that bailers be slowly and gently immersed into the top of the water column, particularly during final stages of purging and during sampling, to minimize turbidity and loss of volatile organic constituents.

If the well has previously been purged with a pump, there is likely stagnant water at the top of the well that was above the pump or tubing inlet. Several bailers of water should be retrieved and discarded to assure the upper stagnant water has been removed unless the pump was slowly pulled all the way through the top of the water column at the conclusion of pumping to remove any potentially stagnant water.

When sampling, containers are filled directly by pouring from the outlet at the top of the bailer. Containers for metals analysis should be filled first in case the bailing process increases well turbidity. VOC vials should be filled carefully and slowly with a laminar flow to reduce agitation and the stripping of VOCs.

2.7 Wells with In-Place Plumbing

Wells with in-place plumbing are commonly found at municipal water treatment plants, industrial water supplies, private residences, and in other applications. Many permanent monitoring wells at active facilities are also equipped with dedicated, in-place pumps.

A permanent monitoring well with an in-place pump may be treated as other monitoring wells without pumps. Since the in-place pump is generally “hard” mounted at a pre-selected depth, it cannot be moved up or down during purging and sampling. If the pump inlet is above the screened interval, the well should be sampled using the Multiple- Volume Purge method. If the pump intake is located within the screened interval, the well can be sampled using Low-Flow procedures. Known details of pump type and construction, tubing types, pump setting depths, and any other available information about the system should be recorded in the field logbook.

In the case of the other types of wells, e.g., municipal, industrial, residential supply wells and irrigation wells, there is often not enough known about the construction aspects of the wells to apply the same criteria as used for monitoring wells. The volume to be purged in these situations therefore depends on several factors: whether the pumps are running continuously or intermittently and whether or not any storage/pressure tanks are located between the sampling point and the pump. The following subsections describe considerations and procedures to be followed when purging wells with in-place plumbing under the conditions described. Procedures for sampling residential wells are described in the LSASD Applied Science Branch Operating Procedure for Potable Water Supply Sampling ASBPROC-305 R4. Most residential supply wells fall under the intermittently running category.

2.7.1 Continuously Running Pumps

If the pump runs more or less continuously, no purge (other than opening a valve and allowing it to flush for a few minutes) is necessary. If a storage tank is present, a spigot, valve or other sampling point should be found located between the pump and the storage tank. If no valve is present, locate and use the valve closest to the tank. Measurements of field parameters are recorded immediately prior to the time of sampling.

2.7.2 Intermittently or Infrequently Running Pumps

If the pump runs intermittently or infrequently, best judgment should be utilized to remove enough water from the plumbing to flush standing water from the piping and any storage tanks that might be present. Often under these conditions, 15 to 30 minutes of purging will be adequate. If samples must be collected from a tap that is past the collection tank, then it is preferable to determine that the pump is cycling and bringing up fresh formation water to the supply tank (usually the switching on and off of the pump or relay can be heard). Measurements of pH, specific conductance, temperature and turbidity should be made and recorded at intervals during the purge and the final measurements made at the time of sampling should be considered the measurements of record for the event.

2.8 Temporary Monitoring Wells

2.8.1 General Considerations

As temporary wells are installed for immediate sample acquisition, the procedures used to purge temporary ground water monitoring wells may differ from those for permanent wells. Temporary wells include standard well screen and riser placed in boreholes created by hand augering or drilling, or they may consist of a drive rod and screen such as a direct-push Geoprobe® Screen Point that is driven into place at the desired sampling interval. As aquifer water enters the sampler immediately upon deployment, the requirement to remove several volumes of water to replace stagnant water does not necessarily apply. In practice, developing and purging the well to usable turbidity levels will usually remove many times the water that would be removed in a Multiple-Volume Purge with calculated well volumes. It is important to note, however, that the longer a temporary well is in place and not sampled, the more stagnant the water column becomes and the more appropriate it becomes to apply standard permanent monitoring well purging criteria to achieve representative aquifer conditions in the sample.

2.8.2 Development of Temporary Wells

In cases where the temporary well is to be sampled immediately after installation, purging is conducted primarily to mitigate the impacts of installation. In most cases, temporary well installation procedures disturb the existing aquifer conditions, causing extreme turbidity. The goal of development and purging is to reduce the turbidity and remove the volume of water in the area directly impacted by the installation procedure.

As an option, a small diameter inertial pump can be used to agitate and help loosen particles in the screen with the back-and-forth action used to actuate the device. The intake can be used at varying depths within the screen. The inertial pump use should be followed by purging with a peristaltic pump, if the water level is sufficiently high enough.

The following procedure has been found to be effective in developing and sampling small

diameter temporary wells where a peristaltic pump can be used. Turbidity can generally be lowered to 50 NTU at the time of sampling and turbidity less than 10 NTU is often achieved.

1. Cut peristaltic tubing to reach to the bottom of the well. Connect to a peristaltic pump and begin pumping at a high rate.
2. Use the tubing to vacuum out sediment at the bottom of the well.
3. Aggressively surge the end of the tubing in the screened interval by cycling the tubing rapidly up and down and moving the intake to different levels within the screen. Periodically repeat vacuuming of the well bottom.
4. When a visible 'break' to a lower turbidity is observed, cease surging the well and begin lowering the pumping rate.
5. When the water clears (turbidity < 100-200 NTU) begin raising the end of the tubing to the top of the water column.
6. Continue purging from the top of the water column, lowering the pump speed as required to lower turbidity. When adequately low turbidity and stable water quality parameters have been achieved, sampling can proceed.

Where the water level is below the limit of suction in a small diameter temporary well, a Geoprobe® mechanical bladder pump can be used for purging and sampling. The well should first be developed with an inertial pump to remove the bulk of silt and suspended particles that could clog the check valves of the bladder pump. The inertial pump is used to vacuum out the bottom of the well and surged in the screened interval until a 'break' to lower turbidity is observed prior to deployment of the bladder pump. Since the mechanical bladder pump requires cumbersome redeployment to change its pumping level, it should be deployed low enough in the water column that the water level will not be lowered below the pump during purging and sampling. The mechanical bladder pump is generally deployed above the screened interval to facilitate the settling of particles, but below the top of the water column to alleviate the need to reset the pump. Detailed instructions on the deployment of the pump can be found in SESDPROC203, Pump Operation.

If practical, temporary wells should be allowed to sit undisturbed after installation and development for approximately 24 hours before purging and collecting samples.

2.8.3 Decommissioning of Temporary Wells

After temporary wells have fulfilled their purpose, they should be properly decommissioned similar to permanent wells. In general, the casings and screens can be easily removed and the borehole should then be pressure grouted from the bottom of the original borehole to prevent surface contamination of the aquifer, cross-connection of aquifers, and to remove a potential vapor pathway.

Direct-push screen-point wells may be decommissioned by one of two methods:

1. A disposable screen is used. The sampling sheath is pulled off of the screen and a 30% solids bentonite grout is pumped down the tool string as the rods are withdrawn. Grout volumes are measured during pumping to assure that the hole is completely filled. The disposable screen is left behind at the bottom of the borehole.
2. The screen is removed with the sampler sheath and tool string. The hole is immediately re-entered with an empty sample sheath with disposable point. Upon reaching the original total depth of the temporary well, 30% solids bentonite grout is pumped down the tool string with the pumped volume monitored during tool string withdrawal to assure that the hole is completely filled.

A system is available to insert a small diameter grouting tube down through the screen- point screen. Grout is pumped through the grouting tube while the tools are withdrawn. LSASD does not use this system as grout denser than 20% solids cannot reliably be installed with this system.

Additional guidance on decommissioning may be found in SESDGUID-101, Design and Installation of Monitoring Wells.

2.8.4 Other Considerations for Direct-Push Groundwater Sampling

With certain direct push sampling techniques, such as the Hydropunch™ and other discrete samplers used with cone-penetrometer rigs, purging is either not practical or not possible. The sampling device may be simply pushed or driven to the desired depth and opened, whereupon the sample is collected and retrieved. As a result, some samples collected in this way may not be satisfactory or acceptable for certain analyses, i.e., the sampler may collect a turbid sample inappropriate for metals analyses or the sample may have inadequate volume to achieve desired reporting levels.

2.9 Wells Purged to Dryness

In some situations, a well may be screened in a low transmissivity zone and slow recovery allows it to be purged dry (extreme drawdown with water level near the bottom of the casing) even if using slow purge rates. That could occur during the Multiple- Volume Purge method or the Low-Flow method. In these cases, the well should be purged to dryness (evacuated) and sampled upon recovery of adequate volume for sampling. Sampling should occur as soon as adequate volume has recovered, if practical (if the well recovers fully during the night then a sample can be collected the next morning). The field parameters should be measured and recorded at the time of sample collection as the measurements of record for the sampling event.

Sampling under these conditions is not ideal and suitable qualifications of the data should be included in the report. Water cascading down the screen into the well may strip volatile compounds and elevate turbidity. Although suffering from other limitations, No- Purge methods may prove useful for these wells.

3.0 Special Purging and Sampling Procedures and Considerations

3.1 Volatile Organic Compounds (VOC) Analysis

Groundwater samples for VOC analysis must be collected in 40 ml glass vials with Teflon® septa. The vial may be either pre-preserved with concentrated hydrochloric acid or they may be unpreserved. Preserved samples have a two-week holding time prior to being extracted by the laboratory, whereas unpreserved samples have only a seven-day holding time prior to extraction. In the majority of cases, the preserved vials are used to take advantage of the extended holding time. In some situations, however, it may be necessary to use the unpreserved vials. For example, if the groundwater has a high amount of dissolved limestone, i.e., is highly calcareous, there will likely be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles and rendering the sample unacceptable. In this case, unpreserved vials should be used and arrangements confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

The samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a meniscus at the top of the vial and no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm or knuckle to check if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be topped off using a minimal amount of sample to re-establish the meniscus. Care should be taken not to flush any preservative out of the vial during topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained and the sample re-collected. While the 8260 method allows for bubbles up to 6 mm at the time of analysis, dissolved or entrained gases can coalesce during shipment. Collecting VOC vials absent of bubbles is generally feasible and is a reasonable precaution.

3.2 Special Precautions for Trace Contaminant Sampling

- Sampling equipment must be constructed of Teflon® or stainless steel materials. Bailers and pumps should be of Teflon® and stainless steel construction throughout.
- New Teflon® tubing should be used at each well, although tubing dedicated to a particular well may be reused, either after decontamination or storage in the well between sampling events. Caution is appropriate in reusing tubing where early sampling events report high concentrations of contaminants.
- A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers for samples suspected of containing high concentrations of contaminants shall be stored separately.
- Sample collection activities shall proceed progressively from the least suspected

contaminated area to the most suspected contaminated area if purging and sampling devices are to be reused. Samples of waste or highly contaminated media must not be placed in the same cooler as environmental (i.e., containing low contaminant levels) or background samples.

- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.
- Clean plastic sheeting will be placed on the ground at each sample location to prevent or minimize contaminating sampling equipment by accidental contact with the ground surface.
- Samplers must use new, verified certified-clean disposable or non-disposable equipment cleaned according to procedures contained in LSASD Operating Procedure for Field Equipment Cleaning and Decontamination (LSASDPROC-205) or LSASD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) for collection of samples for trace metals or organic compound analyses.

3.3 Trace Organic Compounds and Metals

Special sample handling procedures should be instituted when trace contaminant samples are being collected. All sampling equipment, including pumps, bailers, water level measurement equipment, etc., which contacts the water in the well must be cleaned in accordance with the cleaning procedures described in the LSASD Operating Procedure for Field Equipment Cleaning and Decontamination (LSASDPROC-205) or LSASD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (LSASDPROC-206). Pumps should not be used for sampling unless the interior and exterior portions of the pump and the discharge hoses are thoroughly cleaned. Rinse blank samples should be collected to verify the adequacy of cleaning when using a sampling pump other than a peristaltic pump.

3.4 Sampling for Per- and Polyfluoroalkyl Substances (PFAS) Analysis

Sources of Per- and Polyfluoroalkyl Substances (PFAS) contamination in groundwater can be from discharges or releases, applications of some PFAS products such as aqueous film-forming foams (AFFF), air deposition from manufacturing stack emissions, landfill leachate, and land applications of biosolids or effluents. The distribution of PFAS in groundwater is multifaceted and will be dependent on site-specific conditions as well as the individual properties of the PFAS such as chain length and functional group. Heavy PFAS contamination of subsurface soils can serve as long-term sources for both groundwater and surface water contamination. For more information about conducting site investigations for PFAS, please see the Interstate Technology and Regulatory Council's (ITRC's) August 2020 Fact Sheets: *Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances (PFAS)*, and *Environmental Fate and Transport for Per- and Polyfluoroalkyl Substances*.

3.4.1 Sampling Equipment

Special considerations are required for groundwater sampling equipment for PFAS analyses due to considerations related to PFAS behavior. It is important to consider the composition of the sampling equipment related to its ability to leach PFAS into the sample, or to adsorb PFAS from the sample media. Guidance documents recommend direct contact sampling equipment be inert and made of stainless-steel, high-density polyethylene (HDPE) and/or silicone. Disposable, one-time use equipment should be used when feasible. When equipment that will be in contact with sample media such as surge blocks, bailers, bailing rope, downhole pumps, tubing, electronic water level gauges, etc. cannot be verified as being PFAS-free, equipment blanks should be collected and analyzed to measure any PFAS contribution the equipment may have on the groundwater sample data. Reusable direct contact sampling equipment that will be used to collect samples for PFAS analyses should be decontaminated following the procedures in the *Field Equipment Cleaning and Decontamination at the FEC*, LSASDPROC-206.

Peristaltic pumps with HDPE and silicone tubing have been successfully used by LSASD personnel to collect groundwater samples from shallow monitoring wells and temporary wells for PFAS analyses. For sampling monitoring wells where the water table is deeper than the range of peristaltic pumps, stainless-steel single stage electric pumps can be used with HDPE tubing to both purge and sample the well. The pump's specifications should be reviewed to ensure that Teflon® or PTFE components are replaced when possible. It is advisable to collect the appropriate amount of equipment blanks based on the amount of sampling and field decontamination that is being performed (i.e. daily, 5%, 10%, etc.). Equipment blanks can be collected from the stainless-steel single stage pumps and HDPE tubing using PFAS-free water and clean, 4-Liter plastic graduated cylinders.

Depending on a project's data quality objectives (DQOs), it may be necessary to remove dedicated equipment in a monitoring well. It is advisable for sites with long-term monitoring for PFAS to remove dedicated equipment and replace with PFAS-free sampling equipment and components. When components in dedicated sampling equipment may contain Teflon™/polytetrafluoroethylene (PTFE) or other per-fluorinated compounds, efforts should be made to differentiate PFAS detected in the samples that can be attribute to the formation groundwater and the PFAS that may the result of contamination from the dedicated sampling equipment.

3.4.2 Trace Level Sampling Technique for PFAS

To prevent PFAS contamination, **extreme care** is required when handling containers, samples and equipment that will be used to collect samples for PFAS analyses. **New gloves** must be worn when decontaminating and handling sample containers and equipment. When worn gloves become compromised by writing in a logbook, touching clothing, wiping away sweat, etc. by potential PFAS containing materials, they need to be changed for new gloves. Nitrile gloves are recommended for PFAS sampling investigations. Also, sample containers should be kept covered in original packaging or in Whirl-Paks® until ready for use. The sample container should be kept closed until it is filled with the appropriate volume of groundwater sample. The container's cap should only be kept in the sampler's gloved hand and replaced as soon as the sampling is done.

A trace level sampling technique can be used to minimize PFAS contamination of the samples. This process will require two field personnel for PFAS sample collection. When the field investigators are prepared to fill the sample container(s), a designated sampler will don new gloves while a second designee, with new gloves, will assist by opening sample container packaging/Whirl-Pak®. The designated sampler removes the sample container(s) from the packaging but keeps them closed. Only after the second designee, is ready to fill the sample container does the designated sampler remove the cap and hold it in their hand until the appropriate sample volume is obtained. After capping the sample container(s), return them to their Whirl-Pak®. The designated sampler who holds the sample container(s) should not touch anything else during the sample collection process. This is important because of the wide use of PFAS in commercial products such as clothing and personal hygiene products. Additionally, the designated sampler should avoid touching the sample media and the inside of the sample container. The second designee will operate sampling equipment and assist with sample container packaging and labeling. Sampling equipment known or suspected to contain PFASs should be avoided during sampling activities.

Sampling for groundwater during heavy precipitation shall be avoided due to the potential of rain mixing with the groundwater media during the sampling process. If a light rain begins to fall during the purging process of a well, samplers may erect a pop-up tent to minimize the impact of the precipitation on the groundwater PFAS sampling results. Gore-Tex® and other waterproof, breathable fabrics that contain PFAS should be avoided during PFAS sampling but rubberize rain suits are permissible.

Teflon should not be used for PFOS sample collection.

3.4.3 Quality Control Samples and Standard Operating Procedures

For groundwater samples undergoing PFAS analyses, it extremely important that quality control samples be collected as part of the investigation to demonstrate the PFAS contribution of the sample containers, decontamination solutions, gloves, decontaminated equipment and plastic used to store equipment. It is also important to take field quality control samples such as additional equipment rinse blanks, material blanks, field blanks, duplicates, and trip blanks to evaluate the groundwater sampling and sample handling activities of the investigation. Field blanks are very useful in assessing if the sampler's clothing, safety equipment and/or personal hygiene products had an impact on the sampling results.

Along with a good quality assurance program, standard operating procedures (SOPs) and detailed SAPs are required for PFAS investigations to provide consistency between samplers and investigations.

3.4.4 PFAS Groundwater Sample Handling Considerations and High PFAS Concentration Samples

Since PFAS sampling and analyses is non-routine for many field investigators and some laboratories, it is extremely important to coordinate the sample handling and documentation. The Region 4 LSASD laboratory requires that the samples be double

bagged with the inner bag being labeled with sample information. Sample labels **must not** be fixed to the sample container's exterior. Depending on the PFAS analytical method, two tared sample tubes with serial numbers may be used for the sample containers per sample station. These serial numbers need to be written on the sample's label. Samples are shipped cooled on ice at ≤ 10 °C. It is also helpful to double bag the ice which reduces the amount the ice melts during transport.

When collecting samples for multiple analyses, it is recommended to segregate the sample containers for the PFAS analyses into separate coolers from the containers for the other analyses.

When collecting samples of potentially high concentrations of PFAS groundwater, it is recommended that single-use sampling equipment be utilized if possible. High concentration groundwater samples should be segregated from suspected low-level samples during shipping to the laboratory. Samples that may contain high concentrations of PFAS should be clearly identified on the sample Chain of Custody that accompanies the samples to the laboratory.

3.5 Order of Sampling with Respect to Analytes

In many situations when sampling permanent or temporary monitoring wells, sufficiently low turbidity is difficult to achieve and maintain. Removal and insertion of equipment after the purge or during sampling may elevate turbidity back to unacceptable levels. For this reason, it is important that special efforts be used to minimize any disturbance of the water column after purging and to fill sample containers for metals analysis first. The preferred order of sampling is metals first, followed by other inorganic analytes, extractable organic compounds, and finally volatile organic compounds.

3.6 Filtering

As many contaminants are known to sorb to soil particles, the normal goal of sampling is to reduce the presence of these particles (measured by turbidity) in order that the dissolved concentration of contaminants can be obtained. However, transport of sorbed contamination on colloidal particles can be a means of contaminant transport on some sites. For this reason, the LSASD approach is to reduce turbidity through the careful purging of wells, rather than through filtering of samples, in order that the colloidal particles would be included in the sample.

As a standard practice, ground water samples will not be filtered for routine analysis. Filtering will usually only be performed to determine the fraction of major ions and trace metals passing the filter and used for flow system analysis and for the purpose of geochemical speciation modeling. Filtration is not acceptable to correct for improperly designed or constructed monitoring wells, inadequate well development, inappropriate sampling methods, or poor sampling technique.

When samples are collected for routine analyses and are filtered, both filtered and non-filtered samples will be submitted for analyses. Samples for organic compounds analysis

should not be filtered. Prior to filtration of the ground water sample for any reason other than geochemical speciation modeling, the following criteria must be demonstrated to justify the use of filtered samples for inorganic analysis:

1. The monitoring wells, whether temporary or permanent, have been constructed and developed in accordance with the LSASD Guidance Document, Design and Installation of Monitoring Wells (SESDGUID-001).
2. The ground water samples were collected using sampling techniques in accordance with this section, and the ground water samples were analyzed in accordance with USEPA approved methods.
3. Efforts have been undertaken to minimize any persistent sample turbidity problems. These efforts may consist of the redevelopment or re-installation of permanent ground water monitoring wells or the implementation of carefully conducted low flow rate sampling techniques.

If filtration is necessary for purposes of geochemical modeling or other **pre- approved** cases, the following procedures are suggested:

1. Accomplish in-line filtration through the use of disposable, high capacity filter cartridges (barrel-type) or membrane filters in an in-line filter apparatus. The high capacity, barrel-type filter is preferred due to the higher surface area associated with this configuration. If a membrane filter is utilized, a minimum diameter of 142 mm is suggested.
2. When using pumps for sampling, the filter can generally be attached directly to the pump outlet. When sampling with a bailer or when otherwise required, an initial unfiltered sample with extra volume will be collected, and a peristaltic pump with filter used to decant and filter the sample to the final sample container.
3. Use a 0.45 μm pore-size filter to remove most non-dissolved particles. A 5 μm or 10 μm pore-size filter should be used for the purpose of determining colloidal constituent concentrations.
4. Fill the filter and rinse with approximately one additional filter volume prior to filling sample bottles.

Potential differences can result from variations in filtration procedures used to process water samples for the determination of trace element concentrations. A number of factors associated with filtration can substantially alter "dissolved" trace element concentrations; these include filter pore size, filter type, filter diameter, filtration method, volume of sample processed, suspended sediment concentration, suspended sediment grain-size distribution, concentration of colloids and colloidally-associated trace elements, and concentration of organic matter. Therefore, consistency is critical in the comparison of short-term and long-term results. Further guidance on filtration may be obtained from the following: 1) Metals in Ground Water: Sampling Artifacts and Reproducibility; 2) Filtration of Ground Water Samples for Metals Analysis; and 3) Ground Water Sampling - A Workshop Summary. See Section 4.0, References, for complete citation for these

documents.

3.7 Bacterial Sampling

Whenever wells (normally potable wells) are sampled for bacteriological parameters, care must be taken to ensure the sterility of all sampling equipment and all other equipment entering the well. Further information regarding bacteriological sampling is available in the following: 1) Sampling for Organic Chemicals and Microorganisms in the Subsurface; 2) Handbook for Evaluating Water Bacteriological Laboratories; and 3) Microbiological Methods for Monitoring the Environment, Water and Wastes. See Section 4.0, References, for complete citation for these documents.

3.8 Sample Handling and Preservation Requirements

1. Groundwater samples will typically be collected from the discharge line of a pump or from a bailer. Efforts should be made to reduce the flow from either the pump discharge line or the bailer during sample collection to minimize sample agitation.
2. During sample collection, make sure that the pump discharge line or the bailer does not contact the sample container.
3. Place the sample into appropriate, labeled containers. Samples collected for VOC, and alkalinity analysis must be collected without headspace. All other sample containers must be filled with an allowance for ullage.
4. All samples requiring preservation must be preserved as soon as practically possible, ideally immediately at the time of sample collection. If pre-preserved VOC vials are used, these will be preserved with concentrated hydrochloric acid by Analytical Services Branch (ASB) personnel prior to departure for the field investigation. For all other chemical preservatives, LSASD will use the appropriate chemical preservative generally stored in an individual single-use vial as described in the LSASD Operating Procedure for Field Sampling Quality Control (SESDPROC-011). The adequacy of sample preservation will be checked after the addition of the preservative for all samples except for the samples collected for VOC analysis. If additional preservative is needed, it should be added to achieve adequate preservation. Preservation requirements for groundwater samples are found in the USEPA Region 4 Analytical Services Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM), most recent version. Consult the Analytical Services Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM) for the correct preservative for the particular analytes of interest.
All samples preserved using a pH adjustment (except VOCs) must be checked, using pH strips, to ensure that they were adequately preserved. This is done by pouring a small volume of sample over the strip. Do not place the strip in the sample.
5. Sample containers should be placed in an ice-filled cooler as soon as possible after filling. Ice in coolers should be in bags with minimal pooled water and the cooler should be periodically checked and replenished to maintain sample storage

temperature.

3.9 Field Care of Purging Equipment

New plastic sheeting should be placed on the ground surface around the well casing to prevent contamination of the pumps, hoses, ropes, etc., in the event they accidentally come into contact with the ground surface or, for some reason, they need to be placed on the ground during the purging event. It is preferable that hoses used in purging that come into contact with the ground water be kept on a spool or contained in a large wash tub lined with plastic sheeting, both during transportation and during field use, to further minimize contamination by the transporting vehicle or the ground surface.

Careful consideration shall be given to using submersible centrifugal or bladder pumps to purge wells which are excessively contaminated with oily compounds as it may be difficult to adequately decontaminate severely contaminated pumps under field conditions. When wells of this type are encountered, alternative equipment, such as bailers or peristaltic pumps, should be considered.

3.10 Specific Sampling Equipment Quality Assurance Techniques

All equipment used to collect ground water samples shall be cleaned as outlined in the LSASD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or LSASD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206). Malfunctioning equipment should be labeled in the field and repaired, before being stored at the conclusion of field studies. Cleaning procedures utilized in the field or field repairs shall be thoroughly documented in field records.

3.11 Quality Control

Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary to document that low-level contaminants were not introduced by pumps, bailers, tubing, or other sampling equipment.

Where appropriate, a background sample upgradient of all known influences or a control sample upgradient of site influences may be indicated. Background and control samples should be collected as close to the sampled area as possible and from the same water-bearing formation as the site samples.

3.12 Investigation Derived Waste

Purging, well development and field cleaning of equipment generate liquid investigation derived waste (IDW), the disposition of which must be considered. See LSASD Operating Procedure for Management of Investigation Derived Waste (SESDPROC-202) for guidance on management or disposal of this waste.

3.13 Records

Information generated or obtained by LSASD personnel will be organized and accounted for in accordance with LSASD records management procedures found in LSASD Operating Procedure for Control of Records, SESDPROC-002. Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation in accordance with LSASD Operating Procedure for Logbooks, SESDPROC-010 and LSASD Procedure for Sample and Evidence Management, SESDPROC-005.

3.14 Auxiliary Data Collection

During ground water sample collection, it is important to record a variety of ground water related data. Included in the category of auxiliary data are water levels measured according to the LSASD Operating Procedure for Groundwater Level and Well Depth Measurement (SESDPROC-105), well volume determinations, pumping rates during purging, driller or boring logs, and number of drums left on site if IDW is temporarily stored there. This information should be documented in the field records.

3.15 Well Development

Wells may be encountered that are difficult to sample effectively due to inadequate initial development or the need for redevelopment due to scaling, sedimentation, corrosion, or biofouling. Some wells were not properly developed when installed. Those wells may have borehole walls smeared with clays, sandpucks and screens that have not been cleared of smaller particles, and sediments accumulated in the bottom of the casing. These wells may produce water at much lower flow rates than the formations are capable of, and often produce water with chronically elevated turbidity. Transmissivity calculations for poorly developed wells may not be inaccurate. Redevelopment of these wells should be considered as the process can improve sample quality and speed stabilization for purging and sampling events. General well development procedures are described in Design and Installation of Monitoring Wells (SESDGUID-101). Additional well development explanation and description of techniques are included in the book *Groundwater and Wells* (referenced below) and other publications.

Techniques commonly used for conditioning and re-development of monitoring wells include using snug-fitting swabs on sections of tremie pipe to clean screens and create positive and negative pressure surges through the screen, surging, removing sediments in the bottom of the well with inertial pumps, aggressive pumping and surging throughout the screen with submersible pumps, and use of automated inertial pump systems such as the Waterra system. Drillers may be able to use compressed air to assist in development. Techniques used and amount of time and effort for development will depend on the condition of the well and the construction of the well (borehole diameter, depth, screen length).

4.0 References

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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the LSASD Document Control Coordinator on the LSASD local area network (LAN).

History	Effective Date
Replaced Chief with Supervisor; General formatting revisions.	April 22, 2023
<p>LSASDPROC-301-R5, Groundwater Sampling, replaces SESDPROC-301-R4.</p> <p>General: Corrected any typographical, grammatical, and/or editorial errors.</p> <p>General: Reorganization of sections, including moving references section to end of document. Elimination of redundancy. Clarifications of certain aspects of low-flow methods.</p> <p>Specific: Addition of PFAS sampling section.</p>	November 23, 2021
<p>SESDPROC-301-R4, Groundwater Sampling, replaces SESDPROC-301-R3.</p> <p>General: Corrected any typographical, grammatical, and/or editorial errors.</p> <p>General: An extensive rewrite and reorganization of material. Stronger support of low-flow methods while maintaining cautious view of minimal/no purge methods.</p>	April 26, 2017
SESDPROC-301-R3, Groundwater Sampling, replaces SESDPROC-301-R2.	March 6, 2013
SESDPROC-301-R2, Groundwater Sampling, replaces SESDPROC-301-R1.	October 28, 2011
SESDPROC-301-R1, Groundwater Sampling, replaces SESDPROC-301-R0.	November 1, 2007
SESDPROC-301-R0, Groundwater Sampling, Original Issue	February 05, 2007

Region 4 U.S. Environmental Protection Agency Laboratory Services & Applied Science Division Athens, Georgia	
Operating Procedure	
Title: Soil Sampling	ID: LSASDPROC-300-R5
Issuing Authority: Field Services Branch Supervisor	
Effective Date: April 22, 2023	Review Due Date: June 10, 2024
Method Reference: N/A	SOP Author: Kevin Simmons

Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when collecting soil samples for field screening or laboratory analysis.

Scope/Application

The procedures contained in this document are to be used by field personnel when collecting and handling soil samples in the field. On the occasion that LSASD field personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to obtain a soil sample, the variant procedure will be documented in the field logbook and subsequent investigation report, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

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1 General Information

1.1 Documentation/Verification

This procedure was prepared by persons deemed technically competent by LSASD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the LSASD local area network (LAN). The QAC is responsible for ensuring the most recent version of the procedure is placed on the LAN, and for maintaining records of review conducted prior to its issuance.

1.2 General Precautions

1.2.1 Safety

Proper safety precautions must be observed when collecting soil samples. Refer to the LSASD Safety and Occupational Health Manual and any pertinent site-specific Health and Safety Plans (HASP) and Job Hazard Assessments for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. The reader should address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.2.2 Procedural Precautions

The following precautions should be considered when collecting soil samples:

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Documentation of field sampling is done in a bound logbook.

- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as air bills, bills of lading, etc., shall be retained by the project leader in the project files. (Air bills are generated online via UPS Campusship program and package tracking is done online). Receipts are not always received at time of shipping.
- Sampling in landscaped areas: Cuttings should be placed on plastic sheeting and returned to the borehole upon completion of the sample collection. Any 'turf plug' generated during the sampling process should be returned to the borehole.
- Sampling in non-landscaped areas: Return any unused sample material back to the auger, drill or push hole from which the sample was collected.

2 Special Sampling Considerations

2.1 Special Precautions for Trace Contaminant Soil Sampling

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different sample is collected and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers with samples suspected of containing high concentrations of contaminants shall be handled and stored separately.
- All background samples shall be segregated from obvious high-concentration or waste samples. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area. Samples of waste or highly-contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.
- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other member(s) collect the samples.
- Samplers must use new, verified/certified-clean disposable or non-disposable equipment cleaned according to procedures contained in the LSASD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205), for collection of samples for trace metals or organic compound analyses.

2.2 Sample Homogenization

1. If sub-sampling of the primary sample is to be performed in the laboratory, transfer the entire primary sample directly into an appropriate, labeled sample container(s). Proceed to step 4.
2. If sub-sampling the primary sample in the field or compositing multiple primary samples in the field, place the sample into a glass or stainless steel homogenization container and mix thoroughly. Each aliquot of a composite sample should be of the same approximate volume.
3. All soil samples must be thoroughly mixed to ensure that the sample is as representative as possible of the sample media. ***Samples for VOC analysis are not homogenized.*** The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:
 - The material in the sample pan should be divided into quarters and each quarter should be mixed individually.
 - Two quarters should then be mixed to form halves.
 - The two halves should be mixed to form a homogenous matrix.

This procedure should be repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction, and occasionally turning the material over.

4. Place the sample into an appropriate, labeled container(s) by using the alternate shoveling method and secure the cap(s) tightly. The alternate shoveling method involves placing a spoonful of soil in each container in sequence and repeating until the containers are full or the sample volume has been exhausted. Threads on the container and lid should be cleaned to ensure a tight seal when closed.

2.3 Dressing Soil Surfaces

Any time a vertical or near vertical surface is sampled, such as achieved when shovels or similar devices are used for subsurface sampling, the surface should be dressed (scraped) to remove smeared soil. This is necessary to minimize the effects of contaminant migration interferences due to smearing of material from other levels.

2.4 Quality Control

If possible, a control sample should be collected from an area not affected by the possible contaminants of concern and submitted with the other samples. This control sample should be collected as close to the sampled area as possible and from the same soil type. Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary to document that low-level contaminants were not introduced by sampling tools. LSASD Operating Procedure for Field Sampling Quality Control (SESDPROC-011) contains other procedures that may be applicable to soil sampling investigations.

2.5 Records

Field notes, recorded in a bound field logbook, as well as chain-of-custody documentation will be generated as described in the LSASD Operating Procedure for Logbooks (SESDPROC-010) and the LSASD Operating Procedure for Sample and Evidence Management (SESDPROC-005).

3 Samples Collected for Volatile Organic Compounds (VOC) or for Per- and Polyfluoroalkyl Substances (PFAS) Analyses

3.1 Soil Samples Collected for Volatile Organic Compounds (VOC) Analysis

The procedures outlined here are summarized from *Test Methods for Evaluating SolidWaste, Physical/Chemical Methods SW-846, Method 5035*. If samples are to be analyzed for VOCs, they should be collected in a manner that minimizes disturbance of the sample. For example, when sampling with an auger bucket, the sample for VOC analysis should be collected directly from the auger bucket (preferred) or from minimally disturbed material immediately after an auger bucket is emptied into the pan. The sample shall be containerized by filling an En Core® Sampler or other Method 5035 compatible container. ***Samples for VOC analysis are not homogenized.*** Preservatives may be required for some samples with certain variations of Method 5035. Consult the method or the principal analytical chemist to determine if preservatives are necessary.

3.2 Soil Sampling for VOCs (Method 5035)

The following sampling protocol is recommended for site investigators assessing the extent of VOCs in soils at a project site. Because of the large number of options

available, careful coordination between field and laboratory personnel is needed. The specific sampling containers and sampling tools required will depend upon the detection levels and intended data use. Once this information has been established, selection of the appropriate sampling procedure and preservation method best applicable to the investigation can be made.

3.2.1 Equipment

Soil for VOC analyses may be retrieved using any of the LSASD soil sampling methods described in Sections 4 through 8 of this procedure. Once the soil has been obtained, the En Core® Sampler, syringes, stainless steel spatula, standard 2- oz. soil VOC container, or pre-prepared 40 mL vials may be used/required for sub-sampling. The specific sample containers and the sampling tools required will depend upon the data quality objectives established for the site or sampling investigation. The various sub-sampling methods are described below.

3.2.2 Sampling Methodology - Low Concentrations (<200 µg/kg)

When the total VOC concentration in the soil is expected to be less than 200 µg/kg, the samples may be collected directly with the En Core® Sampler or syringe. If using the syringes, the sample must be placed in the sample container (40 mL pre-prepared vial) immediately to reduce volatilization losses. The 40 mL vials should contain 10 mL of organic-free water for an un-preserved sample or approximately 10 mL of organic-free water and a preservative. It is recommended that the 40 mL vials be prepared and weighed by the laboratory (commercial sources are available which supply preserved and tared vials). When sampling directly with the En Core® Sampler, the vial must be immediately capped and locked.

A soil sample for VOC analysis may also be collected with conventional sampling equipment. A sample collected in this fashion must either be placed in the final sample container (En Core® Sampler or 40 mL pre-prepared vial) immediately or the sample may be immediately placed into an intermediate sample container with no head space. If an intermediate container (usually 2-oz. soil jar) is used, the sample must be transferred to the final sample container (En Core® Sampler or 40 mL pre-prepared vial) as soon as possible, not to exceed 30 minutes.

NOTE:After collection of the sample into either the En Core® Sampler or other container, the sample must immediately be stored in an ice chest and cooled.

Soil samples may be prepared for shipping and analysis as follows:

En Core® Sampler - the sample shall be capped, locked, and secured in the original foil bag. All foil bags containing En Core® samplers are then placed in a plastic bag and sealed with custody tape, if required.

Syringe - Add about 3.7 cc (approximately 5 grams) of sample material to 40-mL pre-prepared containers. Secure the containers in a plastic bag. Do not use a custody seal on the container; place the custody seal on the plastic bag. Note: When using the syringes, it is important that no air is allowed to become trapped behind the sample prior to extrusion, as this will adversely affect the sample.

Stainless Steel Laboratory Spatulas - Add between 4.5 and 5.5 grams (approximate) of sample material to 40 mL containers. Secure the containers in a plastic bag. Do not use a custody seal on the container; place the custody seal on the plastic bag.

3.2.3 Sampling Methodology - High Concentrations (>200 µg/kg)

Based upon the data quality objectives and the detection level requirements, this high-level method may also be used. Specifically, the sample may be packed into a single 2-oz. glass container with a screw cap and septum seal. The sample container must be filled quickly and completely to eliminate head space. Soils\sediments containing high total VOC concentrations may also be collected as described in Section 3.2.2, Sampling Methodology - Low Concentrations, and preserved using 10 mL methanol.

3.2.4 Special Techniques and Considerations for Method 5035

Effervescence

If low concentration samples effervesce (rapidly form bubbles) from contact with the acid preservative, then either a test for effervescence must be performed prior to sampling, or the investigators must be prepared to collect each sample both preserved or un-preserved, as needed, or all samples must be collected unpreserved.

To check for effervescence, collect a test sample and add to a pre-preserved vial. If preservation (acidification) of the sample results in effervescence then preservation by acidification is not acceptable, and the sample must be collected un-preserved.

If effervescence occurs and only pre-preserved sample vials are available, the preservative solution may be placed into an appropriate hazardous waste container and the vials triple rinsed with organic free water. An appropriate amount of organic free water, equal to the amount of preservative solution, should be placed

into the vial. The sample may then be collected as an un-preserved sample. Note: the amount of organic free water placed into the vials will have to be accurately measured.

Sample Size

While this method is an improvement over earlier ones, field investigators must be aware of an inherent limitation. Because of the extremely small sample size and the lack of sample mixing, sample representativeness for VOCs may be reduced compared to samples with larger volumes collected for other constituents. The sampling design and objectives of the investigation should take this into consideration.

Holding Times

Sample holding times are specified in the Laboratory Services Branch *Laboratory Operations and Quality Assurance Manual* (ASBLOQAM), Most Recent Version. Field investigators should note that the holding time for an un-preserved VOC soil/sediment sample on ice is 48 hours. Arrangements should be made to ship the soil/sediment VOC samples to the laboratory by overnight delivery the day they are collected so the laboratory may preserve and/or analyze the sample within 48 hours of collection.

Percent Solids

Samplers must ensure that the laboratory has sufficient material to determine percent solids in the VOC soil/sediment sample to correct the analytical results to dry weight. If other analyses requiring percent solids determination are being performed upon the sample, these results may be used. If not, a separate sample (minimum of 2 oz.) for percent solids determination will be required. The sample collected for percent solids may also be used by the laboratory to check for preservative compatibility.

Safety

Methanol is a toxic and flammable liquid. Therefore, methanol must be handled with all required safety precautions related to toxic and flammable liquids. Inhalation of methanol vapors must be avoided. Vials should be opened and closed quickly during the sample preservation procedure. Methanol must be handled in a ventilated area. Use protective gloves when handling the methanol vials. Store methanol away from sources of ignition such as extreme heat or open flames. The vials of methanol should be stored in a cooler with ice at all times.

Shipping

Methanol and sodium bisulfate are considered dangerous goods, therefore shipment of samples preserved with these materials by common carrier is regulated by the U.S. Department of Transportation and the International Air Transport Association (IATA). The rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179) and the current edition of the IATA Dangerous Goods Regulations must be followed when shipping methanol and sodium bisulfate. Consult the above documents or the carrier for additional information. Shipment of the quantities of methanol and sodium bisulfate used for sample preservation falls under the exemption for small quantities.

The summary table on the following page lists the options available for compliance with SW846 Method 5035. The advantages and disadvantages are noted for each option. LASSD's goal is to minimize the use of hazardous material (methanol and sodium bisulfate) and minimize the generation of hazardous waste during sample collection.

Table 1: Method 5035 Summary

OPTION	PROCEDURE	ADVANTAGES	DISADVANTAGES
1	Collect two 40 mL vials with \approx 5 grams of sample, and one 2 oz. glass jar w/septum lid for screening, % moisture and preservative compatibility.	Screening conducted by lab.	Presently a 48-hour holding time for unpreserved samples. Sample containers must be tared.
2	Collect three En Core® samplers, and one 2 oz. glass jar w/septum lid for screening, % solids.	Lab conducts all preservation/preparation procedures.	Presently a 48- hour holding time for preparation of samples.
3	Collect two 40 mL vials with 5 grams of sample and preserve w/methanol or sodium bisulfate, and one 2-oz. glass jar w/septum lid for screening, % solids .	High level VOC samples may be composited. Longer holding time.	Hazardous materials used in the field. Sample containers must be tared.
4	Collect one 2-oz. glass jar w/septum lid for analysis, % solids (high level VOC only).	Lab conducts all preservation/preparation procedures.	May have significant VOC loss.

3.3 Soil Samples for Per- and Polyfluoroalkyl Substances (PFAS) Analysis

Sources of PFAS contamination in soils can include direct discharges, direct applications of some PFAS products such as aqueous film-forming foams (AFFF), air deposition from manufacturing stack emissions, landfill leachate, and land applications of biosolids or effluents. The distribution of PFAS in soils is multifaceted and will be dependent on site-specific conditions and soils as well as the individual properties of the PFAS such as chain length and functional group. Heavy PFAS contamination of subsurface soils can serve as long-term sources for both groundwater and surface water contamination. For more information about conducting site investigations for PFAS, please see the Interstate Technology and Regulatory Council's (ITRC's) April 2020 Fact Sheets: *Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances (PFAS)*, and *Environmental Fate and Transport for Per- and Polyfluoroalkyl Substances*.

3.3.1 Sampling Equipment

Guidance documents recommend sampling equipment be made of stainless-steel, high-density polyethylene (HDPE), polypropylene, and/or silicone. Standard soil sampling equipment such as stainless-steel spoons, hand augers, and direct push samplers with liners that are PFAS-free can be used to collect samples for PFAS analyses. Direct contact sampling equipment that will be used to collect samples for PFAS analyses should be decontaminated following the procedures in the *Field Equipment Cleaning and Decontamination at the FEC*, LSASDPROC-206.

3.3.2 PFAS Soil Sample Mixing and Homogenization Considerations

Because studies have shown the loss of PFAS due to adsorption to surfaces, samples should be minimally handled and directly placed into the sample container when possible. Sample preparation procedures should be specified in the Sampling and Analysis Plan (SAP). If compositing, mixing or homogenization of the sample is desired, it should preferably be done at the laboratory so that a representative subsample will be analyzed. In cases where the homogenization is conducted in the field, extra grab samples should accompany the mixed or composited samples to determine the variability and impacts on PFAS concentrations of the mixed samples.

3.3.3 Trace Level Sampling Technique for PFAS

To prevent PFAS contamination, **extreme care** is required when handling containers, samples and equipment that will be used to collect samples for PFAS analyses. **New gloves** need to be worn when decontaminating and handling sample containers and equipment. When worn gloves become compromised by potential PFAS containing materials, they need to be changed for new gloves. Nitrile gloves are recommended for PFAS sampling investigations. Also, sample containers should be kept covered in original packaging or in Whirl-Paks® until ready for use due to potential PFAS

contamination from air deposition of vapors, aerosols, and particulates.

This trace level sampling technique is used to minimize PFAS contamination of the samples. This process will require two field personnel for PFAS sample collection. When the field investigators are prepared to fill the sample container(s), a designated sampler will don new gloves while a second designee, also with new gloves, will assist by opening sample container packaging/Whirl-Pak®. The designated sampler removes the sample container(s) from the packaging but keeps them closed. Only after the second designee is ready to fill the sample container does the designated sampler remove the cap and hold it in their hand until the appropriate sample volume is obtained. After capping the sample container(s), return them to their Whirl-Pak®. The designated sampler who holds the sample container(s) should not touch anything else during the sample collection process. This is important because of the wide use of PFAS in commercial products such as clothing, field gear, personnel protective equipment, sunscreen, insect repellants, and personal hygiene products. Additionally, the designated sampler should avoid touching the sample media and the inside of the sample container. The second designee will operate sampling equipment and assist with sample container packaging and labeling. Sampling equipment known or suspected to contain PFAS should be avoided during sampling activities.

3.3.4 Quality Control Samples and Standard Operating Procedures

For soil samples undergoing PFAS analyses, it extremely important that quality control samples be collected as part of the investigation to account for the PFAS contribution of the sample containers, decontamination solutions, gloves, decontaminated equipment and plastic used to store equipment. Equipment rinse and material blanks are needed for PFAS sampling investigations to assess the direct contact sampling equipment impact on the sampling results. It is also helpful to take field quality control samples such as field blanks, duplicates, and trip blanks to evaluate the soil sampling and sample handling activities of the investigation. Laboratory sources of water used for equipment decontamination and blank sample collection should be produced as PFAS-free or assessed for background concentrations of PFAS.

Along with a good quality assurance program, standard operating procedures (SOPs) and detailed SAPs are required for PFAS investigations to provide consistency between samplers and investigations.

4 Manual Soil Sampling Methods

4.1 General

These methods are used primarily to collect surface and shallow subsurface soil samples. Surface soils are generally classified as soils between the ground surface and 6 to 12 inches below ground surface. The most common interval is 0 to 6 inches; however, the data quality objectives of the investigation may dictate another interval, such as 0 to 3 inches for risk assessment purposes. The shallow subsurface interval may be considered to extend from approximately 12 inches below ground surface to a site-specific depth at which sample collection using manual collection methods becomes impractical.

If a thick, matted root zone, gravel, concrete, etc. is present at or near the surface, it should be removed before the sample is collected. The depth measurement for the sample begins at the top of the soil horizon, immediately following any removed materials.

When compositing, make sure that each composite location (aliquot) consist of equal volumes, i.e., same number of equal spoonfuls.

4.2 Spoons

Stainless steel spoons may be used for surface soil sampling to depths of approximately 6 inches below ground surface where conditions are generally soft and non-indurated, and there is no problematic vegetative layer to penetrate.

4.2.1 Special Considerations When Using Spoons

When using stainless steel spoons, consideration must be given to the procedure used to collect the volatile organic compound sample. If the soil being sampled is cohesive and holds its in situ texture in the spoon, the En Core® Sampler or syringe used to collect the sub-sample for Method 5035 should be plugged directly from the spoon. If, however, the soil is not cohesive and crumbles when removed from the ground surface for sampling, consideration should be given to plugging the sample for Method 5035 directly from the ground surface at a depth appropriate for the investigation Data Quality Objectives.

4.3 Hand Augers

Hand augers may be used to advance boreholes and collect soil samples in the surface and shallow subsurface intervals. Typically, 3-inch stainless steel auger buckets with cutting

heads are used. The bucket is advanced by simultaneously pushing and turning using an attached handle with extensions (if needed).

4.3.1 Surface Soil Sampling

When conducting surface soil sampling with hand augers, the auger buckets may be used with a handle alone or with a handle and extensions. The bucket is advanced to the appropriate depth and the contents are transferred to the homogenization container for processing. Observe precautions for volatile organic compound and PFAS sample collection found in Section 3.

4.3.2 Subsurface Soil Sampling

Hand augers are the most common equipment used to collect shallow subsurface soil samples. Auger holes are advanced one bucket at a time until the sample depth is achieved. When the sample depth is reached, the bucket used to advance the hole is removed and a clean bucket is attached. The clean auger bucket is then placed in the hole and filled with soil to make up the sample and removed.

The practical depth of investigation using a hand auger depends upon the soil properties and depth of investigation. In sand, augering is usually easily performed, but the depth of collection is limited to the depth at which the sand begins to flow or collapse. Hand augers may also be of limited use in tight clays or cemented sands. In these soil types, the greater the depth attempted, the more difficult it is to recover a sample due to increased friction and torquing of the hand auger extensions. At some point these problems become so severe that power equipment must be used.

4.3.3 Special Considerations for Soil Sampling with the Hand Auger

- Because of the tendency for the auger bucket to scrape material from the sides of the auger hole while being extracted, the top several inches of soil in the auger bucket should be discarded prior to placing the bucket contents in the homogenization container for processing.
- Observe precautions for volatile organic compound (VOC) and PFAS sample collection found in Section 3. Collect the VOC sample directly from the auger bucket, if possible.
- Power augers, such as the Little Beaver® and drill rigs may be used to advance boreholes to depths for subsurface soil sampling with the hand auger. They may not be used for sample collection. When power augers are used to advance a borehole to depth for sampling, care must be taken that exhaust fumes, gasoline and/or oil do not contaminate the borehole or area in the immediate vicinity of sampling.
- When moving to a new sampling location, the entire hand auger assembly must be replaced with a properly decontaminated hand auger assembly.

5 Direct Push Soil Sampling Methods

5.1 General

These methods are used primarily to collect shallow and deep subsurface soil samples. Three samplers are available for use within the Division's direct push tooling inventory. All of the sampling tools involve the collection and retrieval of the soil sample within a thin-walled liner. The following sections describe each of the specific sampling methods that can be accomplished using direct push techniques, along with details specific to each method. While LSASD currently uses the sample tooling described, tooling of similar design and materials is acceptable.

If gravel, concrete, etc. is present at or near the surface, it should be removed before the sample is collected. The depth measurement for the sample begins at the top of the soil horizon, immediately following any removed materials. Turf grass is not typically removed prior to sampling with these devices.

5.2 Large Bore® Soil Sampler

The Large Bore® (LB) sampler is a solid barrel direct push sampler equipped with a piston-rod point assembly used primarily for collection of depth-discrete subsurface soil samples. The sample barrel is approximately 30-inches (762 mm) long and has a 1.5-inch (38 mm) outside diameter. The LB® sampler is capable of recovering a discrete sample core 22 inches x 1.0 inch (559 mm x 25 mm) contained inside a removable liner. The resultant sample volume is a maximum of 283 mL.

After the LB® sample barrel is equipped with the cutting shoe and liner, the piston-rod point assembly is inserted, along with the drive head and piston stop assembly. The assembled sampler is driven to the desired sampling depth, at which time the piston stop pin is removed, freeing the push point. The LB® sampler is then pushed into the soil a distance equal to the length of the LB® sample barrel. The probe rod string, with the LB® sampler attached, is then removed from the subsurface. After retrieval, the LB® sampler is then removed from the probe rod string. The drive head is then removed to allow removal of the liner and soil sample.

5.3 Macro-Core® Soil Sampler

The Macro-Core® (MC) sampler is a solid barrel direct push sampler equipped with a piston-rod point assembly used primarily for collection of either continuous or depth-discrete subsurface soil samples. Although other lengths are available, the standard MC® sampler has an assembled length of approximately 52 inches (1321 mm) with an outside

diameter of 2.2 inches (56 mm). The MC® sampler is capable of recovering a discrete sample core 45 inches x 1.5 inches (1143 mm x 38 mm) contained inside a removable liner. The resultant sample volume is a maximum of 1300 mL. The MC® sampler may be used in either an open-tube or closed-point configuration. Although the MC® sampler can be used as an open-barrel sampler, in LSASD usage, the piston point is always used to prevent the collection of slough from the borehole sides.

5.4 Dual Tube Soil Sampling System

The Dual Tube 21 soil sampling system is a direct push system for collecting continuous core samples of unconsolidated materials from within a sealed outer casing of 2.125-inch (54 mm) OD probe rod. The samples are collected within a liner that is threaded onto the leading end of a string of 1.0-inch diameter probe rod. Collected samples have a volume of up to 800 mL in the form of a 1.125-inch x 48-inch (29 mm x 1219 mm) core. Use of this method allows for collection of continuous core inside a cased hole, minimizing or preventing cross-contamination between different intervals during sample collection. The outer casing is advanced, one core length at a time, with only the inner probe rod and core being removed and replaced between samples. If the sampling zone of interest begins at some depth below ground surface, a solid drive tip must be used to drive the dual tube assembly and core to its initial sample depth.

5.5 Special Considerations When Using Direct Push Sampling Methods

- *Liner Use and Material Selection* – Direct Push Soil Samples are collected within a liner to facilitate removal of sample material from the sample barrel. The liners may only be available in a limited number of materials for a given sample tool, although overall, liners are available in brass, stainless steel, cellulose acetate butyrate (CAB), polyethylene terephthalate glycol (PETG), polyvinyl chloride (PVC) and Teflon®. For most LSASD investigations, the standard polymer liner material for a sampling tool will be acceptable. When the study objectives require very low reporting levels or unusual contaminants of concern, the use of more inert liner materials such as Teflon® or stainless steel may be necessary.
- *Sample Orientation* – When the liners and associated sample are removed from the sample tubes, it is important to maintain the proper orientation of the sample. This is particularly important when multiple sample depths are collected from the same push. It is also important to maintain proper orientation to define precisely the depth at which an aliquot was collected. Maintaining proper orientation is typically accomplished using vinyl end caps. Convention is to place red caps on the top of the liner and black caps on the bottom to maintain proper sample orientation. Orientation can also be indicated by marking on the exterior of the liner with a permanent marker.

- *Core Catchers* – Occasionally the material being sampled lacks cohesiveness and is subject to crumbling and falling out of the sample liner. In cases such as these, the use of core catchers on the leading end of the sampler may help retain the sample until it is retrieved to the surface. Core catchers may only be available in specific materials and should be evaluated for suitability. However, given the limited sample contact that core-catchers have with the sample material, most standard core-catchers available for a tool system will be acceptable.
- *Decontamination* – The cutting shoe and piston rod point are to be decontaminated between each sample, using the procedures specified for the collection of trace organic and inorganic compounds found in Field Equipment and Decontamination – SESDPROC-205, most recent version. Within a borehole, the sample barrel, rods, and drive head may be subjected to an abbreviated cleaning to remove obvious and loose material, but must be cleaned between boreholes using the procedures specified for downhole drilling equipment in Field Equipment and Decontamination – SESDPROC-205, most recent version.
- *Decommissioning* – Boreholes must be decommissioned after the completion of sampling. Boreholes less than 10 feet deep that remain open and do not approach the water table may be decommissioned by pouring 30% solids bentonite grout from the surface or pouring bentonite pellets from the surface, hydrating the pellets in lifts. Boreholes deeper than 10 feet, or any borehole that intercepts groundwater, must be decommissioned by pressure grouting with 30% solids bentonite grout, either through a re-entry tool string or through tremie pipe introduced to within several feet of the borehole bottom.
- *VOC and PFAS Sample Collection* – Observe precautions for volatile organic compounds and Per- and Polyfluoroalkyl Substances sample collection found in Section 3 of this procedure.

6 Split Spoon/Drill Rig Methods

6.1 General

Split spoon sampling methods are used primarily to collect shallow and deep subsurface soil samples. All split spoon samplers, regardless of size, are basically split cylindrical barrels that are threaded on each end. The leading end is held together with a beveled threaded collar that functions as a cutting shoe. The other end is held together with a threaded collar that serves as the sub used to attach the spoon to the string of drill rod. Two basic methods are available for use, including the smaller diameter standard split spoon, driven with the drill rig safety hammer, and the larger diameter continuous split spoon,

advanced inside and slightly ahead of the lead auger during hollow stem auger drilling. The following sections describe each of the specific sampling methods, along with details specific to each method.

If gravel, concrete, etc. is present at or near the surface, it should be removed before the sample is collected. The depth measurement for the sample begins at the top of the soil horizon, immediately following any removed materials. Turf grass is not typically removed prior to sampling with these devices.

6.2 Standard Split Spoon

A drill rig is used to advance a borehole to the target depth. The drill string is then removed and a standard split spoon is attached to a string of drill rod. Split spoons used for soil sampling must be constructed of stainless steel and are typically 2.0-inches OD (1.5-inches ID) and 18-inches to 24-inches in length. Other diameters and lengths are common and may be used if constructed of the proper material. After the spoon is attached to the string of drill rod, it is lowered into the borehole. The safety hammer is then used to drive the split spoon into the soil at the bottom of the borehole. After the split spoon has been driven into the soil, filling the spoon, it is retrieved to the surface, where it is removed from the drill rod string and opened for sample acquisition.

6.3 Continuous Split Spoon

The continuous split spoon is a large diameter split spoon that is advanced into the soil column inside a hollow stem auger. Continuous split spoons are typically 3 to 5 inches in diameter and either 5 feet or 10 feet in length, although the 5-foot long samplers are most common. After the auger string has been advanced into the soil column a distance equal to the length of the sampler being used it is returned to the surface. The sampler is removed from inside the hollow stem auger and the threaded collars are removed. The split spoon is then opened for sampling.

6.4 Special Considerations When Using Split Spoon Sampling Methods

- Always discard the top several inches of material in the spoon before removing any portion for sampling. This material normally consists of borehole wall material that has sloughed off of the borehole wall after removal of the drill string prior to and during inserting the split spoon.
- Observe precautions for volatile organic compounds and Per- and Polyfluoroalkyl Substances sample collection found in Section 3.

7 Shelby Tube/Thin-Walled Sampling Methods

7.1 General

Shelby tubes, also referred to generically as thin-walled push tubes or Acker thin-walled samplers, are used to collect subsurface soil samples in cohesive soils and clays during drilling activities. In addition to samples for chemical analyses, Shelby tubes are also used to collect relatively undisturbed soil samples for geotechnical analyses, such as hydraulic conductivity and permeability, to support hydrogeologic characterizations at hazardous waste and other sites.

If gravel, concrete, etc. is present at or near the surface, it should be removed before the sample is collected. The depth measurement for the sample begins at the top of the soil horizon, immediately following any removed materials. Turf grass is not typically removed prior to sampling with this device.

7.2 Shelby Tube Sampling Method

A typical Shelby tube is 30 inches in length and has a 3.0-inch OD (2.875-inch ID) and may be constructed of steel, stainless steel, galvanized steel, or brass. They also typically are attached to push heads that are constructed with a ball-check to aid in holding the contained sample during retrieval. If used for collecting samples for chemical analyses, it must be constructed of stainless steel. If used for collecting samples for standard geotechnical parameters, any material is acceptable.

To collect a sample, the tube is attached to a string of drill rod and is lowered into the borehole, where the sampler is then pressed into the undisturbed material by hydraulic force. After retrieval to the surface, the tube containing the sample is then removed from the sampler head. If samples for chemical analyses are needed, the soil contained inside the tube is then removed for sample acquisition. If the sample is collected for geotechnical parameters, the tube is typically capped, maintaining the sample in its relatively undisturbed state, and shipped to the appropriate geotechnical laboratory.

7.3 Special Considerations When Using Split Spoon Sampling Methods

Observe precautions for volatile organic compounds and Per- and Polyfluoroalkyl Substances sample collection found in Section 3.

8 Backhoe Sampling Method

8.1 General

Backhoes may be used in the collection of surface and shallow subsurface soil samples. The trenches created by excavation with a backhoe offer the capability of collecting samples from very specific intervals and allow visual correlation with vertically and horizontally adjacent material. If possible, the sample should be collected without entering the trench. Samples may be obtained from the trench wall or they may be obtained directly from the bucket at the surface. The following sections describe various techniques for safely collecting representative soil samples with the aid of a backhoe.

The depth measurement for the sample begins at the top of the soil horizon.

8.2 Scoop-and-Bracket Method

If a sample interval is targeted from the surface, it can be sampled using a stainless steel scoop and bracket. First a scoop and bracket are affixed to a length of conduit and is lowered into the backhoe pit. The first step is to take the scoop and scrape away the soil comprising the surface of the excavated wall. This material likely represents soil that has been smeared by the backhoe bucket from adjacent material. After the smeared material has been scraped off, the original stainless steel scoop is removed and a clean stainless steel scoop is placed on the bracket. The clean scoop can then be used to remove sufficient volume of soil from the excavation wall to make up the required sample volume.

8.3 Direct-from-Bucket Method

It is also possible to collect soil samples directly from the backhoe bucket at the surface. Some precision with respect to actual depth or location may be lost with this method but if the soil to be sampled is uniquely distinguishable from the adjacent or nearby soils, it may be possible to characterize the material as to location and depth. In order to ensure representativeness, it is also advisable to dress the surface to be sampled by scraping off any smeared material that may cross-contaminate the sample.

8.4 Special Considerations When Sampling with a Backhoe

- Do not physically enter backhoe excavations to collect a sample. Use either procedure 8.2, Scoop-and-Bracket Method, or procedure 8.3, Direct-from-Bucket Method to obtain soil for sampling.

- Smearing is an important issue when sampling with a backhoe. Measures must be taken, such as dressing the surfaces to be sampled (see Section 2.3), to mitigate problems with smearing.
- Paint, grease and rust must be removed and the bucket decontaminated prior to sample collection.
- Observe precautions for volatile organic compound and PFAS sample collection found in Section 3.

9 Incremental Sampling Method

9.1 General

ISM is a structured composite sampling and processing protocol that reduces data variability and provides an unbiased estimate of mean contaminant concentrations in the area targeted for sampling. ISM provides representative samples of specific soil volumes defined as decision units (DUs) by collecting numerous increments of soil (typically 30–100) that are combined, processed, and subsampled according to specific protocols. Triplicate samples are collected to measure and evaluate the reproducibility of the sample data.

Like all sampling approaches, ISM should be applied within a systematic planning framework. The size, orientation, and location of a DU is site-specific and represents the smallest volume of soil about which a decision is to be made (USEPA 1999, Ramsey and Hewitt 2005, HDOH 2008a, ADEC 2009). DUs are based on project-specific needs and site-specific DQOs. More detailed information on conducting sampling using ISM can be found in the Interstate Technology and Regulatory Council’s *Incremental Sampling Methodology* (ISM-1).

9.2 Field Implementation, Sample Collection, and Processing

9.2.1 Introduction

The goal of most sampling efforts is to collect a sample that is representative of the target area (or DU). ISM is designed to collect representative and reproducible soil data. To help ensure data quality, all field sampling and field processing activities should be performed and supervised by personnel trained in ISM implementation

9.2.2 Sampling Tools

The selection of the appropriate sampling tool for collecting an ISM sample depends on the cohesiveness and composition of the soil substrate. The sampling tool should obtain cylindrical or core-shaped increments of a constant depth from the presented surface so that each increment collected is the same approximate volume and mass.

See Figures 1 and 2 for examples of sampling tools for nonvolatile ISM sample collection. Various other hand augers, core sampling tools, step probes, etc., are available from environmental or agricultural suppliers and are applicable to ISM if the specifications meet project DQOs. It is highly recommended that the proposed sampling tool is tested at the sample location prior to full mobilization to ensure that the sampling tool is appropriate for site conditions. If a pilot sampling effort is not possible, a variety of tools to address different soil types or site conditions should be taken into the field.

Note: Volatile ISM sample collection should follow Method 5035 recommendations. See Section 3 of this SOP.

9.2.3 Field Collection

Incremental soil samples are prepared by collecting multiple increments of soil (typically 30 or more) from a specified DU and physically combining these increments into a single sample, referred to as the “incremental sample.” Samples are collected in triplicate from different locations within the same DU. Sample increments locations can be selected by a random number generator or evenly spaced across the DU to ensure that the incremental sample is representative of the DU. Survey flags or other markers can be helpful in identifying increment collection locations prior to beginning sample location.

The number of increments to be collected from each DU of a site investigation should be evaluated during systematic planning as part of the DQO process and documented in the sampling and analysis plan (SAP). See section 5.3.2 of ISM-1 for subsurface ISM sample collection.

9.2.4 Field Handling of ISM Samples

ISM samples collect a larger volume of soil than discrete samples and will require a larger collection container than may be specified by the laboratory or that is typically used. For example, a gallon-sized sealable plastic bag or a liter glass jar may be used depending upon the suspect analytes. When building the incremental sample by collecting increments, it may be more practical to collect the sample in an aluminum pan, plastic bucket, stainless-steel bowl, or other easily transported

container until the entire sample has been collected. The final sample can then be processed in the field or transferred to a container for shipment to a laboratory for sample processing and analysis.

Processing of ISM samples is ideally performed in a laboratory. However, subsampling, disaggregation, drying, and sieving are some processing steps that may be required to be performed in the field. Field processing may be necessary if field analysis will be performed on the samples or if the laboratory is unable to perform the sample processing steps required. Any field processing steps should be rigorously performed to ensure that the sample representativeness is maintained through analysis. To ensure proper sample size reduction and representative subsampling, they should be performed using a 2-D Japanese slab cake and specialized subsampling tool, a riffle splitter, rotary cone sample splitter, or similar. Sample volume reduction of ISM samples should not be conducted with a stainless-steel spoon and a stainless-steel bowl. All sample processing equipment should be appropriately decontaminated between sample stations.

9.3 Special Considerations When Using Incremental Sampling Methods

- Selection of an appropriately sized and positioned Decision Unit is important to ensuring quality data and useful results
- Steps should be taken throughout the sampling effort to ensure that the representativeness of the sample is maintained from collection through analysis
- Advance coordination with the laboratory is necessary to ensure that the laboratory has the capability and capacity to conduct any sample processing that may be necessary. If the lab cannot complete the required processing steps, the sampling team may need to perform the sample processing steps in the field.

Figure 1



Figure 2



10 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version

LSASD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

LSASD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, Most Recent Version

LSASD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

LSASD Operating Procedure for Field X-Ray Fluorescence (XRF) Measurement, SESDPROC-107, Most Recent Version

LSASD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

LSASD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version

Title 49 Code of Federal Regulations, Pts. 171 to 179, Most Recent Version

US EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846, Most Recent Version (Method 5035)

US EPA Region 4 Safety and Occupational Health Manual. Region 4 LSASD, Athens, GA, Most Recent Version

ITRC (Interstate Technology & Regulatory Council). 2012. Incremental Sampling Methodology. ISM-1. Washington, D.C.: Interstate Technology & Regulatory Council, Incremental Sampling Methodology Team. www.itreweb.org.

ITRC (Interstate Technology and Regulatory Council) April 2020 Fact Sheets: *Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances (PFAS)*, and *Environmental Fate and Transport for Per- and Polyfluoroalkyl Substances*

11 Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the LSASD Quality Assurance Coordinator (QAC) on the LSASD local area network (LAN).

History	Effective Date
Replaced Chief with Supervisor; General formatting changes.	April 22, 2023
<p>LSASDPROC-300-R4, <i>Soil Sampling</i>, replaces SESDPROC-300-R3. Added Section 3.3. Soil Samples Collected for PFAS Analysis.</p> <p>Added Section 9, Incremental Sampling Method including Figures 1 and 2.</p> <p>General: Throughout the document, mention of SESD was replaced with LSASD as appropriate. Mention of Document Control Coordinator changed to Quality Assurance Coordinator.</p> <p>Cover Page: Changed Kevin Simmons, Environmental Scientist to Life Scientist. Changed Acting Supervisor, John Deatruck of the Enforcement and Investigations Branch to Supervisor, Applied Science Branch. Changed Acting Supervisor, Laura Ackerman, Ecological Assessment Branch to Supervisor, Hunter Johnson, Superfund Section. Changed Bobby Lewis, Field Quality Manager, Science and Ecosystem Support Division to Stacie Masters, Quality Assurance Coordinator, Laboratory Services and Applied Science Division.</p>	June 11, 2020
<p>SESDPROC-300-R3, <i>Soil Sampling</i>, replaces SESDPROC-300-R2.</p> <p>General: Corrected any typographical, grammatical and/or editorial errors.</p> <p>Title Page: Updated the author from Fred Sloan to Kevin Simmons. Updated the Enforcement and Investigations Branch Supervisor from Archie Lee to Acting Supervisor, John Deatruck.</p> <p>Section 1.5.1: Added “The reader should” to last sentence of the paragraph.</p> <p>Section 1.5.2: Omitted “When sampling in landscaped areas,” from first sentence of eighth bullet.</p> <p>Section 3.2.4: In the first paragraph, first sentence, added “(rapidly form bubbles).” Omitted “(rapidly form bubbles)” from second paragraph, second sentence.</p> <p>Any reference to “Percent Moisture and Preservation Compatibility (MOICA)” or “Percent Moisture” was changed to “Percent Solids”, both in the text and in Table 1.</p>	August 21, 2014

SESDPROC-300-R2, <i>Soil Sampling</i> , replaces SESDPROC-300-R1.	December 20, 2011
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SESDPROC-300-R1, <i>Soil Sampling</i> , replaces SESDPROC-300-R0.	November 1, 2007
SESDPROC-300-R0, Soil Sampling, Original Issue	February 05, 2007

**Region 4
U.S. Environmental Protection Agency
Laboratory Services & Applied Science Division
Athens, Georgia**

Operating Procedure

Title: Surface Water Sampling	ID: LSASDPROC-201-R6
Issuing Authority: Field Services Branch Supervisor	
Effective Date: April 22, 2023	Review Due Date: December 22, 2025
Method Reference: N/A	SOP Author: Kevin Simmons

Purpose

This document describes general and specific procedures, methods, and considerations to be used and observed when collecting surface water samples for field screening or laboratory analysis.

Scope/Application

The procedures contained in this document are to be used by field personnel when collecting and handling surface water samples in the field. On the occasion that LSASD field personnel determine that any of the procedures described in this section are either inappropriate, inadequate, or impractical and that another procedure must be used to obtain a surface water sample, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

Note: LSASD is currently migrating to a paperless organization. As a result, this SOP will allow for the use of electronic logbooks, checklists, and report forms as they are developed, which will also be housed in the LIMS and traceable to each project. LSASD is committed to maintaining its quality system by continued traceability of original observations in the final report as migration to an electronic system occurs.

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1. General Information

1.1. Purpose

- 1.1.1. This document describes general and specific procedures, methods, and considerations to be used and observed when collecting surface water samples for field screening or laboratory analysis.

1.2. Scope/Application

- 1.2.1. The procedures contained in this document are to be used by field personnel when collecting and handling surface water samples in the field. On the occasion that LSASD field personnel determine that any of the procedures described in this section are either inappropriate, inadequate, or impractical and that another procedure must be used to obtain a surface water sample, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3. Documentation/Verification

- 1.3.1. This procedure was prepared by persons deemed technically competent by LSASD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed by a subject matter expert. The official copy of this procedure resides on the LSASD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on LAN and for maintaining records of review conducted prior to its issuance.

1.4. General Precautions

1.4.1. Safety

- 1.4.1.1. Proper safety precautions must be observed when collecting surface water samples. Refer to the Region 4 Safety Manual and any pertinent site-specific Health and Safety Plans (HASP) or Job Hazard Assessments (JHAs) for guidelines on safety precautions. These guidelines should be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.4.2. Procedural Precautions

- 1.4.2.1. The following precautions should be considered when collecting surface water samples.

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Documentation of field sampling is done in a bound logbook.
- Completed chain-of-custody documents shall remain with the samples until custody is relinquished whether by common carrier (UPS, FedEx, etc.) or in-person delivery.
- All shipping documents, such as air bills, bills of lading, etc., shall be retained by the project leader and stored in a secure place until confirmation of sample delivery is received.

2. Special Sampling Considerations

2.1. Volatile Organic Compounds (VOC) Analysis

2.1.1. Surface water samples for VOC analysis must be collected in 40 ml glass vials with Teflon® septa. The vial may be either preserved with concentrated hydrochloric acid or left unpreserved. Preserved samples have a two-week holding time, whereas unpreserved samples have only a seven-day holding time. In the great majority of cases, preserved vials are used to take advantage of the extended holding time. In some situations, however, it may be necessary to use the unpreserved vials. For example, if the surface water sample contains a high concentration of dissolved calcium carbonate, there may be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles which will render the sample unacceptable. In this case, unpreserved vials should be used, and arrangements must be confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

2.1.2. The samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a reverse or convex meniscus at the top of the vial and absolutely no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm of one hand to see if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be topped off using a minimal amount of sample to re-establish the meniscus. Care should be taken not to flush any

preservative out of the vial during topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained, and the sample re-collected.

2.1.3. Samples for VOC analysis must be collected using either stainless steel or Teflon[®] equipment.

2.2. Special Precautions for Surface Water Sampling

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled, and the gloves should be donned immediately prior to sampling. The gloves should not contact the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers of material suspected of containing high concentrations of contaminants shall be stored separately from samples suspected of being lower concentration.
- All background or control samples shall be collected and placed in separate coolers or shipping containers whenever possible. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area when possible. Samples of waste or highly contaminated media must not be placed in the same cooler as environmental (i.e., containing low contaminant levels) or background samples.
- If possible, one member of the field sampling team should take all the notes and photographs, etc., while the other members collect the samples.
- Samplers must use new, verified and certified-clean disposable or non-disposable equipment cleaned according to procedures contained in LSASD Operating Procedure for Field Equipment Cleaning and Decontamination, LSASDPROC-205, or LSASD Operating Procedure for Field Cleaning and Decontamination at the FEC, LSASDPROC-206, for collection of samples for trace metals or organic compound analyses.

2.3. Sample Handling and Preservation Requirements

- Surface water samples will typically be collected either by directly filling the container from the surface water body being sampled or by decanting the water from a collection device such as a stainless-steel scoop or other device.
- During sample collection, if transferring the sample from a collection device, make sure that the device does not contact the sample containers.
- Place the sample into appropriate, labeled containers. Samples collected for VOC analysis must not have any headspace (see Section 2.1, Volatile Organic Compounds (VOC) Analysis). All other sample containers must be filled with an allowance for ullage.
- All samples requiring preservation must be preserved as soon as practically possible, ideally immediately at the time of sample collection. If preserved VOC vials are used, these will be preserved with concentrated hydrochloric acid by

LSB personnel prior to departure for the field investigation. For all other chemical preservatives, LSASD will use the appropriate chemical preservative generally stored in an individual single-use vial as described in the LSASD Operating Procedure for Field Sampling Quality Control (LSASDPROC-011). The adequacy of sample preservation will be checked after the addition of the preservative for all samples, except for the samples collected for VOC analysis. If it is determined that a sample is not adequately preserved, additional preservative should be added to achieve adequate preservation, but not more than two times the normal amount to avoid dilution of the sample. Preservation requirements for surface water samples are found in the USEPA Region 4 Laboratory Services Branch Laboratory Operations and Quality Assurance Manual (LSBLOQAM).

- All samples preserved using a pH adjustment (except VOCs) must be checked, using pH strips, to ensure that they were adequately preserved. This is done by pouring a small volume of sample over the strip. Do not place the strip in the sample. Samples requiring reduced temperature storage should be placed on ice immediately.

2.4. Quality Control

2.4.1. If possible, a control sample should be collected from a location not affected by the possible contaminants of concern and submitted with the other samples. In streams or other bodies of moving water, the control sample should be collected upstream of the sampled area. For impounded bodies of water, particularly small lakes, or ponds, it may be difficult or inappropriate to obtain an unbiased control from the same body of water from which the samples are collected. In these cases, it may be appropriate to collect a background sample from a similar impoundment located near the sampled body of water if there is a reasonable certainty that the background location has not been impacted. Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary, to document that low-level contaminants were not introduced by pumps, bailers, or other sampling equipment.

2.5. Records

2.5.1. Information generated or obtained by LSASD personnel will be organized and accounted for in accordance with LSASD records management procedures found in SESD Operating Procedure for Control of Records, LSASDPROC-1001. Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation in accordance with LSASD Operating Procedure for Logbooks, LSASDPROC-1002 and LSASD Operating Procedure for Sample and Evidence Management, LSASDPROC-005.

3. General Considerations

3.1. General

3.1.1. The surface water sampling techniques and equipment described in the following sections of this procedure are designed to minimize effects on the chemical and physical integrity of the sample. If the procedures in these sections are followed, a representative sample of the surface water should be obtained.

3.2. Equipment Selection Considerations

3.2.1. The physical location of the investigator when collecting a sample may dictate the equipment to be used. If surface water samples are required, direct dipping of the sample container into the stream is desirable. Collecting samples in this manner is possible when sampling from accessible locations such as stream banks or by wading or from low platforms, such as small boats or piers. Wading or streamside sampling from banks, however, may cause the re-suspension of bottom deposits and bias the sample. Wading is acceptable if the stream has a noticeable current (is not impounded), and the samples are collected while facing upstream. If the stream is too deep to wade, or if the sample must be collected from more than one water depth, or if the sample must be collected from an elevated platform (bridge, pier, etc.), supplemental sampling equipment must be used.

3.2.2. To collect a surface water sample from a water body or other surface water conveyance, a variety of methods can be used:

- Dipping Using Sample Container
- Scoops
- Peristaltic Pumps
- Discrete Depth Samplers
- Bailers
- Buckets
- Submersible Pumps
- Automatic Samplers

3.2.3. Regardless of the method used, precautions should be taken to ensure that the sample collected is representative of the water body or conveyance. These methods are discussed in the following sections.

3.3. Dipping Using Sample Container

3.3.1. A sample may be collected directly into the sample container when the surface water source is accessible by wading or other means. The sampler should face upstream if there is a current and collect the sample without disturbing the bottom sediment. The surface water sample should always be collected prior to the collection of a sediment sample at the same location. The sampler should be careful not to displace the preservative from a pre-preserved sample container, such as the 40-ml VOC vial.

3.4. Scoops

3.4.1. Stainless steel scoops provide a means of collecting surface water samples from surface water bodies that are too deep to access by wading. They have a limited reach of about eight feet and, if samples from distances too far to access using this method are needed, a mobile platform, such as a boat, may be required.

3.4.2. Stainless steel scoops are useful for reaching out into a body of water to collect a surface water sample. The scoop may be used directly to collect and transfer a surface water sample to the sample container, or it may be attached to an extension to access the selected sampling location.

3.5. Peristaltic Pumps

3.5.1. Another device that can be effectively used to sample a water column, such as a shallow pond or stream, is the peristaltic pump/vacuum jug system. The peristaltic pump can be used to collect a water sample from any depth if the pump is located at or near the surface water elevation. There is no suction limit for these applications. The use of a metal conduit to which the tubing is attached, allows for the collection of a vertical sample (to about a 25-foot depth) which is representative of the water column. The tubing intake is positioned in the water column at the desired depth by means of the conduit. Using this method, discrete samples may be collected by positioning the tubing intake at one depth or a vertical composite may be collected by moving the tubing intake at a constant rate vertically up and down the water column over the interval to be composited.

3.5.2. Samples for VOC analysis cannot be collected directly from the peristaltic pump discharge or from the vacuum jug. If a peristaltic pump is used for sample collection and VOC analysis is required, the VOC sample must be collected using one of the “soda straw” variations. Ideally, the tubing intake will be placed at the depth from which the sample is to be collected and the pump will be run for several minutes to fill the tubing with water representative of that interval. After several minutes, the pump is turned off and the tubing string is retrieved. The pump speed is then reduced to a slow pumping rate and the pump direction is reversed. After turning the pump back on, the sample stream is collected into the VOC vials as it is pushed

from the tubing by the pump. Care must be taken to prevent any water that was in contact with the silastic pump head tubing from being incorporated into the sample.

3.6. Discrete Depth Samplers

3.6.1. When discrete samples are desired from a specific depth, and the parameters to be measured do not require a Teflon[®]-coated sampler, a standard Kemmerer or Van Dorn sampler may be used. The Kemmerer sampler is a brass cylinder with rubber stoppers that leave the ends of the sampler open while being lowered in a vertical position, thus allowing free passage of water through the cylinder. The Van Dorn sampler is plastic and is lowered in a horizontal position. In each case, a messenger is sent down a rope when the sampler is at the designated depth, to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill respective sample containers. With a rubber tube attached to the valve, dissolved oxygen sample bottles can be properly filled by allowing an overflow of the water being collected. With multiple depth samples, care should be taken not to disturb the bottom sediment, thus biasing the sample.

3.6.2. When metals and organic compounds parameters are of concern, then a double-check valve, stainless steel bailer or Kemmerer sampler should be used to collect the sample.

3.7. Bailers

3.7.1. Teflon[®] bailers may also be used for surface water sampling if the study objectives do not necessitate a sample from a discrete interval in the water column. A closed-top bailer with a bottom check-valve is sufficient for many studies. As the bailer is lowered through the water column, water is continually displaced through the bailer until the desired depth is reached, at which point the bailer is retrieved. This technique may not be successful where strong currents are found.

3.8. Buckets

3.8.1. A plastic bucket can be used to collect samples for measurement of water quality parameters such as pH, temperature, and conductivity. Samples collected for analysis of classical water quality parameters including but not limited to ammonia, nitrate-nitrite, phosphorus, and total organic carbon may also be collected with a bucket. Typically, a bucket is used to collect a sample when the water depth is too great for wading, it is not possible to deploy a boat, or access is not possible (excessive vegetation or steep embankments) and the water column is well mixed. The water body is usually accessed from a bridge. The bucket is normally lowered by rope over the side of the bridge. Upon retrieval, the water is poured into the appropriate sample containers

3.8.2. Caution should be exercised whenever working from a bridge. Appropriate measures should be taken to ensure the safety of sampling personnel from traffic hazards.

3.9. Submersible Pumps

3.9.1. Submersible pumps can be used to collect surface water samples directly into a sample container. The constituents of interest should be taken into consideration when choosing the type of submersible pump and tubing to be used. If trace contaminant sampling of extractable organic compounds and/or inorganic analytes will be conducted, the submersible pump and all its components should be constructed of inert materials such as stainless steel and Teflon[®]. The tubing should also be constructed of Teflon[®]. If re-using the same pump between sample locations, the pump should be decontaminated using LSASD Operating Procedure for Field Equipment Cleaning and Decontamination, (LSASDPROC-205). New tubing should be used at each sample location.

3.9.2. If the samples will be analyzed for classical parameters such as ammonia, nitrate-nitrite, phosphorus, or total organic carbon, the pump and tubing may be constructed of components other than stainless steel and Teflon[®]. The same pump and tubing may be re-used at each sampling station after rinsing with deionized water and then purging several volumes of sample water through the pump and tubing prior to filling the sample containers.

3.9.3. Either a grab or composite sample can be collected using a submersible pump. A composite sample can be collected by raising and lowering the pump throughout the water column. If a composite sample is collected, it may be necessary to pump the sample into a compositing vessel for mixing prior to dispensing into the sample containers. If a compositing vessel is required, it should be constructed of materials compatible with the constituents of concern and decontaminated between sample stations according to appropriate procedures, again depending on the constituents of concern.

3.10. Automatic Samplers

3.10.1. Where unattended sampling is required (e.g., storm-event sampling, time-of-travel studies) an automatic sampler may be used. The automatic sampling device may be used to collect grab samples based on time, in-stream flow or water level or used to collect composite samples as dictated by the study data needs. The automatic sampling device should be calibrated prior to deployment to ensure the proper volume is collected. The manufacturer's instruction manual should be consulted for automatic sampler operation.

3.11. Trace-Level Mercury Sampling

- 3.11.1. To prevent contamination during sample collection, Region 4 has developed this sampling procedure for trace-level mercury analysis (< 1 part per trillion). This procedure is based on EPA Method 1669.
- 3.11.2. A vacuum chamber assembly is utilized to collect surface water samples for trace-level mercury analyses. The vacuum chamber assembly consists of the following: 1) an airtight acrylic, cylindrical chamber with an O-ring sealed lid to hold the sample bottle, 2) a Teflon[®] sample tubing that connects to a centered Teflon[®] compression fitting on top of the chamber. The other end of the tubing passes through a rigid Teflon[®] pole for stability and has a modified magnetic screen holder at the intake, and a hand vacuum pump. The chamber is designed to hold a 2-liter sample bottle; however, smaller sample containers may be utilized with a spacer inserted into the chamber. A 2-inch square of 100 µm Nitex[®] screen is used on the magnetic screen holder at the intake to prevent large pieces of debris from entering the sample. The screen does not prevent the passage of particulate organic matter which is often prevalent in surface water. The vacuum chamber has a second off-center compression fitting with a 4-inch piece of Teflon[®] tubing inserted in the fitting. A piece of clear Tygon[®] tubing approximately 18-24 inches long is placed over the small piece of Teflon[®]. The Teflon[®] adds stability to the tubing and keeps it from crimping. The Tygon[®] is attached to the hand pump and the chamber with electrical tape. The Nitex[®] screen intake is inserted into the water to be sampled and a vacuum is pulled on the chamber by means of the hand vacuum pump, thus drawing a water sample into a sample container placed directly beneath the intake tubing within the chamber.
- 3.11.3. Teflon[®] bottles or 300-Series glass bottles with single use Teflon[®]-lined caps may be used for sample collection. All sample containers used for collection of trace-level mercury water samples must be pre-cleaned in a laboratory as described in EPA Method 1631. Teflon[®] containers should also be etched on the outside of the bottle with a unique identification number for QA purposes. All bottles for trace-level sampling must be double bagged in re-sealable bags. Water samples collected for total, inorganic, methyl, or ethyl mercury analyses are pumped into appropriately cleaned bottles. Preservation should be done in a clean room laboratory that has been specifically prepared for the preparation of trace level samples (positive pressure ventilation, sticky floor mats, etc.). Preservation must occur within 48 hours of sample collection, sooner if possible. Region 4 utilizes laboratory preservation of trace-level mercury samples to minimize the potential for contamination, and if split samples are required, they must be split in a trace-level clean room laboratory.

3.11.4. The following quality assurance/quality control (QA/QC) samples are collected in conjunction with low-level mercury samples:

- bottle blanks
- equipment blanks
- air deposition blanks
- trip blanks
- duplicates and
- splits

3.11.5. A bottle blank is prepared in the lab with reagent-grade water to ensure the cleanliness of the bottles prior to use in the field. After decontamination of the Teflon® tubing by pumping and discarding several sample container volumes of reagent-grade water through the tubing, (using the same amount of water used for sample collection in the field) an equipment blank sample is collected into an appropriately pre-cleaned sample container. Equipment blanks are collected at the beginning of each field trip and at the end of each day. The bottle blank and the equipment blank do not go out into the field and are preserved at the end of the day with the regular field samples.

3.11.6. Air deposition blanks are collected to determine if airborne mercury is present at the time of sample collection. The air deposition blanks consist of a pre-cleaned mercury sample container, filled with reagent-grade water by the laboratory that prepared the containers, and is shipped with the containers to the field. The air deposition blank is uncapped using “clean hands”/ “dirty hands” procedures (see below) and set near the sampling location throughout the duration of the mercury sample collection for that particular station. Once the mercury sample is collected, the air deposition blank is recapped and handled and processed with the other mercury samples. One air deposition blank is collected each day by each field crew unless atmospheric conditions or site conditions warrant additional blanks.

3.11.7. Trip blanks are utilized to determine if any contaminants of interest to the study are potentially introduced to the samples during storage and transport to the laboratory. Trip blanks are prepared by the laboratory which supplies the mercury sample containers. The trip blanks consist of cleaned bottles which are filled with reagent-grade water by the laboratory and shipped with the other clean sample containers. A dark plastic bag is placed in each cooler that will hold the trace-level water samples. One trip blank is placed in each trace-level cooler of samples and returned to the laboratory with the ambient trace-level water samples. All trace-level samples should be kept in the dark until they are preserved. The trip blanks are never opened in the field. Trip blanks are preserved in the clean room.

- 3.11.7.1. Duplicate samples are discrete samples collected at the same site and time to measure variability of collected samples and to assess sample collection consistency. Sample splits are aliquots of a minimum 500 ml poured from a single ambient sample. They must be split in a trace-level clean room laboratory.
- 3.11.7.2. To prevent cross contamination in samples analyzed for trace-level mercury in ambient surface waters, clean sampling protocols must be employed throughout the sampling effort. For each sampling event, one sampling team member is designated as “clean hands” and one as “dirty hands” (see below). All operations involving contact with the sample bottle and transfer of the sample from the sample collection device to the sample bottle are handled by the individual designated as “clean hands.” “Dirty hands” is responsible for preparation of the sampling device (except the sample container) and for all other activities that do not involve direct contact with the sample.
- 3.11.7.3. Prior to sample collection with the vacuum chamber assembly, the Teflon[®] line is cleaned at each station by rinsing with ambient water as follows: A 2-liter poly bottle is placed into the chamber and filled half full of ambient water. The bottle is swirled to rinse it and the water is discarded downstream of sampling area. The same 2-liter poly bottle can be used at each station. Additional cleaning measures are not recommended if the chamber assembly is only used to collect ambient surface water samples. Detergent washes and acid rinses are not conducted due to potential mercury contamination from these solutions. If applicable, samples for other analyses can be collected in a poly bottle with the vacuum chamber assembly but should be collected before the trace-level sample as an additional means of flushing the sampling line prior to collection of the trace-level samples. It is not necessary to implement the “clean hands”/ “dirty hands” method for collection of non-mercury samples, but latex or vinyl gloves should be worn when any samples are collected.
- 3.11.7.4. Following are procedures for cleaning the vacuum chamber tubing and collection of ancillary water quality samples, if applicable:
1. Carefully approach the sampling station from downstream and downwind if possible.
 2. While wearing latex or vinyl gloves, place an uncapped 2-liter poly bottle into the chamber and secure the chamber lid by attaching the spring-loaded clamps.
 3. Place a new square of 100 µm Nitex[®] screen in the magnetic screen holder. Place the intake beneath the surface of the water (mid-depth or six inches, whichever is less) and hold firmly in place. Care should be taken not to disturb sediment particles in very shallow waters (< 4 inches deep).

4. Squeeze the hand pump until liquid starts to fill the bottle in the chamber. When the bottle is approximately half full, release the vacuum on the chamber, remove the bottle, swirl the contents, and discard the water downstream. Repeat this rinse. If ancillary water quality samples are to be collected, return the 2-liter poly bottle to the chamber, and pump the required volume of water to fill the appropriate ancillary sample containers. Remove the 2-liter bottle from the chamber and cap. Fill the ancillary sample bottles upon completion of the mercury sample collection.
- 3.11.8. Water samples for trace level mercury analyses should be collected immediately after the ancillary water samples have been collected according to the following procedures:
1. “Clean hands” should put on a pair of latex or vinyl gloves, then a pair of shoulder length polyethylene gloves.
 2. “Dirty hands” should put on a pair of latex or vinyl gloves, retrieve the double bagged trace level sample bottle from the cooler, and open the outer bag. “Clean hands” should open the inner bag and remove the pre-cleaned Teflon[®] or glass bottle.
 3. “Dirty hands” should open the lid on the chamber. “Clean hands” should place the sample bottle in the chamber, remove the bottle top and place it inside the chamber with the bottle.
 4. “Dirty hands” should close and secure the chamber lid and using the hand pump, fill the container. The sample container should be filled to overflowing. “Dirty hands” should then release the vacuum and open the lid on the chamber.
 5. “Clean hands” should place the top on the sample bottle, remove it from the chamber and place it in the inner bag and seal the bag. “Dirty hands” should seal the outer bag and place the sample in the black bag in the dark cooler. Only coolers dedicated to storage and transport of trace-level mercury samples should be used.

3.12. Per- and Polyfluoroalkyl Substances (PFAS) Sampling

- 3.12.1. The persistence and mobility of some PFAS, combined with decades of widespread use in industrial processes, certain types of firefighting foams, and consumer products, have resulted in their being present in most environmental media at trace levels across the globe (ITRC 2020). Both consumer and industrial wastewater are potential sources for PFAS-containing discharges into municipal and industrial WWTPs. Conventional wastewater treatment methods are not effective in removing PFAS and thus may be a major source of PFAS discharge into surface

waters (ITRC 2020). Additionally, nonpoint storm water sources and groundwater seeps contribute to PFAS detected in surface water. For more information about conducting site investigations for PFAS, please see the Interstate Technology and Regulatory Council's (ITRC's) August 2020 Fact Sheets: Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances (PFAS), and Environmental Fate and Transport for Per- and Polyfluoroalkyl Substances.

- 3.12.2. A Sampling and Analysis Plan or site-specific Quality Assurance Project Plan with data quality objectives (DQOs) is necessary for any PFAS surface water investigation. The DQOs should state the desired sample locations as well as the desired depth in the water column considering the potential for stratification of PFAS in solution and their tendency to accumulate at the air/water interface (ITRC 2020). For samples undergoing PFAS analyses, guidance documents recommend sampling equipment be made of high-density polyethylene (HDPE), polypropylene, stainless steel and/or silicone (USEPA 2019). Because studies have shown loss of PFAS due to adsorption to surfaces, when possible, the sample should be collected directly into the appropriate sample container. When the sample container contains a dechlorination agent such as Trizma[®], then a sampling device may be used to collect the sample before transferring it into the container with the dechlorination agent. If the surface water to be sampled cannot be physically reached, an intermediate collection device may be used, such as a stainless-steel scoop, or large stainless-steel spoon. Additionally, samples may be collected with a peristaltic pump using HDPE and silicone tubing.
- 3.12.3. To prevent PFAS contamination, extreme care is required when handling containers, samples and equipment that will be used to collect samples for PFAS analyses (See Trace Level Sampling Technique for PFAS in Section 4.2 of Waste Sampling Operating Procedure, LSASDPROC-302-R4). New gloves need to be worn when decontaminating and handling sample containers and equipment. When worn gloves become compromised by potential PFAS containing materials, they need to be changed for new gloves. Nitrile gloves are recommended for PFAS sampling investigations. Also, sample containers should be kept covered in original packaging or in Whirl Paks[®] until ready for use.
- 3.12.4. For surface water samples undergoing PFAS analyses, it is extremely important that quality control samples be collected as part of the investigation to demonstrate the PFAS contribution of the sample containers, decontamination solutions, gloves, decontaminated equipment, and plastic used to store equipment. It is also important to take field quality control samples such as additional equipment blanks, material blanks, field blanks, duplicates, and trip blanks to evaluate the sample collection and sample handling activities of the investigation. Field blanks, where samplers transfer PFAS-free water from one container into an empty sample container, help assess the PFAS impact of the samplers and sample handling process as well as the airborne PFAS conditions during the sampling event.

References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version

LSASD Operating Procedure for Control of Records, LSASDPROC-1001, Most Recent Version

LSASD Operating Procedure for Sample and Evidence Management, LSASDPROC-005, Most Recent Version

LSASD Operating Procedure for Logbooks, LSASDPROC-1002, Most Recent Version

LSASD Operating Procedure for Field Sampling Quality Control, LSASDPROC-011, Most Recent Version

LSASD Operating Procedure for Field pH Measurement, LSASDPROC-100, Most Recent Version

LSASD Operating Procedure for Field Specific Conductance Measurement, LSASDPROC-101, Most Recent Version

SESD Operating Procedure for Field Temperature Measurement, SESDPROC-102, Most Recent Version

LSASD Operating Procedure for Field Turbidity Measurement, LSASDPROC-103, Most Recent Version

LSASD Operating Procedure for Measurement of Dissolved Oxygen, LSASDPROC-106, Most Recent Version

LSASD Operating Procedure for Equipment Inventory and Management, LSASDPROC-1009, Most Recent Version

SESD Operating Procedure for In-Situ Water Quality Monitoring, SESDPROC-111, Most Recent Version

SESD Operating Procedure of Oxidation Reduction Potential (ORP), SESDPROC-113, Most Recent Version

LSASD Operating Procedure for Field Equipment Cleaning and Decontamination, LSASDPROC-205, Most Recent Version

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LSASD Operating Procedure for Packaging, Marking, Labeling and Shipping of Environmental and Waste Samples, LSASDPROC-209, Most Recent Version

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United States Environmental Protection Agency (US EPA). 1981. "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), April 13, 1981.

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US EPA. Laboratory Services Branch Laboratory Operations and Quality Assurance Manual. Region 4 SESD, Athens, GA, Most Recent Version

US EPA, Region 4. Safety & Occupational Health SharePoint Site:
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LSASD Operating Procedure for Field Sampling Quality Control, LSASDPROC-011, Most Recent Version

Interstate Technology and Regulatory Council (ITRC) August 2020 Fact Sheets, Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances (PFAS), and Environmental Fate and Transport for Per- and Polyfluoroalkyl Substances.

US EPA, Technical Brief Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS) Methods and guidance for sampling and analyzing water and other environmental media, EPA/600/F 1 - 17/022f, Updated June 2019.

Revision History

History	Effective Date
Replaced Chief with Supervisor; General formatting revisions.	April 22, 2023
<p>SESDPROC-201-R5, <i>Surface Water Sampling</i>, replaces SESDPROC-201-R4. The SOP was put in the new SOP format.</p> <p>General:</p> <p>New Title Block added. Changed SESD to LSASD throughout document. Updated SOP references throughout document. Minor editorial changes to make sentences clearer and more concise.</p> <p>Added Section 3.12 concerning Per- and Polyfluoroalkyl Substances (PFAS) Sampling.</p> <p>Added 2 references regarding PFAS Sampling.</p>	December 23, 2021
<p>SESDPROC-201-R4, <i>Surface Water Sampling</i>, replaces SESDPROC-201-R3.</p> <p>General: Corrected any typographical, grammatical, and/or editorial errors.</p> <p>Title Page: Chris Decker was omitted as an author. Updated cover page to represent SESD reorganization. John Deatruck was not listed as the Supervisor of the Field Services Branch</p>	December 16, 2016
SESDPROC-201-R3, <i>Surface Water Sampling</i> , replaces SESDPROC-201-R2.	February 28, 2013
SESDPROC-201-R2, <i>Surface Water Sampling</i> , replaces SESDPROC-201-R1.	January 16, 2013
SESDPROC-201-R1, <i>Surface Water Sampling</i> , replaces SESDPROC-201-R0.	November 1, 2007
SESDPROC-201-R0, <i>Surface Water Sampling</i> , Original Issue	February 05, 2007

Region 4
U.S. Environmental Protection Agency
Laboratory Services & Applied Science Division
Athens, Georgia

Operating Procedure

Title: Pore Water Sampling

ID: LSASDPROC-513-R5

Issuing Authority: Field Services Branch Supervisor

Effective Date: April 22, 2023

Review Due Date: May 12, 2024

Method Reference: N/A

SOP Author: Mel Parsons

Purpose

The purpose of this operating procedure is to describe the methods and considerations to be used when obtaining a pore water sample from soil or sediment.

Scope/Application

This document describes procedures generic to all pore water sampling methods to be used by field personnel when collecting and handling samples in the field. On the occasion that Laboratory Services and Applied Science Division (LSASD) personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to obtain a pore water sample, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

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1 General Information

1.1 Documentation/Verification

This procedure was prepared by persons deemed technically competent by LSASD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the LSASD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.2 General Precautions

1.2.1 Safety

Proper safety precautions must be observed when collecting pore water samples. Refer to the LSASD Safety, Health and Environmental Management Program Procedures and Policy Manual (most recent version) and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When using this procedure, minimize exposure to potential health hazards through the use of protective clothing, eye wear and gloves. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.2.2 Procedural Precautions

The following precautions should be considered when collecting pore water samples:

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Documentation of field sampling is done in a bound logbook. Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.

- All shipping documents, such as bills of lading, will be retained by the project leader and stored in a secure place.

1.2.3 Records

Information generated or obtained by LSASD personnel will be organized and accounted for in accordance with LSASD records management procedures found in LSASD Operating Procedure for Control of Records, LSASDPROC-002 (most recent version). Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation, in accordance with LSASD Operating Procedure for Logbooks, LSASDPROC-010 (most recent version), and LSASD Operating Procedure for Sample and Evidence Management, LSASDPROC-005 (most recent version).

2 Sampling Methodology

2.1 General

The pore water sampling techniques and equipment described in this procedure are designed to minimize effects on the chemical and physical integrity of the sample. If the procedures in this section are followed, a representative sample of the pore water should be obtained.

2.2 Collection Considerations

The physical location of the investigator when collecting a sample may dictate the equipment to be used. Wading is the preferred method for reaching the sampling location, particularly if the stream has a noticeable current (i.e., is not impounded). However, wading may disrupt bottom sediments causing biased results; therefore, the sampler should enter the area downstream of the sampling location and collect the sample facing upstream. If the stream is too deep to wade, the pore water sample may be collected from a platform such as a boat or by SCUBA diving. If sampling from a boat or in water deeper than the length of the sampler, extensions may be utilized. If SCUBA diving, all diving activity must be conducted in accordance with EPA's Diving Safety Manual, current version.

2.3 Summary of Procedure

Pore water is collected using a pore water extracting device (Figure 1). The most common type used by LSASD is the PushPoint™ sampler (M.H.E. Products 2003), made out of stainless-steel tubing. The sampling end of the pore water device is inserted into the sediment to the desired depth, and pore water is extracted using a syringe or peristaltic pump. The device is suitable for use only in fine-grained material (no gravel or cobble). Other similar devices may be used providing that the integrity of the sample is maintained, and no ambient surface water is allowed in contact with the sample.

2.4 Sampling Equipment

A PushPoint™ or similar sampler typically consists of a pointed tubular stainless-steel tube with a screened zone at one end and a sampling port at the other. The pointed end with the screened zone consists of a series of very fine interlaced machined slots to allow pore water to enter the sampler.

A removable guard rod adds rigidity to the sampler during sediment insertion. The length of the screened zone will depend on the site-specific study design. Depending on the data quality objectives (DQO) of the study, filters may be placed over the screened zone if additional screening is needed. Pore water is collected through the opposite end of the device by connecting flexible tubing and using a syringe or peristaltic pump to extract the sample. Teflon® tubing is the preferred tubing to be used for collecting pore water samples. However, other tubing may be used, depending upon the DQOs for the specific application.

There are many modifications that can be incorporated into the procedure to satisfy data quality objectives for a specific application. The procedures discussed in the following sections provide guidance on the basic operation of pore water sampling devices and issues to consider when collecting pore water.

An alternative system is available in LSASD inventory for use in soft sediments in water deeper than wading depth. A well screen and short riser approximately 3/4" in diameter are threaded to fasten to the bottom of a custom flange. Internal threads on the screen accept a tubing adapter. The accompanying rimmed flange has a coupling with both top and bottom threads. The well screen is screwed into the bottom of the flange and Teflon® tubing is attached to the tubing adapter threaded into the well screen. For deployment, the tubing is then inserted through a PVC pipe or well casing which is then screwed into the upper threads of the flange. The entire assembly can be deployed in water up to ten feet of depth from a well anchored boat.

2.5 Pore Water Sampler Deployment Considerations

It is critical in the collection of pore water to avoid surface water intrusion. Water will flow in a path of least resistance. If space is created around the sides of the sampling end of the pore water device during deployment, surface water may flow down the outside of the device to the screened area and into the intended sample. Therefore, the pore water device should be used with a sampling flange (Figure 2), especially when collecting pore water near the sediment-surface water interface. If pore water is collected from deep in sediments, a flange may not be necessary. When inserted through the flange, the body of the pore water device should form a watertight seal to eliminate surface water intrusion during sample collection. Flanges should include an outer vertical cutting ring to enhance sealing. Flange systems can be augmented by flexible plastic sheeting of appropriate material. The sheeting can be weighted to conform to a stream bottom by objects obtained from other areas of the stream away from the sampling location. Several of the flanges in LSASD inventory have a threaded nut and washer to facilitate sealing the flange to a polyethylene sheet.

The flange can be made of any material that will not cross contaminate the intended sample. If both inorganic and organic analyses are required, the flange should be made of inert material such as stainless steel or Teflon®. The size of the flange depends on the volume of pore water to be collected. If large volumes of pore water are to be collected, use a large flange size. A useful estimate can be made for planning by taking the required water volume, tripling it to assume 33% porosity, and then calculating the dimensions of a cylinder of this volume, based upon the penetration depth of the sampler. The flange should cover at least this estimated volume. If it is not practical to use a large flange, then multiple devices may be deployed, and smaller volumes can be collected from several devices for a composite sample. If multiple devices are deployed, they should be spaced an appropriate distance apart so they will not interfere with one another.

In general, the volume of pore water that can be collected at a given location is limited. Collecting large volumes of pore water will ultimately result in the collection of water from the overlying water body. Often, minimum required volumes must be negotiated with the laboratory to limit the volumes withdrawn.

Where significant differences in parameters such as pH or conductivity exist between the surface water and pore water, a check can be made at the end of sampling to assess whether surface water intrusion has occurred by measuring the pore water parameters at the beginning and conclusion of sampling. Fluorescent dye tracing can also be used for this purpose.

2.6 Pore Water Collection

The flange is first placed at the desired sampling point with the push-point removed to allow any water to escape from under the flange. The flange rim should be carefully worked into the soil or sediment until the flange is flush with the surface. The pore water device should then be inserted through the compression adapter on the flange and into the soil or sediment as carefully as possible (Figure 2). When the sampler is inserted to the desired depth, the compression adapter should be tightened. The push-point's guard rod can then be withdrawn. Do not reinsert the guard rod into the sampler for any reason until the sampler has been cleaned (particles rolled between the two metal surfaces will lock the parts together and permanently damage the sampler.)

When deploying the pore water device, care must be taken not to disturb the sampling area. If the sampler is wading, the sampler should lean out and insert the pore water device as far as possible away from where the sampler is standing to reduce potential effects of the sampler on the integrity of the pore water sample. Depth of penetration of the pore water device depends on the objectives of the specific investigation.

After the pore water device has been successfully deployed, attach the sample tubing to the sampling port of the pore water device. Short pieces of Silastic® tubing can be used to splice Teflon® sample tubing to a push-point sampler, taking care to butt the tubing to the sampler at the center of the splice. Then attach the other end of the tubing to a sample withdrawing device, such as a syringe or a peristaltic pump (according to LSASD Operating Procedure for Pump Operation, LSASDPROC-203). Before collecting a pore water sample, be sure to purge out all air and surface water from the pore water sampler and sample tubing with the appropriate amount of pore water. This step can be accomplished by calculating the volume of the sampler and attached tubing and pumping this volume plus an additional 10 percent of pore water through the sampler and tubing prior to collecting the sample. If utilizing a syringe for collection, a three-way valve with a side syringe must be utilized for the surface water purge in order not to cross contaminate the sampling syringe.

2.6.1 Peristaltic Pump/Vacuum Jar Collection

The peristaltic pump/vacuum jug can be used for sample collection of organic or inorganic samples because it allows for the sample to be collected without coming in contact with the pump head tubing, maintaining the integrity of the sample. This is accomplished by placing a Teflon® transfer cap assembly onto the neck of a pre-cleaned standard 1-liter amber glass container (Figure 3). Teflon® tubing (¼-inch O.D.) connects the container to both the pump and the sample source. The

pump creates a vacuum in the container, thereby drawing the sample into the container without it coming into contact with the pump head tubing.

Because the sample is exposed to a vacuum and is agitated as it enters the vacuum jug, this method cannot be used for collection of samples for volatile organic compounds. An alternative method for collecting volatile organics involves filling the Teflon® tubing with sample by running the pump for a short period of time. Once the tubing is full of water, the tubing is removed from the pore water sampler and, then pinched off at the pump in order to maintain the vacuum while it is being disconnected from the pump head tubing. The water is then allowed to carefully drain, by gravity, into the sample vials. Alternatively, without disconnecting the tubing from the pump head, the contained sample can be pushed out of the tubing, into the sample vials, by reversing the peristaltic pump at very low speed. Great care must still be taken with this method in order not to agitate the sample during the transfer process or to transfer water that has been in contact with the Silastic® tubing into the vials.

Because pore water is typically collected from an anaerobic environment, it is preferable, especially when collecting samples for nutrient analysis, to maintain the integrity of the sample by minimizing exposure to air. This can be accomplished by purging the sample container with an inert gas such as nitrogen or argon prior to sampling. In addition, if analyzing for nutrients or metals, the container can be pre-preserved in order to minimize exposure of the sample to ambient conditions.

An alternative, when collecting samples for metals, nutrients, or other sample analyses not affected by Silastic® tubing and when exposure to air is not a concern, is to collect the sample directly from the discharge of the pump head tubing after an adequate purge has been demonstrated. When collecting samples in this manner, there are several considerations of which to be aware. The pump head tubing (Silastic®, etc.) must be changed after each sample and a rinsate blank must be collected from a representative piece of the pump head tubing (only one blank per investigation). Also, precautions must be taken to ensure that the end of the discharge tubing is not allowed to touch the ground or other surface to ensure the integrity of samples collected in this manner.

2.6.2 Syringe

An alternative to using the pump and vacuum container is to use a syringe as the mechanism to draw the pore water through the sampling device. The tubing from the sampling port of the pore water device can be directly attached to a syringe with a three-way valve and a side syringe and the pore water sample can be manually withdrawn. The valve is first switched to the side syringe, which is used for purging air and any ambient surface water in the system prior to sampling. The volume to be purged is determined by the length and diameter of the sampling device and attached tubing. Once the sampler has been purged, the valve is switched to the sampling syringe and the sample is drawn into the syringe. The syringe can be used as the final sample container or the pore water can be transferred to another container, depending on project objectives and analytical requirements. This is the best method to use if the sample is to be collected underwater by SCUBA diving.

2.7 Quality Control

If possible, a control or background sample should be collected from a location not affected by the possible contaminants of concern and submitted with the other samples. In streams or other bodies of moving water, the control sample should be collected upstream of the sampled area. For impounded bodies of water, particularly small lakes or ponds, it may be difficult or inappropriate to obtain an unbiased control from the same body of water from which the samples are collected. In these cases, it may be appropriate to collect a background sample from a similar impoundment located near the sampled body of water if there is a reasonable certainty that the background location has not been impacted. Equipment blanks should be collected if equipment is field cleaned and reused on-site or, if necessary, to document that low-level contaminants were not introduced by pumps, bailers or other sampling equipment.

2.8 Specific Sampling Equipment Quality Assurance Techniques

All equipment used to collect pore water samples shall be cleaned as outlined in the LSASD Operating Procedure for Field Equipment Cleaning and Decontamination, LSASDPROC-205 (most recent version) or LSASD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, LSASDPROC-206 (most recent version) and repaired, if necessary, before being stored at the conclusion of field studies. Cleaning procedures utilized in the field or field repairs shall be thoroughly documented in field records.

3 Special Sampling Considerations

3.1 Volatile Organic Compounds (VOC)

Pore water samples for VOC analysis must be collected in 40 ml glass vials with Teflon® septa. The vial may be either preserved with concentrated hydrochloric acid or they may be unpreserved. Preserved samples have a two-week holding time, whereas, unpreserved samples have only a seven day holding time. During most sampling events, preserved vials are used due to their extended holding time. In some situations, however, it may be necessary to use unpreserved vials. For example, if the surface water sample contains a high concentration of dissolved calcium carbonate, there may be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles. This will render the sample unacceptable. In this case, unpreserved vials should be used, and arrangements must be confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

Samples for VOC analysis must be collected using either stainless steel or Teflon® equipment. Samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a meniscus at the top of the vial and absolutely no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm of one hand to see if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be refilled. Care should be taken not to flush any preservative out of the vial during topping off. If, after attempting to refill and cap the vial, bubbles are still present, a new vial should be obtained, and the sample should be re-collected.

3.2 Dissolved Metals Sample Collection

If a dissolved metals pore water sample is to be collected, an in-line filtration should be used. The use of disposable, high-capacity filter cartridges (barrel-type) or membrane filters in an in-line filter

apparatus is preferred. The high-capacity, barrel-type filter is preferred due to the higher surface area associated with this configuration.

Potential differences could result from variations in filtration procedures used to process water samples for the determination of trace element concentrations. A number of factors associated with filtration can substantially alter "dissolved" trace element concentrations; these include filter pore size, filter type, filter diameter, filtration method, volume of sample processed, suspended sediment concentration, suspended sediment grain-size distribution, concentration of colloids and colloidally-associated trace elements, and concentration of organic matter. Therefore, consistency is critical in the comparison of short-term and long-term results. Further guidance on filtration may be obtained from Section 4.7.3 of the LSASD Groundwater Sampling Procedure (LSASDPROC-301).

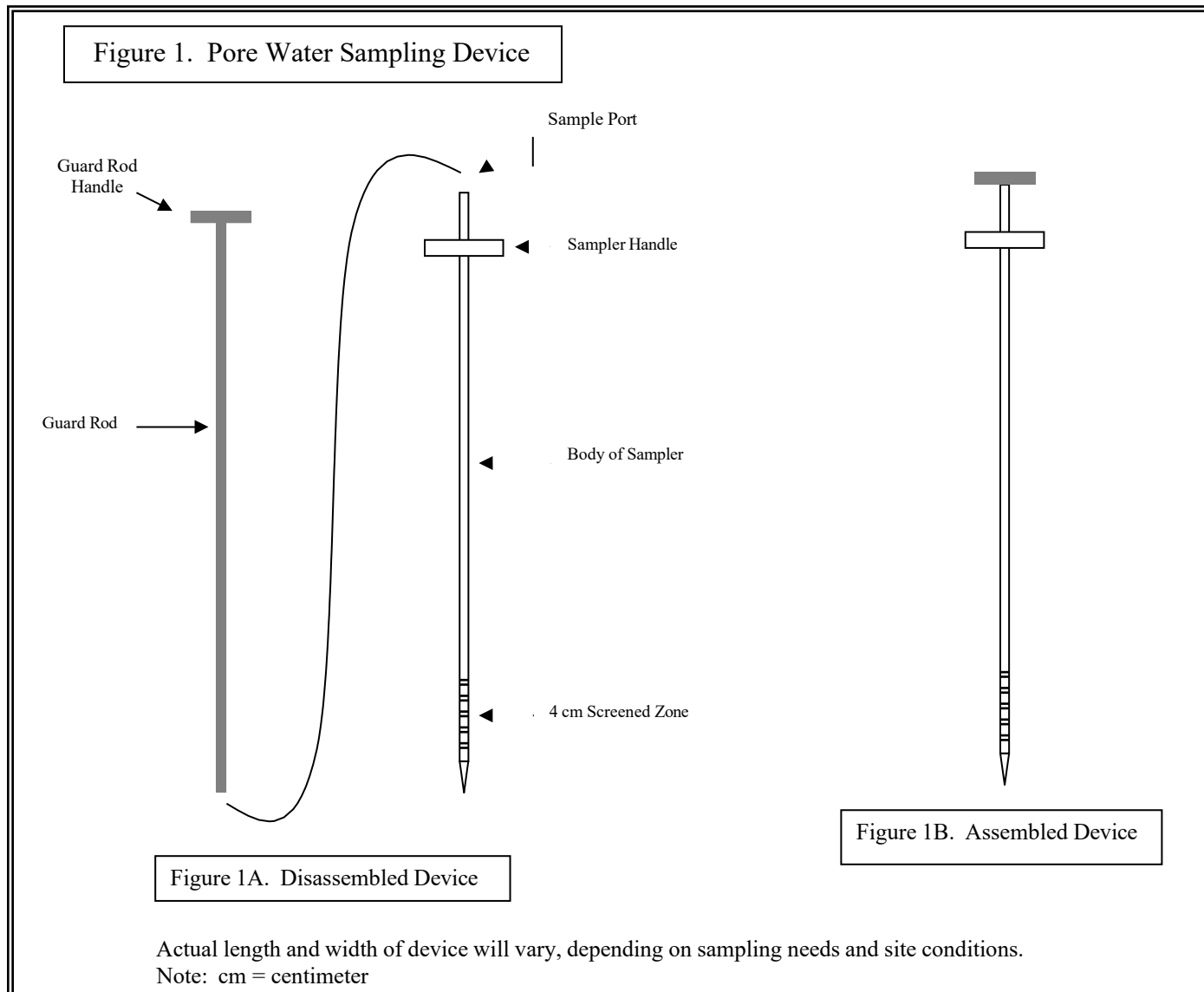
3.3 Special Precautions for Pore Water Sampling

- A clean pair of new, non-powdered, disposable latex gloves will be worn each time a different location is sampled, and the gloves should be donned prior to handling sampling equipment. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- All background or control samples shall be collected and placed in separate ice chests or shipping containers. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.
- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.
- Samplers must use new, verified, certified clean disposable equipment, or pre-cleaned non-disposable equipment. Non-disposable equipment should be pre-cleaned according to procedures contained in LSASD Operating Procedure for Field Equipment Cleaning and Decontamination (LSASDPROC-205), for collection of samples for trace metals or organic compound analyses.

3.4 Sample Handling and Preservation Requirements

- Pore water will typically be collected using a peristaltic pump and placed directly into sampling containers. In some cases, a syringe may be used to collect the pore water and then either left in the syringe as the sample container or transferred into an appropriate container.
- During sample collection, if transferring the sample from a collection device, make sure that the device does not come in contact with the sample containers.
- Place the sample into appropriate, labeled containers. Samples collected for VOC analysis must not have any headspace (see Section 3.1).
- All samples requiring preservation must be preserved as soon as practically possible, soon after sample collection. If pre-preserved VOA vials are used, these will be preserved with concentrated hydrochloric acid prior to departure for the field investigation. For all other chemical preservatives, LSASD will use the appropriate chemical preservative generally stored in an individual single-use vial as described in the LSASD Operating Procedure for Field Sampling Quality Control (LSASDPROC-011). The adequacy of sample preservation will be checked after the addition of the preservative for all samples, except for the samples collected for VOC analysis. If it is determined that a sample is not acceptably preserved, additional preservative should be added to achieve adequate preservation. Preservation requirements for surface water samples are found in

the USEPA Laboratory Services Branch *Laboratory Operations and Quality Assurance Manual* (LOQAM).



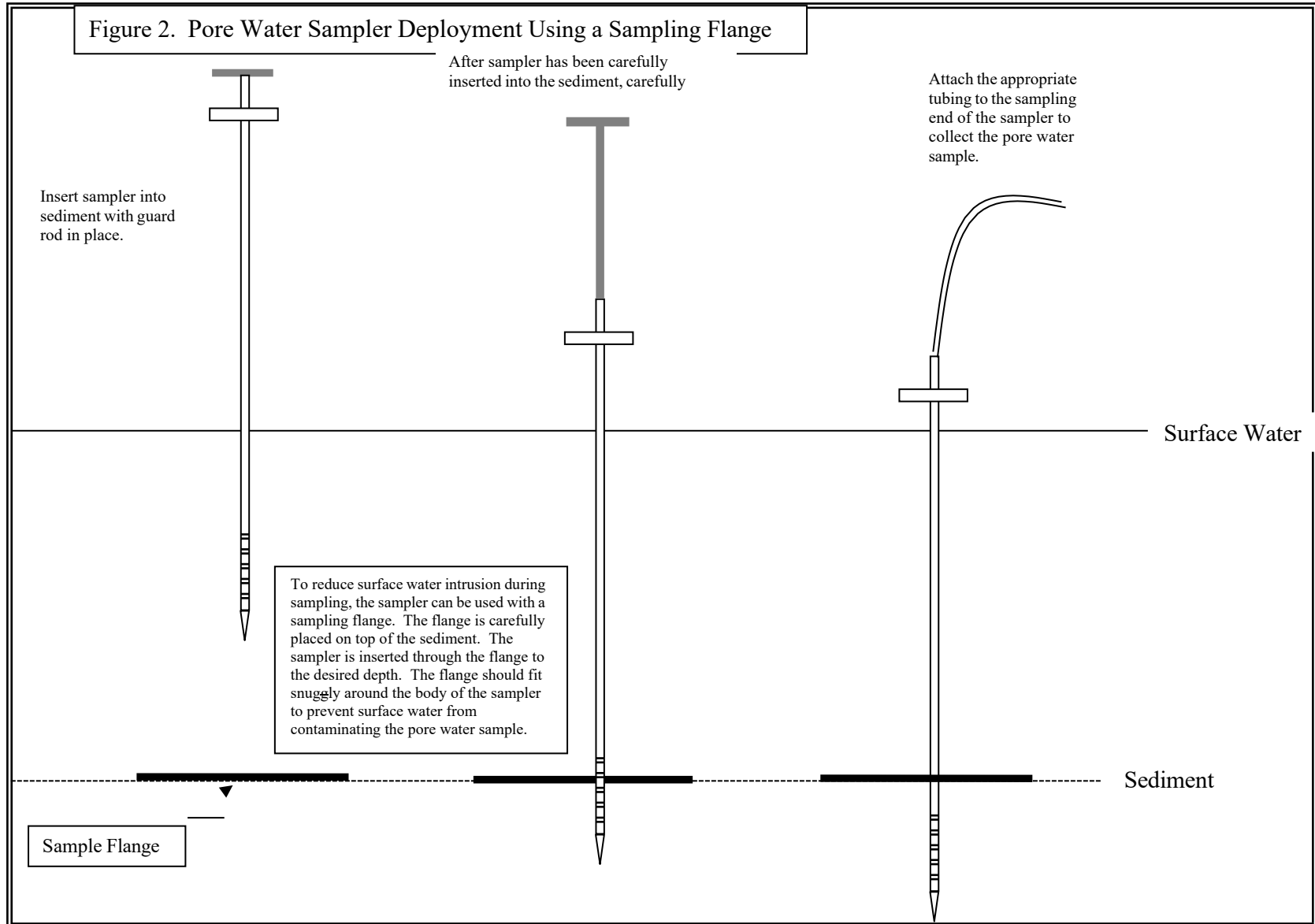
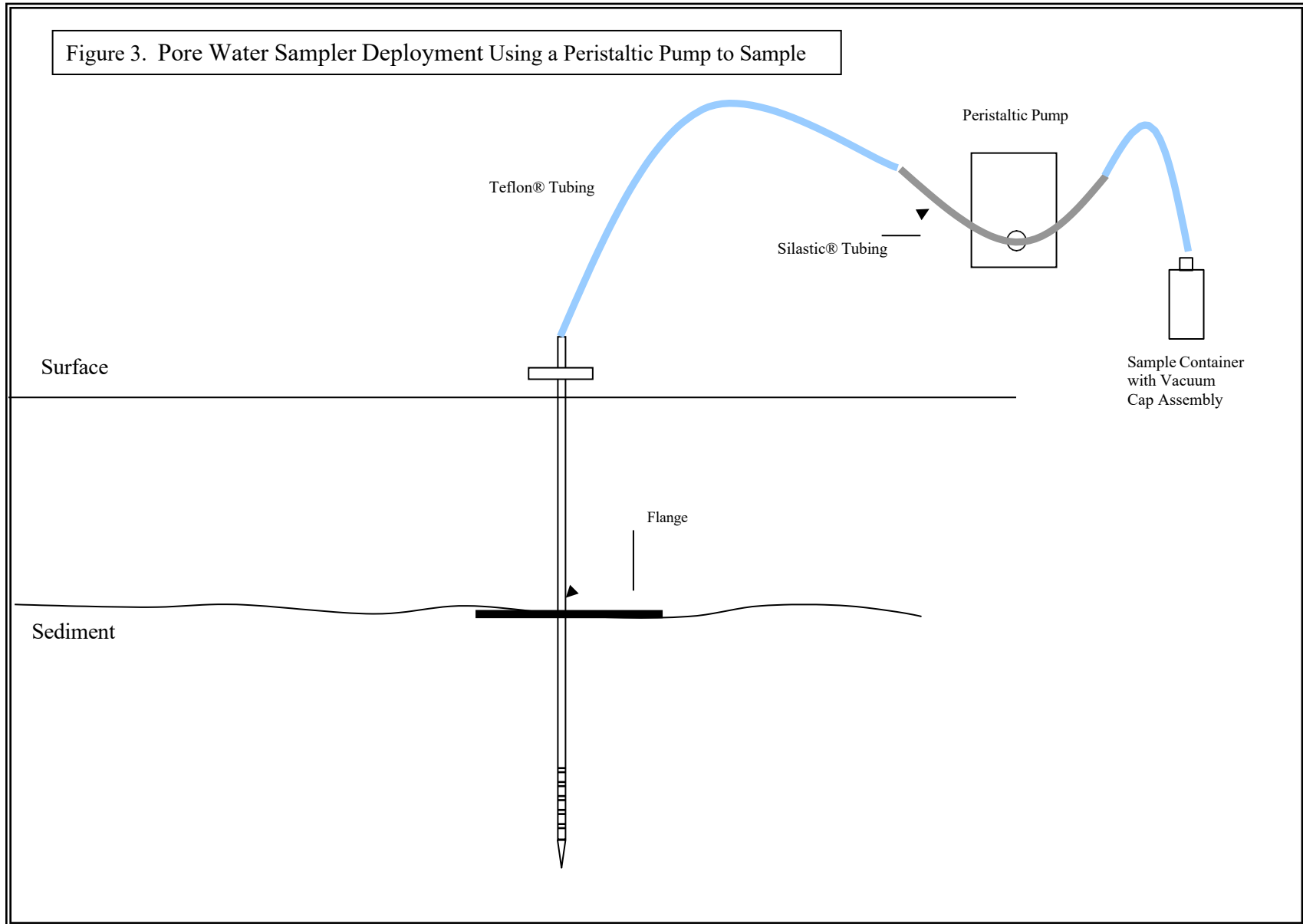


Figure 3. Pore Water Sampler Deployment Using a Peristaltic Pump to Sample



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M.H.E. Products. 2003. PushPoint Sampler (US Pat. # 6,470,967) Operators Manual and Applications Guide, Version 2.01. East Tawas, MI. <http://www.mheproducts.com>

LSASD Operating Procedure for Control of Records, LSASDPROC-002, Most Recent Version.

LSASD Operating Procedure for Sample and Evidence Management, LSASDPROC-005, Most Recent Version.

LSASD Operating Procedure for Logbooks, LSASDPROC-010, Most Recent Version.

LSASD Operating Procedure for Surface Water Sampling, LSASDPROC-201, Most Recent Version.

LSASD Operating Procedure for Pump Operation, LSASDPROC-203, Most Recent Version.

LSASD Operating Procedure for Field Equipment Cleaning and Decontamination, LSASDPROC-205, Most Recent Version.

LSASD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, LSASDPROC-206, Most Recent Version.

LSASD Operating Procedure for Groundwater Sampling, LSASDPROC-301, Most Recent Version.

LSASD Operating Procedure for Potable Water Supply Sampling, LSASDPROC-305, Most Recent Version.

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USEPA SHEMP Safety, Health and Environmental Management Program Procedures and Policy Manual. Laboratory Services and Applied Science Division, Region 4, Athens, GA. Most Recent Version.

LSASD Operating Procedure for Field Sampling Quality Control, LSASDPROC-011, Most Recent Version.

USEPA. 2016. Diving Safety Manual, current version. US Environmental Protection Agency, Washington, DC.

Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the LSASD Document Control Coordinator on the LSASD local area network (LAN).

History	Effective Date
Replaced Chief with Supervisor; General formatting revisions.	April 22, 2023
<p>LSASDPROC-513-R4, Pore Water Sampling, <i>replaces SESDPROC-513-R3.</i></p> <p>Laboratory Services and Applied Science Division replaces Science and Ecosystem Support Division</p> <p>Title Page: Changed the Field Quality Manager from Hunter Johnson to Stacie Masters.</p> <p>General: Corrected typographical, grammatical, and/or editorial errors.</p> <p>Added language to clarify some procedures.</p>	May 13, 2020
<p>SESDPROC-513-R3, Pore Water Sampling, <i>replaces SESDPROC-513-R2.</i></p> <p>General: Corrected any typographical, grammatical, and/or editorial errors.</p> <p>Title Page: Changed the Field Quality Manager from Bobby Lewis to Hunter Johnson. Updated cover page to represent SESD reorganization. John Deatruck was not listed as the Supervisor of the Field Services Branch</p>	December 16, 2016
SESDPROC-513-R2, Pore Water Sampling, <i>replaces SESDPROC-513-R1.</i>	February 28, 2013
SESDPROC-513-R1, Pore Water Sampling, <i>replaces SESDPROC-513-R0.</i>	January 29, 2013
SESDPROC-513-R0, Pore Water Sampling, <i>Original Issue</i>	February 05, 2007

Region 4
U.S. Environmental Protection Agency
Laboratory Services & Applied Science Division
Athens, Georgia

Operating Procedure

Title: Sample and Evidence Management	ID: LSASDPROC-005-R5
Issuing Authority: Field Services Branch Supervisor	
Effective Date: April 22, 2023	Review Due Date: October 30, 2026
Method Reference: N/A	SOP Author: Paula Whiting

Purpose

This document describes general and specific procedures, methods, and considerations to be used and observed by the Laboratory Services and Applied Science Division (LSASD) field investigators when handling and managing samples and other types of evidence after their collection and during delivery to the laboratory. While this SOP may be informative for other businesses, it is not intended for and may not be directly applicable to operations in other organizations. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

Scope/Application

The procedures contained in this document are to be used by field investigators when handling and managing samples and other evidence collected to support LSASD field investigations. On the occasion that LSASD field investigators determine that any of the procedures described in this section are either inappropriate, inadequate, or impractical and that another procedure must be used, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use.

Note: LSASD is currently migrating to a paperless organization. As a result, this SOP will allow for the use of electronic logbooks, checklists, signatures, SOPs, and forms as they are developed, which will also be housed on the Local Area Network (LAN) and traceable to each project.

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PROCEDURAL SECTION

1. General Information

1.1. Documentation/Verification

1.1.1. This procedure was prepared by persons deemed technically competent by LSASD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the LSASD Local Area Network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

2. Sample and Evidence Identification

2.1. Introduction

2.1.1. Sample identification, chain-of-custody records, receipt for sample records and other field records will be legibly recorded with waterproof, non-erasable ink, unless otherwise specified. If errors are made in any of these documents, corrections will be made by crossing a single line through the error and entering the correct information. All corrections must be initialed and dated. If possible, all corrections should be made by the individual making the error.

2.1.2. Following are definitions of terms used in this section:

2.1.2.1. **Field Investigator:** Any individual who performs or conducts field sampling, observation and/or measurement activities in support of field investigations

2.1.2.2. **Project Leader:** The individual with overall responsibility for conducting a specific field investigation in accordance with this procedure

2.1.2.3. **Field Sample Custodian:** Individual responsible for identifying the sample containers and maintaining custody of the samples and the Chain-of-Custody Record

2.1.2.4. **Sample Team Leader:** An individual designated by the project leader to be present during and responsible for all activities related to the collection of samples by a specific sampling team

2.1.2.5. **Sampler:** The individual responsible for the actual collection of a sample

- 2.1.2.6. **Transferee:** Any individual who receives custody of samples subsequent to release by the field sample custodian
- 2.1.2.7. **Laboratory Sample Custodian:** Individual responsible for accepting custody of samples from the field sample custodian or a transferee
- 2.1.3. One individual may fulfill more than one of the roles described above.
- 2.2. Sample and Evidence Identification Procedures
- 2.2.1. Sample Identification

2.2.1.1. The method of sample identification used depends on the type of sample collected. Field measurement samples are those collected for specific field analysis or measurement where the data are recorded directly in bound field logbooks or on the Chain-of-Custody Record. Examples of field measurements and analyses include XRF, pH, temperature, dissolved oxygen, and conductivity. Samples collected for laboratory analysis will be identified by using a stick-on label or a tag which is attached to the sample container. In some cases, such as biological samples, the label or tag may have to be affixed to a bag containing the sample. If a sample tag is used, the sample should be placed in a bag, then the sample and the tag will be placed in a second bag.

2.2.1.2. The following information will be included on the sample label or tag using waterproof, non-erasable ink:

- Project number;
- Field identification or sample station number;
- Date and time of sample collection;
- Designation of the sample as a grab or composite;
- Whether the sample is preserved or unpreserved;
- The general types of analyses to be performed.

2.2.1.3. Other information such as readily detectable or identifiable odor, color, or known toxic properties may be added as deemed necessary by the project leader or sample custodian.

2.2.2. Digital Images – Photographs and Videos

2.2.2.1. When digital images, which include but are not limited to photographs, digital still images, and videos, are taken for purposes of documenting, and supporting a field investigation, a record, containing relevant information, will be kept in a field logbook. The following information will be recorded in the log:

- Digital Image Location or Station Identification
- Description of what the digital image shows
- Date and time the digital image was taken
- Name of the individual that took the digital image
- Digital file name (assigned by camera)
- Orientation, if applicable
- Other pertinent information

2.2.2.2. When digital images are obtained during a field investigation, the permanent record for the official project file, will be stored on a project dedicated data storage device, which includes but is not limited to Secure Digital (SD) card, Compact Discs (CD), or Flash Drives. A new data storage device will be utilized for each project and once the project is completed the device, containing the unaltered investigation-related images, will be labeled with the project's unique identification number, and placed in the official file. Photographs taken for educational or other purposes should be stored on an additional storage device and should not be included in the official project file.

2.2.2.3. It is LSASD's policy that, during a field investigation, official project specific digital images should be obtained using LSASD issued electronics but if the only option field personnel have is to utilize their own electronic equipment the following steps should be taken:

- Record all required digital image information, as described above
- Record devices make, model and owner's name
- Transfer all digital images to a project specific data storage device and adhere to requirements outlined in the EPA Records Management Policy (CIO 2155.3).
- If personal cell phones are used, email a copy of the photograph to the employee's official EPA email account.

Note: On November 30, 2021, the Office of Mission Support (OMS), EPA Headquarters, has informed the Agency that removable/external media (thumb drives, external hard drives, and solid-state drives) will be banned for use. OMS has provided a process to request an exemption by routing an exemption request through the LSASD Deputy Director to the Region 4 Information Security Officer (ISO). At the time that this SOP was approved for use, EPA

mobile devices, keyboards and mice, printers, digital cameras, and lab and/or field instruments are authorized. Check for updates to the policy when instituting work following this SOP.

2.2.3. Identification of Physical Evidence

2.2.3.1. Physical evidence, other than samples, will be identified, when possible, by recording the necessary information on the evidence. When samples are collected from vessels or containers which can be moved (drums for example), the vessel or container should be marked with the field identification or sample station number for future identification. The vessel or container may be labeled with an indelible marker (e.g., paint stick or spray paint). The vessel or container need not be marked if it already has a unique marking; however, these markings will be recorded in the bound field logbooks. In addition, it is suggested that photographs of any physical evidence (markings, etc.) be taken and the necessary information recorded in the field logbook.

2.2.3.2. Occasionally, it is necessary to obtain copies of recorder and/or instrument charts from facility owned analytical equipment, flow recorders, etc., during field investigations and inspections. A unique identifier will be recorded on the document with that information as well as the following recorded in the logbook:

- Starting and ending time(s) and date(s) for the chart;
- An instantaneous measurement of the media being measured by the recorder will be taken and entered at the appropriate location on the chart along with the date and time of the measurement; and
- A description of the location being monitored, and other information required to interpret the data such as type of flow device, chart units, factors, etc.

2.2.3.3. The field investigator will indicate who the chart (or copy of the chart) was received from and enter the date and time, as well as the field investigator's initials.

2.2.3.4. Documents such as technical reports, laboratory reports, etc., should be marked with the field investigator's signature, the date, the number of pages and from whom they were received. Documents that are claimed by a facility to be "confidential" and, therefore, potentially subject to the Confidential Business Information requirements, will be handled in accordance with LSASD Operating Procedure for Control of Records (LSASDPROC-1001).

3. Chain-of-Custody Procedures

3.1. Introduction

3.1.1. Chain-of-custody procedures are comprised of the following elements: 1) maintaining custody of samples or other evidence, and 2) documentation of the chain-of-custody for evidence. To document chain-of-custody, an accurate record must be maintained to trace the possession of each sample, or other evidence, from the moment of collection to its introduction into evidence.

3.2. Sample Custody

3.2.1. A sample or other physical evidence is in custody if:

- It is in the actual possession of an investigator;
- It is in the view of an investigator, after being in their physical possession;
- It was in the physical possession of an investigator and then they secured it to prevent tampering; and/or,
- It is placed in a designated secure area.

3.3. Documentation of Chain-of-Custody

3.3.1. The following are used to identify and demonstrate how sample integrity is maintained and custody is ensured.

3.3.1.1. **Sample Identification:** A stick-on sample label or a tag should be completed for each sample container using waterproof, non-erasable ink as specified in Section 2.2.1.

3.3.1.2. **Sample Seals:** If appropriate, samples should be sealed as soon as possible following collection using a custody seal with EPA identification. The sample custodian or project leader will write the date and their initials on the seal. The use of custody seals may be waived if field investigators keep the samples in their custody as defined in Section 3.2, from the time of collection until the samples are delivered to the laboratory analyzing the samples.

3.3.1.3. **Field Sample Custodian:** The field sample custodian is the person designated by the project leader to receive and manage custody of samples while in the field, including labeling and custody sealing.

3.3.1.4. **Chain-of-Custody Record:** The field Chain-Of-Custody record is used to document the custody of all samples or other physical evidence collected and maintained by investigators. All physical evidence or samples will be accompanied by a Chain-of-Custody Record. This form may be generated by sample custody management software (Section 5) or it may be a pre-printed multi-sheet carbonless form for hand entry of required information. The Chain-of-Custody Record documents transfer of custody of samples from the sample custodian to another person, to the laboratory or other organizational elements. The Chain-of-Custody Record will not be used to document the collection of split samples where there is a legal requirement to provide a receipt for samples (see Section 4, Receipt for Samples Form (CERCLA/RCRA/TSCA)). The Chain-of-Custody Record also serves as a sample logging mechanism for the laboratory sample custodian. A separate Chain-of-Custody Record should be used for each destination or laboratory used during the investigation.

3.3.1.5. All information necessary to fully and completely document the sample collection and required analyses must be recorded in the appropriate spaces to complete the field Chain-Of-Custody Record. The following requirements apply to Chain-Of-Custody records generated by either sample custody management software or by hand entry on pre-printed forms:

- All sampling team leaders must sign in the designated signature block.
- One sample should be entered on each line and not be split among multiple lines.
- If multiple sampling teams are collecting samples, the sampling team leader's name should be clearly indicated for each sample.
- The total number of sample containers for each sample must be listed in the appropriate column. Required analyses should be entered in the appropriate location on the Chain-of-Custody Record.
- The field sample custodian, project leader or other designee, and subsequent transferee(s) should document the transfer of the samples listed on the Chain-of-Custody Record. Both the person relinquishing the samples and the person receiving them must sign the form. The date and time that this occurs should be documented in the proper space on the Chain-of-Custody Record. The exception to this requirement would be when packaged samples are shipped with a common carrier. Even though the common carrier accepts the samples for shipment, they do not sign the Chain-of-Custody Record as having received the samples.
- The last person receiving the samples or evidence will be the laboratory sample custodian or their designee(s).

- 3.3.1.6. The Chain-of-Custody Record is a uniquely identified document. Once the Record is completed, it becomes an accountable document and must be maintained in the project file. The suitability of any other form for chain-of-custody should be evaluated based upon its inclusion of all the above information in a legible format.
- 3.3.1.7. If chain-of-custody is required for documents received during investigations, the documents should be placed in large envelopes, and the contents should be noted on the envelope. The envelope will be sealed, and an EPA custody seal placed on the envelope such that it cannot be opened without breaking the seal. A Chain-of-Custody Record will be maintained for the envelope. Any time the EPA seal is broken, that fact will be noted on the Chain-of-Custody Record and a new seal affixed, as previously described in this section.
- 3.3.1.8. Physical evidence such as video tapes or other small items will be placed in an evidence bag or envelope and an EPA custody seal should be affixed so that they cannot be opened without breaking the seal. A Chain-of-Custody Record will be maintained for these items. Any time the EPA seal is broken, that fact will be noted on the Chain-of-Custody Record and a new seal affixed.
- 3.3.1.9. EPA custody seals can be used to maintain custody of other items when necessary by using similar procedures as those previously outlined in this section.
- 3.3.1.10. Samples should not be accepted from other sources unless the sample collection procedures used are known to be acceptable, can be documented and the sample chain-of-custody can be established. If such samples are accepted, a standard sample label containing all relevant information and the Chain-of-Custody Record will be completed for each set of samples.

3.4. Transfer of Custody with Shipment

3.4.1. Transfer of custody is accomplished by the following:

- Samples will be properly packaged for shipment in accordance with the procedures outlined in LSASD Operating Procedure for Packing, Marking, Labeling and Shipping of Environmental and Waste Samples (LSASDPROC-209).
- All samples will be accompanied by the laboratory copy of the Chain-of-Custody Record. If pre-printed forms are used, the white and pink sheets will be sent. If sample custody management software is used to generate the Chain-of-Custody Record, the laboratory copy is identified with an "L" in the upper right corner. If multiple coolers are needed for shipment to a

particular laboratory, the laboratory copy of the Chain-of-Custody Record for the entire shipment is placed in a sealed plastic bag in one of the coolers. When shipping samples via common carrier, the "Relinquished By" box should be filled in; however, the "Received By" box should be left blank. The laboratory sample custodian is responsible for receiving custody of the samples and will fill in the "Received By" section of the Chain-of-Custody Record. One copy of the Record will be provided to and retained by the project leader. After samples have been received and accepted by the laboratory, a copy of the Chain-of-Custody Record, with LSB sample identification numbers, will be transmitted to the project leader. This copy will become a part of the project file.

- If sent by mail, the package will be registered with return receipt requested. If sent by common carrier, an Air Bill should be used. The Air Bill number, shipment tracking number or registered mail serial number will be recorded in the remarks section of the Chain-of-Custody Record.

4. Receipt for Samples Form (CERCLA/RCRA/TSCA)

4.1. Introduction

4.1.1. Section 3007 of the Resource Conservation and Recovery Act (RCRA) of 1976 and Section 104 of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund) of 1980 require that a "receipt" for all facility samples collected during inspections and investigations be given to the owner/operator of each facility before the field investigator departs the premises. The Toxic Substances Control Act (TSCA) contains similar provisions. The laws do not require that homeowners or other off-site property owners be given this form.

4.2. Receipt for Samples Form

4.2.1. If necessary, a Receipt for Samples form, using either the pre-printed form or one generated by sample custody management software, is to be used to satisfy the receipt for samples provisions of RCRA, CERCLA and TSCA. The form also documents that split samples were offered and either "Received" or "Declined" by the owner/operator of the facility or site being investigated (if a sample is split with a facility, state regulatory agency or other party representative, the recipient should be provided (if enough sample is available) with an equal weight or volume of sample). All information must be supplied in the indicated spaces to complete the Receipt for Samples form.

- The sampler(s) must sign the form in the indicated location
- Each sample collected from the facility or site must be documented in the sample record portion of the form. The sample station number, date and time of sample collection, composite or grab sample designation, whether split samples were collected (yes or no should be entered under the split sample column), a brief description of each sampling location and the total number of sample containers for each sample must be entered.
- The bottom of the form is used to document the site operator's acceptance or rejection of split samples. The project leader must sign and complete the information in the "Split Samples Transferred By" section (date and time must be entered). If split samples were not collected, the project leader should initial and place a single line through "Split Samples Transferred By" in this section. The operator of the site must indicate whether split samples were received or declined and sign the form. The operator must give their title, telephone number and the date and time they signed the form. If the operator refuses to sign the form, the sampler(s) should note this fact in the operator's signature block and initial this entry.
- The Receipt for Samples form is an accountable document after it is completed. A copy of the form is to be given to the facility or site owner/operator. The original form must be maintained in the project files.

5. Sample Custody Management Software

5.1. The container labels and the Chain-of-Custody record should be generated using a sample custody management software to streamline the documentation required by LSASD and/or the Contract Laboratory Program (CLP) for sample identification and chain-of-custody. When possible, the sample custody management software should be used during all field investigations. Once the appropriate information is entered into the computer, the software will generate stick-on labels for the sample containers and will generate sample receipt forms and chain-of-custody records for the appropriate laboratory. The advantages to this system include faster processing of samples and increased accuracy. Accuracy is increased because the information is entered only once, and consequently, consistent for the bottle labels, sample receipt forms and chain-of-custody records.

References

LSASD Operating Procedure for Control of Records, LSASDPROC-1001, Most Recent Version

LSASD Operating Procedure for Packing, Marking, Labeling and Shipping of Environmental and Waste Samples, LSASDPROC-209, Most Recent Version

USEPA Region 4 Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM), November 2001

USEPA Digital Camera Guidance for EPA Civil Inspections and Investigations, July 2006

Revision History

History	Date
Replaced Chief with Supervisor	April 22, 2023
<p>LSASDPROC-005-R4, Sample and Evidence Management, replaces SESDPROC-005-R3</p> <p>SOP put into the new SOP format.</p> <p>General: Corrected any typographical, grammatical, editorial errors and SOP reference updates.</p> <p>Added text in Section 2.2.2 for transferring digital files and EPA policy requirements for using removable storage media.</p>	October 2021
<p>SESDPROC-005-R3, Sample and Evidence Management, replaces SESDPROC-005-R2</p> <p>General: Corrected any typographical, grammatical, and/or editorial errors.</p> <p>Cover Page: SESD's reorganization was reflected in the authorization section by making John Deatrick the Supervisor of the Field Services Branch. The FQM was changed from Bobby Lewis to Hunter Johnson.</p> <p>Revision History: Changes were made to reflect the current practice of only including the most recent changes in the revision history.</p> <p>Section 2.2.2: Revised to clarify accreditation and agency requirements for digital images. Also, language was added to accommodate new storage techniques.</p>	May 25, 2016
SESDPROC-005-R2, Sample and Evidence Management, replaces SESDPROC-005-R1	January 29, 2013
SESDPROC-005-R1, Sample and Evidence Management, replaces SESDPROC-005-R0	November 1, 2007
SESDPROC-005-R0, Sample and Evidence Management, Original Issue	February 05, 2007

Region 4 U.S. Environmental Protection Agency Laboratory Services & Applied Science Division Athens, Georgia	
Operating Procedure	
Title: Field Sampling Quality Control	ID: LSASDPROC-011-R7
Issuing Authority: Field Services Branch Supervisor	
Effective Date: April 22, 2023	Review Due Date: November 22, 2025
Method Reference: N/A	SOP Author: Paula Whiting

Purpose

This document describes procedures established to ensure the quality of Laboratory Services and Applied Science Division (LSASD) field sampling activities, including Field Equipment Center (FEC) operations involving preparation of sampling and support equipment for field operations. Collectively, these procedures ensure that field sampling teams are provided with equipment that is suitable for sampling use, and that field sampling is conducted using proper procedures, resulting in the collection of representative samples. Strict adherence to these procedures forms the basis for an acceptable field sampling quality assurance program. While this SOP may be informative for other businesses, it is not intended for and may not be directly applicable to operations in other organizations. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

Scope/Application

The procedures contained in this document are to be used by field investigators when collecting and handling samples in the field and when preparing sampling equipment for LSASD field investigations. On the occasion that LSASD field investigators determine that any of the procedures described in this SOP are either inappropriate, inadequate, or impractical and that another procedure must be used, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use.

Note: LSASD is currently migrating to a paperless organization. As a result, this SOP will allow for the use of electronic logbooks, checklists, signatures, SOPs, and forms as they are developed, which will also be housed on the Local Area Network (LAN) and traceable to each project.

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PROCEDURAL SECTION

1. General Information

1.1. Documentation/Verification

1.1.1. This procedure was prepared by persons deemed technically competent by LSASD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the LSASD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.2. Definitions

1.2.1. **Sample:** A part of a larger lot, usually a volume, area, period, or population.

1.2.2. **Variability:** The range or “distribution” of results around the mean value obtained from samples within a population. There are three types of variability which should be measured or otherwise accounted for in field sampling, depending on the data quality objectives (DQO) for the study:

1.2.2.1. **Temporal Variability:** Temporal variability is the range of results due to changes in contaminant concentrations over time. An example would be the range of concentrations obtained for a given parameter in wastewater samples collected at different times from an outfall where contaminant concentrations vary over time.

1.2.2.2. **Spatial Variability:** Spatial variability is the range of results due to changes in contaminant concentrations as a function of their location. An example would be the range of concentrations obtained for a given parameter in surface soil from a site where discrete "hot spots" are present due to localized releases of contaminants on otherwise uncontaminated soil.

1.2.2.3. **Sample Handling Variability:** Sample handling variability is the range of results due to the sample collection and handling techniques used by the sampler. This variability manifests itself as a positive bias due to errors such as unclean sampling equipment, cross contamination, etc., or a negative bias due to improper containers or sample preservation.

1.2.3. **Grab Sample:** An individual sample collected from a single location at a specific time or period. Grab samples are generally authoritative in nature.

1.2.4. **Composite Sample:** A sample collected over a temporal or spatial range that typically consists of a series of discrete, equal samples (or “aliquots”) which are combined or “composited.” A composite sample represents the average characteristics of the population under consideration. Four types of composite samples are listed below:

1.2.4.1. **Time Composite (TC):** – a sample comprised of a varying number of discrete samples or “aliquots” collected at equal time intervals during the compositing period. The TC sample is typically used to sample wastewater or streams.

1.2.4.2. **Flow Proportional Composite (FPC):** – A sample consisting of discrete samples or “aliquots” collected at a rate proportional to flow. The aliquots are collected during the compositing period by either a time-varying/constant volume (TV/CV) method (“automated flow proportioning”) or a time-constant/varying volume (TC/VV) method (“manual flow proportioning”). The TV/CV method is typically used with automatic samplers that are paced by a flow meter. The TC/VV method is a manual method that individually proportions a series of discretely collected aliquots. The FPC is typically used when sampling wastewater.

1.2.4.3. **Aerial Composite:** – a sample composited from individual, equal aliquots collected on an aerial or horizontal cross-sectional basis. Each aliquot is collected in an identical manner. Examples include sediment composites from quarter-point sampling of streams and soil samples from within grids.

1.2.4.4. **Vertical Composite:** – a sample composited from individual, equal aliquots collected from a vertical cross section. Each aliquot is collected in an identical manner. Examples include vertical profiles of soil/sediment columns, lakes, and estuaries.

1.2.5. De-ionized Water

1.2.5.1. Tap water that has been treated by passing it through a standard de-ionizing resin column. At a minimum, the finished water should contain no detectable heavy metals or other inorganic compounds (i.e., at or above analytical detection limits) as defined by a standard Inductively Coupled Argon Plasma Spectrophotometer (ICP) (or equivalent) scan. De-ionized water obtained by other methods is acceptable, as long as it meets the above analytical criteria. Organic-free water may be substituted for de-ionized water.

1.2.6. Organic-Free Water

1.2.6.1. Tap water that has been treated with activated carbon and deionizing units. At a minimum, the finished water must meet the analytical criteria of deionized water and it should contain no detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels as determined by the Region 4 laboratory for a given set of analyses. Organic-free water obtained by other methods is acceptable if it meets the above analytical criteria.

1.2.7. Branch Field Equipment Manager

1.2.7.1. Staff, designated by management, who are responsible for ensuring that the procedures for Equipment Inventory and Management are followed. At least one Branch Field Equipment Manager will be designated for the Field Services Branch (FSB).

2. Field Sampling Quality Control Considerations

2.1. This section provides guidelines for establishing quality control procedures for sampling activities. Strict adherence to all the standard operating procedures outlined in this subsection forms the basis for an acceptable sampling quality assurance program.

2.1.1. Experience Requirements

2.1.1.1. There is no substitute for field experience. This field experience will be gained by on-the-job training using the "buddy" system. Each new investigator will accompany an experienced employee on as many different types of field studies as possible. During this training period, the new employee will be permitted to perform all facets of field investigations, including sampling, under the direction and supervision of senior investigators. Specific requirements covering experience, competency and proficiency are found in the LSASD Training and Demonstration of Competency (LSASDPROC-1003, Current Version).

2.1.2. Traceability Requirements

2.1.2.1. All sample collection and measurement activities will be traceable through field records to the person collecting the sample or making the measurement. All maintenance and calibration records for sampling and measurement equipment (where appropriate) will be kept so that they are similarly traceable. The LSASD Operating Procedure for Equipment Inventory and Management (LSASDPROC-1009, Current Version) contain specific procedures to be followed that ensure traceability.

2.1.3. Chain-of-Custody

2.1.3.1. Specific chain-of-custody procedures are included in LSASD Operating Procedure for Sample and Evidence Management (SESDPROC-005, Current Version). These procedures will ensure that evidence collected during an investigation will withstand scrutiny during litigation. To assure that procedures are being followed, it is recommended that field investigators or their designees audit chain-of-custody entries, tags or labels, field notes, and any other recorded information for accuracy. Additionally, the LSASD Quality Assurance Manager (QAM) will randomly conduct reviews of project files to ensure that quality procedures are being followed.

2.1.4. Sampling Equipment Construction Material

2.1.4.1. Sampling equipment construction materials can affect sample analytical results. Field investigators will ensure the sample equipment construction material will not introduce contaminants to the sample being collected.

2.1.5. Sample Preservation

2.1.5.1. Samples for some analyses must be preserved to maintain their integrity. Preservatives required for routine analyses of samples collected are found in the USEPA Region 4 Laboratory Services Branch Laboratory Operations and Quality Assurance Manual (LSBLOQAM). Chemical preservatives used will be supplied by the Region 4 laboratory or purchased by the Branch Field Equipment Manager. All samples requiring preservation should be preserved immediately upon collection in the field. Records of sample preservation, including ice, will be documented in the field log books.

2.1.5.2. Samples that should not be preserved in the field are:

- Those collected within a hazardous waste site that are known or thought to be highly contaminated with toxic materials which may be highly reactive. Barrel, drum, closed container, spillage, or other source samples from hazardous waste sites are not to be preserved with any chemical.
- Those that have extremely low or high pH or samples that may generate potentially dangerous gases if they were preserved according to the LSBLOQAM.

2.1.5.3. All samples preserved with chemicals will be clearly identified by indication on the sample tag or label that the sample is preserved. If samples normally requiring preservation were not preserved, field records should clearly specify the reason. Samples shipped by air will not be preserved with nitric acid, hydrochloric acid, sodium hydroxide or sulfuric acid in excess of the amount specified in the LSBLOQAM.

2.2. Sample Collection Precautions

2.2.1. To prevent cross-contamination during sample collection, the following precautions will be taken:

- A clean pair of new, non-powdered, disposable latex or nitrile gloves will be worn each time a different location is sampled, and the gloves should be donned immediately prior to sampling. The gloves should not come into contact with the media being sampled.
- Sample containers for source samples or samples suspected of containing high concentrations of contaminants will be placed in separate plastic bags immediately after collecting, tagging, etc.
- If possible, environmental (low concentration) samples and source or waste samples (high concentration) should be collected by different field teams. If different field teams cannot be used, all environmental samples should be collected first and placed in separate ice chests or shipping containers. Samples of waste or highly contaminated samples should never be placed in the same ice chest as environmental samples. Ice chests or shipping containers for source or waste samples or any samples suspected to contain high concentrations of contaminants will be lined with new, clean, plastic bags.
- If possible, one member of the field sampling team should record all the field notes, collect GPS data, etc., while the other members collect the samples.
- When sampling surface water and sediment at the same location, the water sample should always be collected before the sediment sample is collected.
- Sample collection activities should proceed progressively from the least suspected contaminated area to the most suspected contaminated area.

- Investigators should use equipment constructed of Teflon[®], stainless steel, or glass that has been properly pre-cleaned according to either the LSASD Operating Procedure for Field Equipment Cleaning and Decontamination (LSASDPROC-205, Current Version) or the LSASD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (ASBPROC-206, Current Version) for collection of samples for trace metals or organic compounds analyses. Teflon[®] or glass is preferred for collecting samples where trace metals are of concern. Equipment constructed of plastic or PVC should not be used to collect samples for trace organic compounds analyses.
- Field investigators should ensure the sample containers they are using have been verified as suitable for the analyses that will be conducted on the samples through the quality control procedures discussed in Section 4 of this procedure.

2.2.2. Upon returning from the field, un-used sample containers will be examined by project leaders to determine whether bottles should be discarded, recycled or re-shelved for use on other projects. A FEC load-in form (SESDFORM-011, Current Version) will be completed and signed by project leaders to identify the future use of sample containers returning from the field. Opened boxes of sampling containers that can be re-used, will be segregated from sealed boxes of new containers.

2.2.3. Opened bags of latex or nitrile gloves returning from the field will be segregated from unopened gloves and will not be re-used for sample collection on other projects.

2.3. Sample Handling and Mixing

2.3.1. Once a sample has been collected, it may have to be transferred into separate containers for different analyses. Sample transfer should be done as soon as possible. If necessary, aqueous samples may be collected into a single, larger container for homogenization and transferred into individual sample containers. However, aqueous samples collected for volatile organic compounds, oil and grease, bacteria, sulfides, and phenols analyses may not be transferred using this procedure.

2.3.2. It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is representative of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

- The material in the sample pan should be divided into quarters and each quarter should be mixed individually.
- Two quarters should then be mixed to form halves.
- The two halves should be mixed to form a homogenous matrix.

2.3.3. This procedure should be repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction, and occasionally turning the material over.

2.4. Special Handling of Samples for Volatile Organic Compounds Analysis

2.4.1. Water samples to be analyzed for volatile organic compounds should be stored in 40-ml septum vials with screw cap and Teflon[®]-silicone disk in the cap to prevent contamination of the sample by the cap. The disks should be placed in the caps (Teflon[®] side to be in contact with the sample) in the laboratory prior to the beginning of the field investigation.

2.4.2. The vials should be completely filled with no headspace to prevent volatilization, and extreme caution should be exercised when filling a vial to avoid any turbulence which could also produce volatilization. The sample should be carefully poured down the side of the vial to minimize turbulence. As a rule, it is best to gently pour the last few drops into the vial so that surface tension holds the water in a convex meniscus. The cap is then applied, and some overflow is lost, but the air space in the bottle is eliminated. After capping, turn the bottle over and tap it to check for bubbles. If a bubble or bubbles are present, the vial should be topped off using a minimal amount of sample to re-establish the meniscus. Care should be taken not to flush any preservative out of the vial during topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained, and the sample re-collected.

2.4.3. Soil and sediment samples for VOC analyses should be collected and handled as specified in the LSASD Operating Procedure for Soil Sampling (LSASDPROC-300, current version), Waste Sampling (LSASDPROC-302, current version) or the LSASD Operating Procedure for Sediment Sampling (LSASDPROC-200, current version). Soil and sediment samples collected for VOC analyses should not be mixed.

2.5. Sample Storage and Transport

2.5.1. After collection, sample handling should be minimized. Field investigators should use extreme care to ensure that samples are not contaminated during storage. Environmental and waste samples are typically stored in coolers. To reduce the risk of cross contamination, smaller sample containers such as 8-ounce glass jars, 40 ml volatile organic analyses (VOA) vials, and one-liter amber bottles should be placed inside of sealed, plastic bags before being placed in the cooler. If ice is required for preservation of the samples, the ice should be contained in a plastic bag or some equivalent container to prevent the potential for cross contamination of the samples by water produced from melting ice. If ice is used, the coolers should be checked regularly, and water should be drained as needed. Custody of samples will be maintained according to the LSASD Operating Procedure for Sample and Evidence Management (LSASDPROC-005, current version).

2.5.2. Samples will either be transported to the analytical laboratory by field investigators or shipped by common carrier. Shipping of samples will be conducted in accordance with the LSASD Operating Procedure for Packing, Marking, Labeling and Shipping of Environmental and Waste Samples (LSASDPROC-209, current version).

3. Quality Control Samples

3.1. Quality control samples are collected during field studies for various purposes, among which are to isolate site effects (control samples), to define background conditions (background sample), and to evaluate field/laboratory variability (spikes and blanks, trip blanks, duplicate, split samples, etc.).

3.1.1. Control Sample

3.1.1.1. A control sample is typically a discrete grab sample collected to isolate a source of contamination. Isolation of a source could require the collection of both an upstream sample at a location where the medium being studied is unaffected by the site being studied, as well as a downstream control which could be affected by contaminants contributed from the site under study.

3.1.2. Background Sample

3.1.2.1. A background sample (usually a grab sample) is collected from an area, water body or site similar to the one being studied but located in an area known or thought to be free from pollutants of concern.

3.1.3. Variability Samples

3.1.3.1. Variability may be defined as a variation in concentrations of compounds or analytes across a site or area of investigation or variations, across time, of waste streams or surface water bodies. Variation can also be introduced during sample handling. The following procedures are used to assess and evaluate variability. When appropriate, spatial duplicate grab and/or composite samples should be collected during investigations and studies in accordance with the project DQOs. In general, no more than ten percent of all samples should be collected as spatial duplicates.

3.1.3.1.1. Spatial Variability Duplicate

3.1.3.1.1.1. The following spatial duplicate sampling procedures should be used during the collection of samples as a measure of variability within the area represented by the sample. These samples should be collected at the same time, using the same procedures, the same type of equipment, and in the same types of containers as the original samples. They should also be preserved in the same manner and submitted for the same analyses as the required samples.

3.1.3.1.1.2. Spatial variability duplicate samples are typically collected during investigations where samples are collected from grids that are positioned at fixed intervals over the study area and a sample collection pattern is established within the grids. Spatial variability duplicate samples are collected using the same compositing pattern as the original sample and are collected within the same general area of representativeness; however, the pattern is shifted relative to the original aliquot locations. This amount and direction of shift for the duplicate sample is dependent upon the size of the grid or area being sampled and should be specified in the Quality Assurance Project Plan (QAPP) for the investigation. Data from spatial duplicates will be examined by the investigation project leader to determine if the observed spatial variability is acceptable, based on the investigation or study objectives.

3.1.3.1.2. Temporal Variability Duplicate

3.1.3.1.2.1. When appropriate, temporal variability at a given sampling location will be measured by collecting temporal duplicate samples. These samples will be collected from the same sampling location, using the same techniques and the same type of equipment, but at a time different from the original sample. The time selected for the temporal duplicate sample will be similar to

the time or span of time specified for the original sample in the project work plan. Data from temporal duplicates will be examined by the project leader to determine if samples represent the time span intended in the project work plan.

3.1.3.1.3. Sample Handling Variability

3.1.3.1.3.1. The effectiveness of sample handling techniques will be measured by collecting split and blank samples.

3.1.3.1.3.1.1. Split Samples: Split samples will be collected by initially collecting twice as much volume as is normally collected. The material will be apportioned, after mixing, if appropriate, into two sets of containers. Both sets of containers will be submitted for analyses with one set designated as an "original sample," the other designated as a "split sample." Data from the split samples will be examined by the project leader to assess sample handling variability. On large studies (more than 20 samples collected), a minimum of 5 percent, but no more than 10 percent, of all samples will be collected as split samples unless required by site data quality objectives.

3.1.3.1.3.1.2. Blank Samples: The following blank samples will be prepared by the laboratory and obtained by the project leader prior to traveling to a sample site.

- Water Sample VOC Trip Blank - A water sample VOC trip blank is required for every study where water samples are collected for VOC analysis. Sealed preserved (or unpreserved, if unpreserved vials were used during the investigation) 40-ml VOC vials will be transported to the field. Two sealed VOC vials will be submitted per trip blank sample. At least one trip blank sample will be submitted per sample shipment. Trip blanks will be prepared by lab personnel. Investigators should submit their request for trip blanks at least one week in advance of scheduled field investigations and inspections and never (except in emergency situations) less than two days in advance of scheduled field investigations and inspections. These samples should not be picked up earlier than the morning of departure for the scheduled inspection/investigation. These trip blanks will be handled and treated in the same manner as the water samples collected for volatile organic compounds analysis on that study. These samples will be clearly identified on sample labels and Chain-of-Custody Records as trip blanks.

- Soil/Sediment Sample VOC Trip Blank - A soil/sediment sample VOC trip blank is required for every study where soil and/or sediment samples are collected for VOC analysis. The required containers are specified the USEPA Region 4 LSBLOQAM. The request and pick up of the soil blank sample will be the same as for the water trip blank. En Core[®] containers will be transported to the field. These blanks will be handled and treated by field investigators in the same manner as the soil samples collected for VOC analysis on that study. These samples will be clearly identified on sample labels and Chain-Of-Custody Records as trip blanks. Two sealed En Core[®] containers will be submitted per trip blank sample. At least one set of trip blank samples will be submitted per sample shipment.

3.1.3.1.3.1.3. The following blanks are prepared in the field:

- Sample Preservative Blanks - LSASD will generally use chemical preservatives stored in individual single-use vials. The chemical preservative will be tested prior to use for the appropriate analytes. The use of pre-tested, single-use vials eliminates the need to routinely collect preservative blanks in the field. If the preservatives are stored in containers that will be used to preserve multiple samples, blanks will be collected to evaluate the potential for cross-contamination resulting from the preservation process. If preservative blanks are collected, sample containers will be filled with de-ionized water by LSASD personnel and transported to the field and preserved and submitted for the same analyses as the other inorganic samples collected. These samples will be clearly identified as preservatives blanks on sample labels and the Chain-Of-Custody Record(s). At least one preservative blank for each type of preserved sample should be collected at the end of routine field investigations. In addition, one preservative blank will be collected for each multi-use bottle of preservative used.

Note: The deionized water will be generated from a water treatment unit provided by the LSASD laboratory.

- Equipment Rinsate Blanks - Equipment rinsate blanks will be collected whenever field decontamination of equipment to be re-used in sampling activities is performed.

- When field cleaning of equipment is required during a sampling investigation, a piece of the field-cleaned equipment will be selected for collection of a rinse blank. At least one rinse blank will be collected during each week of sampling operations. After the piece of equipment has been field cleaned and prior to its being used for sample operations, it will be rinsed with organic-free water. The rinse water will be collected and submitted for analyses of all constituents for which normal samples collected with that piece of equipment are being analyzed.
- Organic-Free Water System Blanks - When using a portable organic-free water generating system in the field, a sample of the water generated by the system will be collected at least once during each week of operations. Based on the objectives of the study or investigation, it may be appropriate to collect a sample of the raw source water. The collected water sample will be submitted for analyses of all constituents for which normal samples are being analyzed.
- Material Blanks - When construction materials are being used on a site in such a way as to have a potential impact on constituent concentrations in the sample, a sample of each material will be submitted for analysis.

Note: For drilling operations where materials are shipped directly to the site from the supplier, see LSASD Guidance for Design and Installation of Monitoring Wells (SESDGUID-101, Current Version) for material blank collection and reporting requirements.

- Automatic Sampler Blanks - In general, cleaning procedures outlined in the LSASD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (ASBPROC-206, Current Version) should be adequate to ensure sample integrity. However, it is the standard practice of the Field Services Branch to submit automatic sampler blanks for analyses when automatic samplers are used to collect samples for organic compounds and metals analyses. Automatic sampler blanks for other standard analyses may be submitted in the event of a special investigation (e.g., criminal or civil).

- **Field Blank** - A field blank is a sample that is prepared in the field to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected (for example air-borne dust or organic vapors which could contaminate a soil sample). Organic-free water is taken to the field in sealed containers or generated on-site. The water is poured into the appropriate sample containers at pre-designated locations at the site. Field blanks should be collected in dusty environments and/or from areas where volatile organic contamination is present in the atmosphere and originating from a source other than the source being sampled.
- **Temperature Blank** - A temperature blank is a container of water shipped with each cooler of samples requiring preservation by cooling to 6°C (ice). The temperature of the blank is measured at the time of sample receipt by the laboratory. No temperature blank is necessary for waste samples since waste samples do not require ice for preservation.
- **Wipe Sample Blank** - A wipe sample blank is a sample of the material and solvent used for collecting wipe samples. The blank is handled, packaged and transported in the same manner as all other wipe samples with the exception that it is not exposed to actual contact with the sample medium.
- **Water Filter Blank** - When filters are used for sampling a dissolved constituent, de-ionized water should be run through at least one filter from each lot and the filtered water submitted for the same analyses. When filters are used for chlorophyll sampling, the filter should be prepared using de-ionized water and submitted for the same chlorophyll analysis.

3.2. Spikes

- 3.2.1. Spike samples are used to measure bias due to sample handling or analytical procedures. Spike samples are typically used by LSASD to evaluate the performance of contract laboratories and are shipped directly to the Contract Laboratory Program (CLP) laboratory by the ESAT contractor.

3.3. Matrix Spike/Matrix Spike Duplicate Samples for Water and Soil Samples for Organic Compounds Analyses

- 3.3.1. Matrix spike and matrix spike duplicate (MS/MSD) samples will be submitted to the laboratory for volatile organic compounds, extractable organic compounds, pesticides/PCBs and/or herbicides analyses from at least one sampling location per project and laboratory used. One MS/MSD sample should be collected per 20 samples per media collected.
- 3.3.2. Additional volume will be required for the soil MS/MSD samples. Semi-volatile organic compounds, pesticides, and PCB analyses of soil/sediment samples require the collection of one additional eight-ounce glass jar. For VOC soil/sediment samples, double volume, i.e., six En Cores[®] or six 40 ml vials with syringe collected sample, is needed for the MS/MSD samples.
- 3.3.3. Additional volume will be required for the water MS/MSD samples. For routine full scan analysis, i.e., extractable organic compounds, pesticides, and PCBs, four one-liter amber containers provide the required sample volume. Eight containers, therefore, should be submitted for the MS/MSD sample. For VOC water samples, a total of six 40 ml vials should be collected.
- 3.3.4. MS/MSD samples should be collected from a location expected to be relatively free from contamination, since the samples will be used for laboratory quality control purposes. The duplicate samples should be clearly identified as "Duplicate Sample for Matrix Spike" or "MS/MSD" on the Chain-Of-Custody Record, in the field logbook and on the Contract Laboratory Program Traffic Report Form (if appropriate). This procedure will be followed for all projects where water samples are collected for the indicated analyses. For non-routine sampling events, the Region 4 LSASD laboratory should be consulted for specific sample volume and container requirements.

3.4. Matrix Spike/Matrix Spike Duplicate Samples for Water and Soil Samples for Inorganic Analyses

- 3.4.1. A matrix spike sample and a duplicate sample (MS/MSD) will be submitted to the laboratory for inorganic analyses from at least one sampling location per project and laboratory used. One matrix spike and duplicate sample should be collected per 20 samples per media collected per laboratory.
- 3.4.2. Soil/sediment and water samples collected for inorganic analyses will normally have sufficient sample volume to perform the matrix spike analyses without requiring the collection of extra sample volume. The project leader should designate a sample, typically one considered to be representative of background or relatively uncontaminated conditions, as the matrix spike sample. For water samples, the

sample volume collected will normally provide adequate volume for the MS/MSD analyses.

- 3.4.3. MS/MSD samples should be collected from a location expected to be relatively free from contamination, since the samples will be used for laboratory quality control purposes. MS/MSD samples should be clearly identified as "Duplicate Sample for Matrix Spike" or "MS/MSD" on the Chain-Of-Custody Record, in the field logbook and on the Contract Laboratory Program Traffic Report Form (if appropriate). This procedure will be followed for all projects where water samples are collected for the indicated analyses. For non-routine sampling events, the Region 4 LSASD laboratory should be consulted for specific sample volume and container requirements.

3.5. Special Quality Control Procedures for EPA Contract Laboratories

- 3.5.1. On a case-by-case basis, field investigators may be required to collect split samples (or duplicate samples if appropriate) for analyses by either the Region 4 LSASD laboratory or contract laboratories. The split samples are to be submitted to the Region 4 laboratory using established procedures. The contract laboratory involved will not be notified that samples were split, i.e., there should be no indication on Chain-Of-Custody Records or CLP Traffic Report Forms submitted to the contract laboratories that these samples were split with the Region 4 LSASD laboratory.

3.6. Special Quality Control Procedures for Dioxins and Furans

- 3.6.1. The Region 4 laboratory does not conduct in-house analyses for dioxins and furans. Dioxin and furans analyses are conducted by contract laboratories. The Region 4 laboratory may accept environmental samples (soil, sediment, groundwater, and surface water) suspected of being contaminated with polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF), as long as suspected PCDD and PCDF contamination is not due to RCRA hazardous waste classified as F020-023 and/or F026-028. If these environmental samples are not contaminated with a F020-023 and/or F026-028 waste, it may be analyzed for parameters other than dioxin and furans. Environmental samples known or suspected to be contaminated with the RCRA hazardous waste F020-023 and or F026-028 will not be accepted.

Note: Environmental samples suspected of being contaminated with RCRA hazardous waste classified in 40 CFR, 261.31 as F032 will be accepted. The F032 waste is defined as wastewaters (except those that have not come into contact with process contaminants), process residuals, preservative drippage, and spent formulations from wood preserving processes generated at plants that currently use or have previously used chlorophenolic formulations. The F032 listing does not include K001 bottom sediment sludge from the treatment of wastewater from wood preserving processes that use creosote and or pentachlorophenol. Prior to a

sampling event, the project leaders should consult with the Analytical Services Branch Sample Control Coordinator to determine if the Region 4 laboratory can accept the samples. The Region 4 LSASD laboratory should also be consulted for the current quality control procedures for dioxins and furans samples prior to a sampling investigation.

4. Internal Quality Control Procedures

4.1. The focus of this section is on Field Equipment Center (FEC) operations involving preparation of sampling and support equipment for field operations, as well as field data generated under the specific sample collection quality control procedures discussed in Section 2. Quality control checks of these operations ensure that field sampling teams are provided with equipment that is suitable for sampling use, and that field sampling is conducted using proper procedures.

4.1.1. Traceability Requirements

4.1.1.1. Records will be kept by designated LSASD staff or FEC personnel documenting the dates of operations and the person performing operations for the following:

- Organic-Free Water System Maintenance (FEC System) - Maintenance on the FEC organic-free water system will be performed at least once per 180 days.
- Air Monitoring Safety Instrumentation Checkouts - Pre-loadout checks on safety monitoring instrumentation will be recorded each time they are performed. Discrepancies will be immediately reported to the Branch Safety Officer.
- Self-Contained Breathing Apparatus (SCBA) Checkouts - Pre-loadout checks on SCBAs will be recorded when they are performed. SCBA checkouts will be performed at least once per calendar quarter in the absence of loadout requests. Any discrepancies will be reported immediately to the Branch Safety Officer.
- Other Equipment Maintenance - Maintenance performed on equipment other than that listed above will be accordance to the LSASD Operating Procedure for Equipment Inventory and Management (LSASDPROC-1009, Current Version). All required repairs will be reported to appropriate Branch Field Equipment Manager.
- Tubing, Sampling Containers and Latex Gloves - The Field Services Branch Quality Assurance Officer (FSB QAO) is responsible for conducting verification sampling for tubing, sample containers, and latex gloves that are used during field investigations. Upon receipt, the tubing,

containers, and gloves are placed in the quarantine room at the FEC. A record is kept of the lot numbers for each shipment received. The FSB QAO, or designee, will collect blank samples from tubing, containers and gloves within each lot received and will review the results to ensure the sample containers and gloves are suitable for use during field investigations. Once the supplies are deemed suitable, the FSB QAO will release the items for use.

- Chemical preservatives commercially purchased will be tested prior to use. Each lot of chemical preservative will be tested for the appropriate analytes by either the Branch Field Equipment Manager or the FSB QAO. Once released by FSB QAO, the preservatives can be used in the field.
- Equipment - All equipment cleaned and wrapped for field use will be marked with the date on which preparation was completed. Equipment will be stored at the FEC in specified areas to minimize the risk of contamination while awaiting use.

4.1.2. Specific Quality Control Checks

4.1.2.1. When collecting samples during field investigations, it is necessary to take measures to prevent cross contamination to ensure the integrity of the data generated. The FSB conducts verification sampling of sample containers, gloves, sampling equipment, tubing and water utilized during field investigations as one of these measures. At least twice per calendar year, the FSB QAO will conduct the following checks and issue a written report to the QAM with the results.

4.1.2.1.1. Collect and submit for analyses samples of each new lot of containers received. Bottles from each lot will be tagged and sealed, then submitted for the following analyses:

- Amber, 1-liter – extractable organics, pesticides, and PCBs.
- Clear Glass, 8-oz. – metals, cyanide, extractable organics, pesticides, PCBs, and volatile organic compounds.
- Polyethylene, 1-liter – metals and cyanide.

Note: In addition to the quality control checks listed above, samples may be collected during field investigations for classical inorganic parameters such as nitrates, nitrites, sulfides, etc. Due to the detection levels generally required for these parameters, it is unlikely that cross contamination may occur in association with the sample containers and sampling equipment used during sample collection. Therefore, classical inorganic analyses are not conducted as part of the routine quality control checks. If the data quality objectives require additional quality control checks, bottles will be submitted to the laboratory for analyses.

- 4.1.2.2. Collect and submit for analyses a rinsate blank for each new lot of latex or nitrile gloves received during the calendar quarter. Samples will be collected as rinse blanks using organic-free water. The rinsate will be submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides, and PCBs. A new glove will be rinsed for each parameter (e.g., one glove for the VOC sample, another glove for metals, etc.) to avoid dilution of potential contaminants on the gloves. Water for the VOC samples should be provided by the LSB laboratory.
 - 4.1.2.3. Collect and submit for analyses a sample of water from the FEC organic-free water system. The sample will be submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides, and PCBs.
 - 4.1.2.4. Collect and submit for analyses a rinsate blank of at least one piece of sampling or sample related equipment stored at the FEC. The sample will be submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides, and PCBs. Water for the VOC samples should be provided by the LSB laboratory.
 - 4.1.2.5. Collect and submit for analyses a rinsate blank for each new lot of Silastic[®] or Tygon[®] tubing used in peristaltic pump head. The sample will be submitted for metals and cyanide analysis.
 - 4.1.2.6. Teflon[®] tubing – Collect and submit for analyses a rinsate blank for each new lot of Teflon[®] tubing received. Rinse blanks will be collected through the Teflon tubing. The sample will be submitted for metals, cyanide, extractable organics, volatile organic compounds, pesticides, and PCBs. Water for the VOC samples should be provided by the LSB laboratory.
- 4.1.3. Quality Control for Special Order Equipment and Supplies
- 4.1.3.1. Some equipment and supplies ordered for specific projects are received in what can be considered ready to use condition. In order to ensure the integrity of these materials, an equipment rinsate blank will be collected from at least one item in each lot. The equipment and supplies will not be used until the QAO has reviewed the analytical data for the blanks and released the items.
- 4.1.4. Quality Control Evaluation and Corrective Action
- 4.1.4.1. All field investigation reports will contain a clearly identified section where the results for all field generated quality control (QC) samples are discussed and reported. Quality control data review includes but is not limited to detections of organic and inorganic compounds at any concentration in quality control blanks (i.e., trip blanks, equipment rinsate blanks, portable organic-free water system blanks, etc.).

- 4.1.4.2. All detections of organic and inorganic compounds will be immediately reported to the FSB QAO. The project leader will analyze the results to determine if the source of contamination can be identified. If the source of contamination cannot be determined by the project leader, the branch QAO will conduct an additional review of the results to assess the source of contamination. If the source of contamination cannot be determined, the QAO will monitor all quality control results generated by the branch and assess the data for trends of contamination.
- 4.1.4.3. If it is determined by the project leader and the FSB QAO that the contamination adversely impacts the data collected during the investigation, the project leader will report the results to their Section Supervisor and the QAM. The project leader, in consultation with management, will determine whether the impacted data are usable or should be rejected. If data are rejected, the project leader and their management will determine whether samples must be recollected.
- 4.1.4.4. Data reported to the QAM will be analyzed to determine if the contamination is due to non-conforming work. If it is determined by the QAM, in consultation with management, that the contamination is due to non-conforming work, a corrective action is warranted and will be selected and implemented in a timely manner. If a corrective action is required, it must be implemented and reported according to the LSASD Operating Procedure for Corrective Action, Improvements, and Risk (LSASDPROC-1005, current version). If contamination is not due to non-conforming field work, then the source of contamination will be identified, if possible, and documented by the QAM. If the source of contamination cannot be determined, QAM will monitor all quality control results generated by LSASD and assess the data for trends of contamination.
- 4.1.4.4.1. Quality Assurance Reports
- 4.1.4.4.1.1. It is each project leader's responsibility to ensure that a copy of the quality assurance data from each field investigation report is provided to the FSB QAO, who will compile a quarterly report of field quality assurance data and forward the report to the QAM.
- 4.1.4.4.1.2. The QAM will prepare an annual quality assurance report based on the quarterly reports. This report will be distributed to all field investigators each year and will document and discuss all quality control issues or trends identified during the data review. This report will be retained by the QAM to document that QC measures have been taken, that the QC measures are appropriate, that the QC results are acceptable or, if not, that corrective actions were taken.

References

LSASD Safety, Health and Environmental Management Program (SHEMP) Manual, Current Version

LSASD Operating Procedure for Sample and Evidence Management (LSASDPROC-005, Current Version)

LSASD Training and Demonstration of Competency (LSASDPROC-1003, Current Version)

LSASD Operating Procedure for Equipment Inventory and Management (LSASDPROC-1009, Current Version)

LSASD Operating Procedure for Sediment Sampling (LSASDPROC-200, Current Version)

LSASD Operating Procedure for Field Equipment Cleaning and Decontamination (LSASDPROC-205, Current Version)

LSASD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (ASBPROC-206, Current Version)

LSASD Operating Procedure for Soil Sampling (LSASDPROC-300, Current Version)

LSASD Operating Procedure for Waste Sampling (LSASDPROC-302, Current Version)

USEPA Region 4 Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM), November 2001

USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM), Current Version

Loan-In Form (LSASDFORM-011, Current Version)

LSASD Operating Procedure for Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples (LSASDPROC-209, Current Version)

LSASD Operating Procedure for Corrective Action, Improvements, and Risk (LSASDPROC-1005, Current Version)

SESD Guidance for Design and Installation of Monitoring Wells (SESDGUID-101, Current Version)

Revision History

History	Effective Date
Replaced Chief with Supervisor; General formatting revisions.	April 22, 2023
LSASDPROC-011-R6, Field Sampling Quality Control, replaces SESDPROC-011-R5 Changed SESD reference to LSASD throughout. SOP put into the new SOP format. Updated several references and made several editorial changes.	November 12, 2021
SESDPROC-011-R5, Field Sampling Quality Control, replaces SESDPROC-011-R4 Cover Page: SESD's reorganization was reflected in the authorization section by making John Deatrick the Supervisor of the Field Services Branch. The QAM was changed from Bobby Lewis to Hunter Johnson. Revision History: Changes were made to reflect the current practice of only including the most recent changes in the revision history. General: Corrected any typographical, grammatical and/or editorial errors. Changed name of Enforcement and Investigations Branch to Field Services Branch. Removed references to Ecological Assessment Branch. Added Section 2.9 to the Table of Contents. Section 1.4.6: Added definition for Organic-Free Water Section 3.5: Changed volume needed for soil MS/MSD samples from triple to double volume. Section 4.1: Modified statement to read: Each lot of chemical preservative will be tested for the appropriate analytes by either FEC Staff or the Branch QAO.	April 26, 2017
SESDPROC-011-R4, Field Sampling Quality Control, replaces SESDPROC-011-R3	February 5, 2013
SESDPROC-011-R3, Field Sampling Quality Control, replaces SESDPROC-011-R2	October 15, 2010
SESDPROC-011-R2, Field Sampling Quality Control, replaces SESDPROC-011-R1	January 28, 2008
SESDPROC-011-R1, Field Sampling Quality Control, replaces SESDPROC-011-R0.	October 19, 2007
SESDPROC-011-R0, Field Sampling Quality Control, Original Issue	February 5, 2007

Region 4 U.S. Environmental Protection Agency Laboratory Services and Applied Science Division Athens, Georgia	
Operating Procedure	
Title: Packing, Marking, Labeling and Shipping of Environmental and Waste Samples	ID: FSBPROC-209-R6
Issuing Authority: Field Services Branch Chief	
Effective Date: May 31, 2024	Next Review Date: May 31, 2028
Method Reference: N/A	SOP Author: Paula Whiting

Purpose

Regulations for packing, marking, labeling, and shipping of dangerous goods by air transport are promulgated by Department of Transportation under 49 CFR, Subchapter C, Hazardous Materials Regulations, and the International Air Transport Authority (IATA), which is equivalent to United Nations International Civil Aviation Organization (UN/ICAO). Transportation of hazardous materials (dangerous goods) by EPA personnel is covered by EPA Order 1000. This document describes general and specific procedures, methods, and considerations to be used and observed by LSASD field investigators when packing, marking, labeling, and shipping environmental and waste samples to ensure that all shipments are in compliance with the above regulations and guidance.

Scope/Application

The procedures contained in this document are to be used by field personnel when packing, marking, labeling, and shipping environmental samples and dangerous goods by air transport. Samples collected during field investigations or in response to a hazardous materials incident must be classified prior to shipment, as either environmental or hazardous materials (dangerous goods) samples.

In general, environmental samples include drinking water, most groundwater and ambient surface water, soil, sediment, treated municipal and industrial wastewater effluent, biological specimens, or any samples not expected to be contaminated with high levels of hazardous materials. Samples collected from process wastewater streams, drums, bulk storage tanks, soil, sediment, or water samples from areas suspected of being highly contaminated may require shipment as dangerous goods.

Government employees transporting samples or hazardous materials (i.e., preservatives or waste samples) in government vehicles are not subject to the requirements of this section in accordance with 49 CFR 171.1(d)(5). EPA contractors, however, are not covered by this exemption and may not transport these materials without full compliance with 49 CFR. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

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1 General Information

1.1 Documentation/Verification

This procedure was prepared by persons deemed technically competent by LSASD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the LSASD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.2 General Precautions

1.2.1 Safety

Proper safety precautions must be observed when packing, marking, labeling, and shipping environmental or waste samples. Refer to the LSASD Safety, Health, and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Minimally, gloves and safety glasses should be utilized when completing work covered in this operating procedure.

1.2.2 Training

Employees required to handle samples to be shipped as dangerous goods shall be trained in DOT hazardous materials regulations for bulk or non-bulk ground shipments and IATA Dangerous Goods Regulations (DGR) to ship hazmat/dangerous goods by passenger and/or cargo aircraft as required in the 49 CFR Hazardous Materials Regulations (HMR). The employees shall receive certifications in Hazmat Ground Shipper Certification (DOT) and Hazmat Air Shipper Certification (IATA). Under DOT rules, all hazmat employee training must be repeated at least every three years. For IATA, training shall be repeated every two years.

2 Shipment of Dangerous Goods

2.1 The project leader is responsible for determining if samples collected during a specific field investigation meet the definitions for dangerous goods. If a sample is collected of a material that is listed in the Dangerous Goods List, Section 4.2, IATA, then that sample must be identified, packaged, marked, labeled, and shipped according to the instructions given for that material. If the composition of the collected sample(s) is unknown, and the project leader knows or suspects that it is a regulated material (dangerous goods), the sample may not be offered for air transport. If the composition and properties of the waste sample or highly

contaminated soil, sediment, or water sample are unknown, or only partially known, the sample may not be offered for air transport.

In addition, the shipment of pre-preserved sample containers or bottles of preservatives (e.g., NaOH pellets, HCL, etc.) which are designated as dangerous goods by IATA is regulated. Shipment of nitric acid (HNO₃) is strictly regulated. Consult the IATA Dangerous Goods Regulations for guidance. ***Dangerous goods must not be offered for air transport by any personnel except LSASD's dangerous goods shipment designee or other personnel trained and certified by IATA in dangerous goods shipment.***

3 Shipment of Environmental Samples

3.1 Guidance for the shipment of environmental laboratory samples by personnel is provided in a memorandum dated March 6, 1981, subject "Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Laboratory Samples". By this memorandum, the shipment of the following unpreserved samples is not regulated:

- 3.1.1 Drinking water
- 3.1.2 Treated effluent
- 3.1.3 Biological specimens
- 3.1.4 Sediment
- 3.1.5 Water treatment plant sludge
- 3.1.6 POTW sludge

3.2 In addition, the shipment of the following preserved samples is not regulated, provided the amount of preservative used does not exceed the amounts found in 40 CFR 136.3 or the USEPA Region 4 Laboratory Services Branch Laboratory Operations and Quality Assurance Manual (LOQAM), Most Recent Version. This provision is also discussed in correspondence between DOT and EPA (Department of Transportation, Letter from Edward T. Mazzullo, Director, Office of Hazardous Materials Standards, to Henry L. Longest II, Acting Assistant Administrator, USEPA, Ref No.: 02-0093, February 13, 2003). It is the shipper's (individual signing the air waybill) responsibility to ensure that proper amounts of preservative are used:

- 3.2.1 Drinking water
- 3.2.2 Ambient water
- 3.2.3 Treated effluent
- 3.2.4 Biological specimens
- 3.2.5 Sediment
- 3.2.6 Wastewater treatment plant sludge
- 3.2.7 Water treatment plant sludge

- 3.3** Samples determined by the project leader to be in these categories are to be shipped using the following protocol, developed jointly between USEPA, OSHA, and DOT. This procedure is documented in the "Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Environmental Laboratory Samples."
- 3.4** Untreated wastewater and sludge from Publicly Owned Treatment Works (POTWs) are considered to be "diagnostic specimens" (not environmental laboratory samples). However, because they are not considered to be etiologic agents (infectious) they are not restricted and may be shipped using the procedures outlined below.
- 3.5** Environmental samples should be packed prior to shipment by air using the following procedures:
- 3.5.1** Allow sufficient headspace (ullage) in all bottles (except VOA containers with a septum seal) to compensate for any pressure and temperature changes (approximately 10 percent of the volume of the container).
 - 3.5.2** Ensure that the lids on all bottles are tight (will not leak).
 - 3.5.3** Place bottles in separate and appropriately sized polyethylene bags and seal the bags. If available, the use of Whirl-Pak bags is preferable; if unavailable, seal regular bags with tape (plastic electrical tape).
 - 3.5.4** Select a sturdy cooler in good repair. Secure and tape the drain plug with fiber or duct tape inside and outside. Line the cooler with a large heavy duty plastic bag.
 - 3.5.5** Place cushioning/absorbent material in the bottom of the cooler and then place the containers in the cooler with sufficient space to allow for the addition of cushioning between the containers.
 - 3.5.6** If required by the method for preservation, put "blue ice" (or ice that has been "double bagged" in heavy duty polyethylene bags and properly sealed) on top of and/or between the containers. Fill all remaining space between the containers with absorbent material.
 - 3.5.7.** If the samples are preserved with ice, include a temperature blank for the laboratory to verify that the samples are received at the appropriate temperature.
 - 3.5.8** Securely fasten the top of the large garbage bag with tape (preferably plastic electrical tape).

- 3.5.9** Place the Chain-of-Custody Record or the CLP Traffic Report Form (if applicable) into a plastic bag and tape the bag to the inner side of the cooler lid.
- 3.5.10** Close the cooler and securely tape (preferably with fiber tape) the top of the cooler shut. Chain-of-custody seals should be affixed to the top and sides of the cooler within the securing tape so that the cooler cannot be opened without breaking the seal.

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4 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version.

Title 40 Code of Federal Regulations (CFR), Pt. 136.3, Identification of Test Procedures, July 1, 2001. See Table II, Footnote 3.

Title 49 CFR, Pt. 171.1(d)(5), Applicability of Hazardous Materials Regulations (HMR) to Persons and Functions.

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US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 LSASD Athens, GA, Most Recent Version.

5 Revision History

This table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the LSASD Quality Assurance Coordinator on the LSASD local area network (LAN).

History	Effective Date
<p>FSBPROC-209-R6, <i>Packing, Marking, Labeling and Shipping of Environmental and Waste Samples</i>, replaces LSASDPROC-209-R5</p> <p>General: Corrected any typographical, grammatical, and/or editorial errors. Section 1.2.1: Included recommended PPE. Section 1.2.2: New section added to include relevant training requirements.</p>	<p>May 31, 2024</p>
<p>Replaced Chief with Supervisor; General formatting revisions.</p>	<p>April 22, 2023</p>
<p>LSASDPROC-209-R4 <i>Packing, Marking, Labeling and Shipping of Environmental and Waste Samples</i>, replaces LSASDPROC-209-R3</p> <p>Reformatted document to Divisional Format</p>	<p>February 23, 2020</p>
<p>LSASDPROC-209-R3, <i>Packing, Marking, Labeling and Shipping of Environmental and Waste Samples</i>, replaces LSASDPROC-209-R2.</p> <p>Cover Page: Changes made to reflect reorganization of LSASD from two field branches to one: John Deatrick listed as the Chief, Field Services Branch. The FQM was changed from Liza Montalvo to Hunter Johnson.</p> <p>Revision History: Changes were made to reflect the current practice of only including the most recent changes in the revision history.</p>	<p>February 4, 2015</p>
<p>LSASDPROC-209-R2, <i>Packing, Marking, Labeling and Shipping of Environmental and Waste Samples</i>, replaces LSASDPROC-209-R1.</p>	<p>April 20, 2011</p>
<p>LSASDPROC-209-R1, <i>Packing, Marking, Labeling and Shipping of Environmental and Waste Samples</i>, replaces LSASDPROC-209-R0.</p>	<p>November 1, 2007</p>
<p>LSASDPROC-209-R0, <i>Packing, Marking, Labeling and Shipping of Environmental and Waste Samples</i>, Original Issue</p>	<p>February 05, 2007</p>

Region 4 U.S. Environmental Protection Agency Laboratory Services and Applied Science Division Athens, Georgia	
Operating Procedure	
Title: Field Equipment Cleaning and Decontamination	ID: LSASDPROC-205-R4
Issuing Authority: LSASD Field Branch Chief	
Effective Date: June 22, 2020	Review Due Date: June 22, 2023

Purpose

This procedure is to be used by Region 4 Laboratory Services and Applied Science Division staff . This document describes general and specific procedures, methods and considerations to be used and observed when cleaning and decontaminating sampling equipment during the course of field investigations. This procedure is to be used by all Region 4 Laboratory Services and Applied Science Division (LSASD) staff.

Scope/Application

The procedures contained in this document are to be followed when field cleaning sampling equipment, for both re-use in the field, as well as used equipment being returned to the Field Equipment Center (FEC). On the occasion that LSASD field investigators determine that any of the procedures described in this section are either inappropriate, inadequate or impractical and that other procedures must be used to clean or decontaminate sampling equipment at a particular site, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

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1 General Information

1.1 Documentation/Verification

This procedure was prepared by persons deemed technically competent by LSASD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the LSASD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on LAN and for maintaining records of review conducted prior to its issuance.

1.2 Definitions

- Decontamination: The process of cleaning dirty sampling equipment to the degree to which it can be re-used, with appropriate QA/QC, in the field.
- Deionized water: Tap water that has been treated by passing through a standard deionizing resin column. At a minimum, the finished water should contain no detectable heavy metals or other inorganic compounds (i.e., at or above analytical detection limits) as defined by a standard inductively coupled Argon Plasma Spectrophotometer (ICP) (or equivalent) scan. Deionized water obtained by other methods is acceptable, as long as it meets the above analytical criteria. Organic-free water may be substituted for deionized water.
- Detergent shall be a standard brand of phosphate-free laboratory detergent such as Liquinox® or Luminox®. Liquinox® is a traditional anionic laboratory detergent and is used for general cleaning and where there is concern for the stability of the cleaned items in harsher cleaners. Luminox® is a specialized detergent with the capability of removing oils and organic contamination. It is used in lieu of a solvent rinse step in cleaning of equipment for trace contaminant sampling. Where not specified in these procedures, either detergent is acceptable.
- Drilling Equipment: All power equipment used to collect surface and sub-surface soil samples or install wells. For purposes of this procedure, direct push is also included in this definition.
- Field Cleaning: The process of cleaning dirty sampling equipment such that it can be returned to the FEC in a condition that will minimize the risk of transfer of contaminants from a site.
- Organic-free water: Tap water that has been treated with activated carbon and deionizing units. At a minimum, the finished water must meet the analytical criteria of deionized water and it should contain no detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels as determined by the Region 4 laboratory for a given set of analyses. Organic-free water obtained by other methods is acceptable, as long as it meets the above analytical criteria.
- Tap water: Water from any potable water supply. Deionized water or organic-free water may be substituted for tap water.

1.3 General Precautions

1.3.1 Safety

Proper safety precautions must be observed when field cleaning or decontaminating dirty sampling equipment. Refer to the LSASD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate. At a minimum, the following precautions should be taken in the field during these cleaning operations:

- When conducting field cleaning or decontamination using laboratory detergent, safety glasses with splash shields or goggles, and latex gloves will be worn.
- No eating, smoking, drinking, chewing, or any hand to mouth contact should be permitted during cleaning operations.

1.3.2 Procedural Precaution

Prior to mobilization to a site, the expected types of contamination should be evaluated to determine if the field cleaning and decontamination activities will generate rinses and other waste waters that might be considered RCRA hazardous waste or may require special handling.

2 Introduction to Field Equipment Cleaning and Decontamination

2.1 General

The procedures outlined in this document are intended for use by field investigators for cleaning and decontaminating sampling and other equipment in the field. These procedures should be followed in order that equipment is returned to the FEC in a condition that will minimize the risk of transfer of contaminants from a site.

Sampling and field equipment cleaned in accordance with these procedures must meet the minimum requirements for the Data Quality Objectives (DQOs) of the study or investigation. If deviations from these procedures need to be made during the course of the field investigation, they will be documented in the field logbook along with a description of the circumstances requiring the use of the variant procedure.

Cleaning procedures for use at the Field Equipment Center (FEC) are found in LSASD Operating Procedure for Equipment Cleaning and Decontamination at the FEC (LSASDPROC-206).

2.2 Handling Practices and Containers for Cleaning Solutions

Improperly handled cleaning solutions may easily become contaminated. Storage and application containers must be constructed of the proper materials to ensure their integrity. Following are acceptable materials used for containing the specified cleaning solutions:

- Detergent must be kept in clean plastic, metal, or glass containers until used. It should be poured directly from the container during use.
- Tap water may be kept in tanks, hand pressure sprayers, squeeze bottles, or applied directly from a hose.
- Deionized water must be stored in clean, glass or plastic containers that can be closed for transport. It can be applied from plastic squeeze bottles.
- Organic-free water must be stored in clean glass or Teflon® containers prior to use. It may be applied using Teflon® squeeze bottles, or with the portable system.

2.3 Disposal of Cleaning Solutions

Procedures for the safe handling and disposition of investigation derived waste (IDW); including used wash water and rinse water are in LSASD Operating Procedure for Management of Investigation Derived Waste (LSASDPROC-202).

2.4 Sample Collection Equipment Contaminated with Concentrated Materials

Equipment used to collect samples of concentrated materials from investigation sites must be field cleaned before returning from the study. At a minimum, this should consist of washing with detergent and rinsing with tap water. When the above procedure cannot be followed, the following options are acceptable:

- Leave with facility for proper disposal;
- If possible, containerize, seal, and secure the equipment and leave on-site for later disposal;
- Containerize, bag, or seal the equipment so that no odor is detected and return to the Field Equipment Center.

It is the project leader's responsibility to evaluate the nature of the sampled material and determine the most appropriate cleaning procedures for the equipment used to sample that material.

2.5 Sample Collection Equipment Contaminated with Environmental Media

Equipment used to collect samples of environmental media from investigation sites should be field cleaned before returning from the study. Based on the condition of the sampling equipment, one or more of the following options must be used for field cleaning:

- Wipe the equipment clean;
- Water-rinse the equipment;
- Wash the equipment in detergent and water followed by a tap water rinse.
- For grossly contaminated equipment, the procedures set forth in Section 2.4 must be followed.

Under extenuating circumstances such as facility limitations, regulatory limitations, or during residential sampling investigations where field cleaning operations are not feasible, equipment can be containerized, bagged or sealed so that no odor is detected and returned to the FEC without being field cleaned. If possible, FEC personnel should be notified that equipment will be returned without being field cleaned. It is the project leader's responsibility to evaluate the nature of the sampled material and determine the most appropriate cleaning procedures for the equipment used to sample that material.

2.6 Handling of Decontaminated Equipment

After decontamination, equipment should be handled only by personnel wearing clean gloves to prevent re-contamination. In addition, the equipment should be moved away (preferably upwind) from the decontamination area to prevent re-contamination. If the equipment is not to be immediately re-used, it should be covered with plastic sheeting or wrapped in aluminum foil to prevent re-contamination. The area where the equipment is kept prior to re-use must be free of contaminants.

3 Field Equipment Decontamination Procedures

3.1 General

Sufficient equipment should be transported to the field so that an entire study can be conducted without the need for decontamination. When equipment must be decontaminated in the field, the following procedures are to be utilized.

Note: Equipment utilized for PFAS sampling will not be cleaned in the field.

3.2 Specifications for Decontamination Pads

Decontamination pads constructed for field cleaning of sampling and drilling equipment should meet the following minimum specifications:

- The pad should be constructed in an area known or believed to be free of surface contamination.
- The pad should not leak.
- If possible, the pad should be constructed on a level, paved surface and should facilitate the removal of wastewater. This may be accomplished by either constructing the pad with one corner lower than the rest, or by creating a sump or pit in one corner or along one side. Any sump or pit should also be lined.
- Sawhorses or racks constructed to hold equipment while being cleaned should be high enough above ground to prevent equipment from being splashed.
- Water should be removed from the decontamination pad frequently.
- A temporary pad should be lined with a water impermeable material with no seams within the pad. This material should be either easily replaced (disposable) or repairable.

At the completion of site activities, the decontamination pad should be deactivated. The pit or sump should be backfilled with the appropriate material designated by the site project leader, but only after all waste/rinse water has been pumped into containers for disposal. See LSASD Operating Procedure for Management of Investigation Derived Waste (LSASDPROC-202) for proper handling and disposal of these materials. If the decontamination pad has leaked excessively, soil sampling may be required.

3.3 "Classical Parameter" Sampling Equipment

"Classical Parameters" are analyses such as oxygen demand, nutrients, certain inorganic compounds, sulfide, flow measurements, etc. For routine operations involving classical parameter analyses, water quality sampling equipment such as Kemmerers, buckets, dissolved oxygen dunkers, dredges, etc., may be cleaned with the sample water or tap water between sampling locations as appropriate.

Flow measuring equipment such as weirs, staff gages, velocity meters, and other stream gauging equipment may be cleaned with tap water between measuring locations, if necessary.

Note: The procedures described in Section 3.3 are not to be used for cleaning field equipment to be used for the collection of samples undergoing trace organic or inorganic constituent analyses.

3.4 Sampling Equipment used for the Collection of Trace Organic and Inorganic Compounds

For samples undergoing trace organic or inorganic constituent analyses, the following procedures are to be used for all sampling equipment or components of equipment that come in contact with the sample:

3.4.1 Standard LSASD Method

- An optional Liquinox[®] detergent wash step may be useful to remove gross dirt and soil.
- Clean with tap water and Luminox[®] detergent using a brush, if necessary, to remove particulate matter and surface films.
- Rinse thoroughly with tap water.
- Rinse thoroughly with organic-free water and place on a clean foil-wrapped surface to air-dry.
- Wrap the dry equipment with aluminum foil or bag in clean plastic. If the equipment is to be stored overnight before it is wrapped in foil, it should be covered and secured with clean, unused plastic sheeting.

3.4.2 Alternative Solvent Rinse Method

The historical solvent rinse method of cleaning equipment for trace contaminant sampling remains an acceptable method.

- Clean with tap water and Liquinox[®] detergent using a brush, if necessary, to remove particulate matter and surface films. Equipment may be steam cleaned (Liquinox[®] detergent and high-pressure hot water) as an alternative to brushing. Sampling equipment that is steam cleaned should be placed on racks or saw horses at least two feet above the floor of the decontamination pad. PVC or plastic items should not be steam cleaned.
- Rinse thoroughly with tap water.

- Rinse thoroughly with deionized water.
- Rinse with an appropriate solvent (generally isopropanol).
- Rinse with organic-free water and place on a clean foil-wrapped surface to air-dry.
- Wrap the dry equipment with aluminum foil or plastic. If the equipment is to be stored overnight before it is wrapped, it should be covered and secured with clean, unused plastic sheeting.

3.5 Well Sounders or Tapes

The following procedures are recommended for decontaminating well sounders (water level indicators) and tapes. Unless conditions warrant, it is only necessary to decontaminate the wetted portion of the sounder or tape.

- Wash with Liquinox[®] detergent and tap water.
- Rinse with tap water.
- Rinse with deionized water.

3.6 Redi-Flo2[®] Pump

CAUTION – Do not wet the controller. Always disconnect power from the pump when handling the pump body.

The Redi-Flo2[®] pump and any associated connected hardware (e.g., check valve) should be decontaminated between each monitoring well. The following procedures are required, depending on whether the pump is used solely for purging or used for purging and sampling.

3.6.1 Purge Only (Pump and Wetted Portion of Tubing or Hose)

- Disconnect power and wash exterior of pump and wetted portion of the power lead and tubing or hose with Liquinox[®] detergent and water solution.
- Rinse with tap water.
- Final rinse with deionized water.
- Place pump and reel in a clean plastic bag and keep tubing or hose contained in clean plastic or galvanized tub between uses.

3.6.2 Purge And Sample

Grundfos Redi-Flo2® pumps are extensively decontaminated and tested at the FEC to prevent contamination from being transmitted between sites. The relevant sections of LSASDPROC-206, *Field Equipment Cleaning and Decontamination at the FEC*, should be implemented in the field where a high risk of cross-contamination exists, such as where NAPL or high-concentration contaminants occur. In most cases, the abbreviated cleaning procedure described below will suffice, provided that sampling proceeds from least to most contaminated areas.

- Disconnect and discard the previously used sample tubing from the pump. Remove the check valve and tubing adapters and clean separately (See Section 3.6.3 for check valve). Wash the pump exterior with detergent and water.
- Prepare and fill three containers with decontamination solutions, consisting of Container #1, a tap water/detergent washing solution. Luminox® is commonly used. An additional pre-wash container of Liquinox® may be used; Container #2, a tap water rinsing solution; and Container #3, a deionized or organic-free water final rinsing solution. Choice of detergent and final rinsing solution for all steps in this procedure is dependent upon project objectives (analytes and compounds of interest). The containers should be large enough to hold the pump and one to two liters of solution. An array of 2' long 2" PVC pipes with bottom caps is a common arrangement. The solutions should be changed at least daily.
- Place the pump in Container #1. Turn the pump on and circulate the detergent and water solution through the pump and then turn the pump off.
- Place the pump in Container #2. Turn the pump on and circulate the tap water through the pump and then turn the pump off.
- Place the pump in Container #3. Turn the pump on and circulate deionized or organic-free water through the pump and then turn the pump off.
- Disconnect power and remove pump from Container #3. Rinse exterior and interior of pump with fresh deionized or organic-free water.
- Decontaminate the power lead by washing with detergent and water, followed by tap water and deionized water rinses. This step may be performed before washing the pump if desired.
- Reassemble check valve and tubing adapters to pump. ALWAYS use Teflon® tape to prevent galling of threads. Firm hand-tightening of fittings or light wrench torque is generally adequate.
- Place the pump and reel in a clean plastic bag.

3.6.3 Redi-Flo2® Ball Check Valve

- Remove the ball check valve from the pump head. Check for wear and/or corrosion, and replace as needed. During decontamination check for free-flow in forward direction and blocking of flow in reverse direction.
- Using a brush, scrub all components with detergent and tap water.

- Rinse with deionized water.
- Rethread the ball check valve to the Redi-Flo2® pump head.

3.7 Mega-Monsoon® and GeoSub® Electric Submersible Pump

As these pumps have lower velocities in the turbine section and are easier to disassemble in the field than Grundfos pumps, the outer pump housing should be removed to expose the impeller for cleaning prior to use and between each use when used as a sampling pump for trace contaminant sampling.

- Remove check valves and adapter fittings and clean separately.
- Remove the outer motor housing by holding the top of the pump head and unscrewing the outer housing from its O-ring sealed seat.
- Clean all pump components per the provisions of section 3.4. Use a small bottle brush for the pump head passages
- Wet the O-ring(s) on the pump head with organic-free water. Reassemble the outer pump housing to the pump head.
- Clean cable and reel per Section 3.4.
- Conduct final rinse of pump with organic-free water over pump and through pump turbine.

3.8 Bladder Pumps

Bladder pumps are presumed to be intended for use as low flow purge-and-sample pumps. The Geotech® bladder pump and Geoprobe Systems® mechanical bladder pump can be cleaned similarly.

- Discard any tubing returned with the pump.
- Completely disassemble the pump, being careful to note the initial position of and retain any springs and loose ball checks.
- Discard pump bladder.
- Clean all parts as per the standard cleaning procedure in Section 3.4.
- Install a new Teflon® bladder and reassemble pump.

3.9 Downhole Drilling Equipment

While LSASD does not currently operate drilling equipment, LSASD personnel do oversee and specify drilling operations. The following procedures are to be used for drilling activities involving the collection of soil samples for trace organic and inorganic constituent analyses and for the construction of monitoring wells to be used for the collection of groundwater samples for trace organic and inorganic constituent analyses.

3.9.1 Introduction

Cleaning and decontamination of all equipment should occur at a designated area (decontamination pad) on the site. The decontamination pad should meet the specifications of Section 3.2 of this procedure.

Tap water brought on the site for drilling and cleaning purposes should be contained in a pre-cleaned tank.

A steam cleaner and/or high pressure hot water washer capable of generating a pressure of at least 2500 PSI and producing hot water and/or steam, with a detergent compartment, should be obtained.

3.9.2 Preliminary Cleaning and Inspection

Drilling equipment should be clean of any contaminants that may have been transported from off-site to minimize the potential for cross-contamination. The drilling equipment should not serve as a source of contaminants. Associated drilling and decontamination equipment, well construction materials, and equipment handling procedures should meet these minimum specified criteria:

- All downhole augering, drilling, and sampling equipment should be sandblasted before use if painted, and/or there is a buildup of rust, hard or caked matter, etc., that cannot be removed by steam cleaning (detergent and high pressure hot water), or wire brushing. Sandblasting should be performed prior to arrival on site, or well away from the decontamination pad and areas to be sampled.
- Any portion of the drilling equipment that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) should be steam cleaned (detergent and high pressure hot water) and wire brushed (as needed) to remove all rust, soil, and other material which may have come from other sites before being brought on site.
- Printing and/or writing on well casing, tremie tubing, etc., should be removed before use. Emery cloth or sand paper can be used to remove the printing and/or writing. Most well material suppliers can provide materials without the printing and/or writing if specified when ordered. Items that cannot be cleaned are not acceptable and should be discarded.
- Equipment associated with the drilling and sampling activities should be inspected to insure that all oils, greases, hydraulic fluids, etc., have been removed, and all seals and gaskets are intact with no fluid leaks.

3.9.3 Drill Rig Field Cleaning Procedure

Any portion of the drill rig, backhoe, etc., that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) should be steam cleaned (detergent and high pressure hot water) between boreholes.

3.9.4 Field Decontamination Procedure for Drilling Equipment

The following is the standard procedure for field cleaning augers, drill stems, rods, tools, and associated equipment. This procedure does not apply to well casings, well screens, or split-spoon samplers used to obtain samples for chemical analyses, which should be decontaminated as outlined in Section 3.4 of this procedure.

- Wash with tap water and detergent, using a brush if necessary, to remove particulate matter and surface films. Steam cleaning (high pressure hot water with detergent) may be necessary to remove matter that is difficult to remove with the brush. Drilling equipment that is steam cleaned should be placed on racks or saw horses at least two feet above the floor of the decontamination pad. Hollow-stem augers, drill rods, etc., that are hollow or have holes that transmit water or drilling fluids, should be cleaned on the inside with vigorous brushing.
- Rinse thoroughly with tap water.
- Remove from the decontamination pad and cover with clean, unused plastic if not used immediately. If stored overnight, the plastic should be secured to ensure that it stays in place.

3.9.5 Field Decontamination Procedure for Direct Push Technology (DPT) Equipment

- Certain specific procedures for the decontamination of DPT tools are described in the various sampling procedures, but the following general guidelines apply:
- Prior to return to the Field Equipment Center, all threaded tool joints should be broken apart and the equipment cleaned per the provisions of *Section 2.5, Sample Collection Equipment Contaminated with Environmental Media* of this procedure.
- Equipment that contacts the sample media and is cleaned in the field for reuse should be cleaned per the provisions of *Section 3.4, Sampling Equipment used for the Collection of Trace Organic and Inorganic Compounds* of this procedure. This would include piston sampler points and shoes, screen point sampler screens and sheaths, and the drive rods when used for groundwater sampling.
- Equipment that does not directly contact the sample media and is cleaned in the field for reuse can generally be cleaned per the provisions of *Section 3.7.4, Field Decontamination Procedure for Drilling Equipment* of this procedure.
- Stainless steel SP15/16 well screens require special care as the narrow slots are difficult to clean under even controlled circumstances and galvanic corrosion can release chrome from the screen surface. As soon as possible after retrieval, the screen slots should be sprayed from the outside to break loose as much material as possible before it can dry in place. To prevent galvanic corrosion, the screens must be segregated from the sampler sheaths, drive rods, and other carbon steel during return transport from the field.

3.10 Rental Pumps

Completing a groundwater sampling project may require the use of rental pumps. Rental pumps are acceptable where they are of suitable stainless steel and Teflon[®] construction. These pumps should be cleaned prior to use using the procedures specified herein and a rinse-blank collected prior to use.

4 References

LSASD Operating Procedure for Management of Investigation Derived Waste, LSASDPROC-202, Most Recent Version

LSASD Operating Procedure for Equipment Cleaning and Decontamination at the FEC, LSASDPROC-206, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 LSASD, Athens, GA, Most Recent Version

Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the LSASD Document Control Coordinator on the LSASD local area network (LAN).

History	Effective Date
<p>LSASDPROC-205-R4, <i>Field Equipment Cleaning and Decontamination</i>, replaces SESDPROC-205-R3</p> <p>General: Updated format, Division and Branch names and naming conventions post agency re-alignment.</p> <p>Section 3.1: Added note that PFAS sampling equipment will not be cleaned in the field.</p> <p>Clarified in Section 3.9 that LSASD does not performing drilling activities.</p>	June 22, 2020
<p>SESDPROC-205-R3, <i>Field Equipment Cleaning and Decontamination</i>, replaces SESDPROC-205-R2.</p> <p>Cover Page: The author was changed to Brian Striggow. LSASD's reorganization was reflected in the authorization section by making John Deatruck the Chief of the Field Services Branch. The FQM was changed from Bobby Lewis to Hunter Johnson.</p> <p>Revision History: Changes were made to reflect the current practice of only including the most recent changes in the revision history.</p> <p>General: Corrected any typographical, grammatical and/or editorial errors.</p> <p>Section 1.4: Differentiate between Liquinox® and Luminox® detergents.</p> <p>Section 3.4: Restore solvent rinse as alternative cleaning method.</p> <p>Section 3.7: Added section on cleaning of 12 Volt electric submersible pumps.</p> <p>Section 3.8: Added section on cleaning of bladder pumps.</p> <p>Section 3.9: Added language on cleaning and transport of SP15/16 screens</p> <p>Section 3.10: Added section on cleaning of rental pumps</p>	December 18, 2015
<p>SESDPROC-205-R2, <i>Field Equipment Cleaning and Decontamination</i>, replaces SESDPROC-205-R1.</p>	December 20, 2011
<p>SESDPROC-205-R1, <i>Field Equipment Cleaning and Decontamination</i>, replaces SESDPROC-205-R0.</p>	November 1, 2007

SESDFPROC-205-R0, <i>Field Equipment Cleaning and Decontamination</i> , Original Issue	February 05, 2007
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Region 4
U.S. Environmental Protection Agency
Laboratory Services and Applied Science Division
Athens, Georgia

Operating Procedure

Title: Logbooks

ID: LSASDPROC-1002-R1

Issuing Authority: LSASD Deputy Director

Review Issue Date: May 19, 2021

Next Review Due: May 19, 2025

Purpose

This procedure is specific to the Region 4 Laboratory Services and Applied Science Division (LSASD) to maintain conformance to technical and quality system requirements. This procedure defines the process for documenting direct observations in logbooks or other record formats related to laboratory analyses, field investigations, or assessments of field sampling processes and laboratory operations of external entities, such as drinking water assessments and Technical Systems Audits.

Scope/Application

The requirements of this procedure apply to all personnel who perform work under LSASD's quality system. This procedure contains requirements for documenting activities related to laboratory analyses, field investigations, or assessments of field sampling processes and laboratory operations of external entities and serves as a supplement to the overarching requirements for LSASD records, outlined in the LSASD Operating Procedure for Control of Records. While this SOP may be informative, it is not intended for and may not be directly applicable to operations in other organizations. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

Note: LSASD is migrating to a paperless organization and as a result, this SOP will allow original observations to be recorded in electronic format. Original observation to electronic format **MUST** be observation(s) recorded electronically with no intermediate use of pen to paper.

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Administrative Procedure

1.1. General Requirements

1.1.1. General requirements for LSASD logbook or other record format entries (e.g., electronic) related to laboratory analyses, field investigations, or assessments of field sampling processes and laboratory operations of external entities, such as drinking water assessments and Technical Systems Audits are presented in this SOP.

1.1.2. LSASD recognizes the following electronic formats to be used for recording original observations.

- Laptop computers,
- Tablets,
- Transfer software from balances to laptops/tablets,
- Element®,
- Use of thumb drives to transfer data from lab instruments to Element®,
- Excel Spreadsheets; and,
- Commercially available software specific for this purpose.

1.1.3. Recording of Original Observations

1.1.3.1. The following procedure should be used when recording original observation electronically or to paper logbooks.

- Observations, data collection, and calculations will be recorded at the time they are made or performed;
- Dedicated bound hardcopy logbooks can be used, however, migration towards electronic media is preferred;
- Entries in hardcopy logbooks will be legible, containing pertinent, accurate, and inclusive documentation of project activities, calibrations, audits, field measurements/observations, method/standards/reagent preparation and laboratory analysis, etc. For field and lab audit reports, entries must be free of personal feelings or other terminology which might prove inappropriate. Stick to the facts of the observation and provide a regulatory or SOP references as applicable;
- Write in the rain pens and notebooks should be used to record all observations when working outdoors. When environmental conditions do not make it feasible to use permanent ink, entries using a non-smear lead pencil (e.g., 2H or 3H) is permitted. Upon returning from the field, the project leader will photocopy the penciled section of the logbook and certify, in writing, that the photocopied record is a true copy of the original logbook entry. The photocopy will be included in the project file;
- Entries in logbooks shall be dated and signed or initialed by staff. Electronic files should contain an electronic signature and date stamp;

- For handwritten entries, data or other information that has been entered incorrectly shall only be corrected using a single strike-through, date and initials of the person making the correction. Under no circumstances should the incorrect material be erased, made illegible or obscured so that it cannot be read. For changes or corrections made to electronic files (e.g., spreadsheets, pdf documents, etc.) changes should be documented in a comment section documenting the change along with the initials of the person making the change and dated;
- To demonstrate continuity of the work and maintain the integrity of the data collection process, pages must not be removed from any bound logbook. Electronic data shall not be deleted unless documented;
- Blank pages or spaces should not be present in logbooks. Blank areas should be lined through and initialed and dated to prevent the opportunity for back-filling;
- Hardcopy and electronic logbook pages and field books shall be paginated. The numbering format shall be “page x of y”, where “x” is the current page number and “y” is the total number of pages of the. However, if pre-paginated logbooks are used, page numbers are acceptable. The last page of any logbook should be identified as the final page before placement in the project file;
- To facilitate accurate and complete documentation of activities, LSASD-generated forms may be used. To be utilized, LSASD-generated forms must be bound prior to use and adhere to the requirements outlined in this procedure. In cases where unbound pages/forms are necessary due to project requirements or practicality, the appropriate Section Chief and Divisional Quality Control Coordinator (QAC) will determine the best course of action;
- If pre-printed adhesive labels are used in logbooks or bound forms to facilitate organization of information entry, the LSASD staff responsible for taking notes will sign the label with the signature beginning on the label and ending on the page of the logbook such that the label cannot be removed without detection;
- When direct observations are entered directly into Element, a witness or a second analyst should be present to verify all hand entries; and,
- Element® uses an audit trail for tracking modifications made to the database. The audit trail marks the data, time, and analyst’s initials of all changes.

1.2. Laboratory Logbooks and Element®

1.2.1. The following are requirements for laboratory logbooks, in addition to those established above. Each sample preparation, analysis or equipment check is maintained using logbooks in the appropriate laboratory. Active logbooks are maintained within the laboratory where the instrument or equipment is located and should be maintained with the instrument throughout its useful life. At such time the instrument is removed from service, or the logbook is deemed full, the logbook is transferred with a completed Logbook Transfer form located on the LAN at: M:\LSB\Current Documents\Forms\Branch Forms\Log Book Forms given to the QAC. The QAC will transfer the logbook to the LSASD Records Room.

1.2.1.1. Instrument Maintenance Logbooks

1.2.1.1.1. Each laboratory instrument shall have a maintenance logbook. At a minimum the following information will be included:

- Manufacture's Name
- Instrument serial/model number
- Instrument's unique name
- Software version and firmware version
- In-service date (if known)

1.2.1.1.2. Maintenance, service, and repair records are maintained in these logbooks. Preventive maintenance schedules should be noted in the log if known. When service or maintenance is performed and completed by a vendor or analyst, the analyst should place a copy of the vendor's documentation or transcribe the details for the work that was performed on the instrument in the logbook. Each analyst that performs maintenance or repairs must record a description of the work performed to include the date, parts installed, and lot numbers. The original work order invoice should be provided to the Program Services Section for payment. Instrument maintenance logbooks are purchased as bound record books that contain pre-numbered pages.

1.2.1.2. Instrument Logbooks

1.2.1.2.1. Equipment Logbooks, such as balance logs, are maintained in the lab near the equipment. At a minimum the following information must be included:

- Instrument's unique name
- Date of analysis
- Analyst and samples/QC which have been analyzed
- A reference or a record of which options or analytical conditions were used for analysis
- Method reference
- Where appropriate, instrument acceptance criteria (e.g., tune criteria, sensitivity checks)
- Include the calibration date of the instrument and acceptance criteria for the calibration.

1.2.1.3. Preparation Logbooks

- Analyst's name.
- Weights.
- Volumes.
- Lot number of digestion tubes.
- ID of any preparation equipment used (pipettes, pipettors, balances, pH meters, thermometers, barometers).
- Certification dates of equipment, if applicable.
- Reagents/standards/lot# used.
- Preservation checks including Element ID of the pH paper used for the checks.
- Units.
- Cleanup procedures used and calibration of clean-up units (GPC).
- Project Number/Name and Workorder Number will be included on each page.
- Samples/QC which have been prepared.
- Electronic traceability via Element® is used for documenting standard preparation. Element® is subject to all the requirements of this section.

1.2.1.4. Analysis Logbooks

- 1.2.1.4.1. Electronic records, including spreadsheets which contain original measurements, may be used to create logbooks if all the required information can be captured by the instrumental software; however, a sequential analysis log must still be created and maintained. This is accomplished by printing a copy of the electronic record and including it in a notebook. These sequential logs must also include failed runs, or sequences which were abandoned prior to completion. When a pre-determined number of pages has been accumulated (e.g., 50 pages), the individual records are combined into a single bound logbook and retained as specified in the LSASD Operating Procedure for Records Management. Any electronic records must accurately reflect actual analytical information. For analyses with holding times < 72 hours, or when time-critical or method-specified times are included in the analysis, the time of analysis must also be recorded.

1.2.1.5. Field Operation Logbooks

- 1.2.1.5.1. The following are requirements for field logbooks, in addition to those established in the "Section General Information" required in all Field Operations Logbooks.

1.2.1.5.2. The following information shall be included either on the front cover or the first page of all field logbooks:

- Project name
- Project location
- Project identification number
- Project leader (full name)
- Sample team leader (full name) and initials
- Sample team member(s) (full names) and initials

1.2.1.5.3. In addition, the project's unique identifier (unique identification number(s)) will be included on each page.

1.2.1.5.4. Any deviations from the quality assurance project plan (QAPP) that occur while in the field shall be noted in the logbook(s). Field logbook entries that may be considered privileged or confidential information shall be handled in accordance with the relevant sections of LSASD Operating Procedure for Control of Records. The logbooks will be placed in the LSASD project file upon transmittal of the final report to the project requestor.

1.2.1.6. Information Required for Sample Collection

1.2.1.6.1. In addition to previously listed requirements, the following information will be included in all field logbooks when samples are collected:

- References to applicable LSASD Operating Procedures for field sampling.
- Date and time of collection.
- Station identification, including GPS coordinates (non-logging GPS units), if applicable.
- Sample identification.
- Method of collection.
- Number and type of containers (describe, as needed).
- Sample collection equipment.
- LSASD equipment identification number, if applicable.
- Matrix sampled.
- Physical description of each sample.
- Sample team member duties (calibration, collection, deployment, etc.).
- Sample preservation method (e.g., indirect contact with ice), if applicable.
- Environmental conditions such as rain, wind, smoke, dust, extreme temperature, etc.
- Location of electronic data file backups, if applicable.
- Monitoring of condition of ice in coolers or sampler, if applicable.
- Date and time of sample arrival to LSASD. Storage room for samples, even if it is for initial, temporary purposes.
- Overnight courier tracking information, if applicable.
- Other pertinent information.

1.2.1.7. Information Required for Field Measurements

1.2.1.7.1. In addition to previously listed requirements, the following information will be included in all logbooks when field measurements are conducted:

- References to applicable LSASD Operating Procedures for field measurement.
- Date and time of measurement or instrument/equipment deployment.
- Sample identification, if appropriate.
- Station identification, including GPS coordinates (non-logging GPS units), if applicable.
- Sample measurement equipment.
- LSASD sample measurement equipment identification number.
- Manufacturer name, lot number and expiration date of all pH buffers and chemical standards.
- Calibration information, including before and after calibration readings.
- Meter and check information.
- Equipment deployment depth and total depth, if applicable.
- Pinger identification number and frequency for deployed equipment, if applicable.
- Date and time of retrieval for deployed equipment, if applicable.
- Matrix measured.
- Physical description of matrix.
- Measurement values for non-logging equipment.
- Ambient air temperature, where applicable.
- Environmental conditions that may adversely impact quality of measurement (e.g., rain, wind, smoke, dust, extreme temperatures), if applicable.
- Equipment or instrument maintenance performed, if applicable.
- Meter malfunctions, troubleshooting efforts and final status, if applicable.
- Location of electronic data file backups, if applicable.
- Measurement team member duties (e.g., calibration, equipment deployment, measurement, maintenance, troubleshooting, etc.).
- Other pertinent information.

1.2.1.7.2. Entry of calibration information in logbooks is only required for calibrations conducted in the field. All calibrations conducted at the LSASD Field Equipment Center or laboratory will be recorded in the appropriate equipment tracking logbook.

1.2.1.7.3. The following visual information may be included in field logbooks, as appropriate:

- Maps/sketches.
- Photographic or video-graphic log.
- Process diagrams

1.2.1.8. Quality Assurance Field/Lab Audit Documentation

1.2.1.8.1. The following are requirements for Quality Assurance (QA) field documentation, in addition to those established in Section 1.1. The following information shall be included either on the front cover or the first page of all QA field documentation:

- Audit/facility name
- Audit/facility location
- Audit leader
- Audit team members
- Audit dates
- Audit/Project Identification Number
- Program/Authority

1.2.1.8.2. In addition to previously listed requirements, the following information will be included in all audit logbooks when field/lab assessments are conducted:

- Analytical method or field procedure reference.
- Lab Analyst/Field Sampler(s) being assessed.
- Document findings with reference, opportunities for improvement, and commendations for good work.
- Other pertinent information.

1.2.2. In addition to logbooks, QA uses checklists to document audits of methods and procedures during the onsite evaluation of laboratories and facilities. These checklists are printed and/or used electronically to document field observations. These electronic checklists are located on the LSASD LAN and updated periodically as changes are needed.

1.2.2.1. Other Logbooks and Documentation

1.2.2.1.1. Some methods and measurements do not use instrumentation to generate a result. For these methods LSASD relies on spreadsheets or other calculating software for recording/documenting original observations made (e.g., weights). All spreadsheets or other calculating software used as logbooks or used in support of data generation will be validated and controlled. All cells, except informational input cells, will be locked to prevent alteration of a formula or essential static information, such as the unique identifier. All calculations in electronic spreadsheets and calculating software files will be hand-validated by the responsible party and submitted through the Section Chief to the appropriate System Manager for approval and posting. The entire spreadsheet or software-generated electronic file will be password protected, which will be assigned by the System Manager at the time of posting on the LAN.

1.3. Definitions

None

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1.4. References

U.S. EPA, Region 4, LSASD, Analytical Services Branch, Logbook Transfer Form.

U.S. EPA, Region 4, LSASD, LSASD Field Branches Quality Management Plan, LSASDPLAN-001, most recent version.

U.S. EPA, Region 4, LSASD, ASB, Laboratory Operations and Quality Assurance Manual, most recent version.

U.S. EPA, Region 4, LSASD, Records Management Standard Operating Procedures, most recent version.

U.S. EPA, Region 4, LSASD, Quality Management Plan, most recent version.

U.S. EPA, Region 4, LSASD, LSASD Operating Procedure for Control of Records, LSASDPROC-002, most recent version.

ISO/IEC 17025: General Testing for the Competence of Testing and Calibration Laboratories, 2nd Ed., 2005, 05/15/2015, Switzerland.

ANAB, ISO/IEC 17025: Accreditation Requirements for Forensic Testing Laboratories, pp. 49, Document Number MA 3011, Effective Date: 02/02/2015.

Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the LSASD Document Control Coordinator.

History	Effective Date
LSASDPROC-1002-R0, Logbooks, Original Issue	October 1, 2017
Document put in the new SOP format. Since LSASD is instituting electronic files, this SOP has been modified to include using electronic devices and Element in place of hardcopy logbooks, files, etc.	5/19/21

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Field Site Location:	Ascend Performance Materials Operations, LLC 1515 Highway 246 South Greenwood, South Carolina 29646
Activity Description:	<ul style="list-style-type: none"> • Previous routine groundwater monitoring has involved sampling with passive diffusion bags (PDBs) and sampling from a spigot, as opposed to conventional purge-and-sample methods that allow for collection accurate measurement and of the treatability study parameters. Where PDBs are deployed or where samples were previously collected via spigot, VOC samples will be collected in the same manner to remain consistent with previous routine monitoring events. Samples for treatability studies analyses will be collected using a peristaltic or downhole pump following low-flow purging techniques. • Sample collection for the pH buffering assessment and abiotic panel testing involves direct push technology. • Surface water samples will be collected using a 1-liter amber glass container. The amber will be carefully submerged 4-6 inches (where possible) below the water surface with the cap left secure on the bottle. • Sediment interstitial (pore) water samples will be collected co-located to surface water samples using PushPoint™ samplers from the top 6 inches of the sediment column. • Investigation-derived wastes include decontamination fluids and purge water. These waste streams will be placed through the groundwater extraction system, which conveys wastewater through underground piping to the Ascend facility wastewater system prior to discharge to the Greenwood Metropolitan Sewer District Publicly Owned Treatment Works.
Field Date(s):	6/24/2024 - 12/31/2024

A field safety plan serves as a tool to document the hazard assessment, communication plan, emergency procedures, and required training.

This plan should not only identify hazards but indicate how they will be addressed and mitigated. For any identified hazards indicate what steps will be taken to minimize the risk to participants.

Steps to prepare for field work:

1. Complete the field safety checklist.
2. Complete appropriate training for your site and operations (heat illness, first aid, etc.)
3. Hold a pre-trip meeting to review your field safety checklist, travel logistics, pack list (including first aid kit), etc. and cover any remaining training needs.
4. Add appropriate Activity Hazard Analysis documents (if necessary).
5. Review the checklist with your local or regional health and safety representative.

Site Information	
Geographic Location	1515 Hwy 246 S, Greenwood, SC 29646 Coordinates (Latitude, Longitude): 34.232241215084336, -82.05275036022958 Link to online map: Ascend Performance Materials Operations
Site Information	The Site encompasses approximately 410 acres extending from Highway 246 South to Lake Greenwood. The Site was originally developed by Monsanto Chemical Company in September 1960 for manufacturing bulk continuous filament, industrial fibers, and polymer flake. Site improvements include two primary manufacturing areas (referred to as the North and South Plants), several warehouse buildings, a process water holding basin, railroad spur, and employee parking areas. Two streams originating off-Site meander through the Site: North Creek, which flows in an easterly direction between the northern property boundary and the facility; and South Creek, which flows to the north along the eastern boundary of the facility operation. The operational areas of concern include the former Burn Pit (“BP”) Area and the Construction Debris Site (“CDS”).

Travel to Site	Field trucks will be used to travel from the Montrose Atlanta office to site location.		
Wildlife	Ticks, wasps, ants, mosquitos, snakes and small mammalian wildlife. (spray and general bug spray kept inside trucks and used as needed.		
Go/No Go Criteria	Severe rain or thunderstorms, or high velocity in the creeks that would preclude safe sampling conditions.		
Site Access	Site access will be given through the perimeter gates. Montrose and its subcontractors are required by Ascend to complete a safety orientation prior to entry into the facility.		
Expected Weather	Expected high heat and humidity with a chance of thunderstorms.		
Drinking Water Availability	<i>If forecast exceeds 80°, Access to at least one quart (4 cups) per person per hour for the entire shift, i.e., an 8-hour shift requires 2 gallons per person. Water must be fresh and suitably cool.</i> <input type="checkbox"/> Plumbed water available <input checked="" type="checkbox"/> Water cooler with ice to be provided <input checked="" type="checkbox"/> Bottled water provided <input type="checkbox"/> Other:		
Access to Shade/Shelter	<i>If forecast exceeds 80°, shade must be provided by any natural or artificial means for rest breaks. Shade is not considered adequate when heat in the area does not allow the body to cool (e.g., sitting in a hot car).</i> <input type="checkbox"/> Building structures <input checked="" type="checkbox"/> Trees <input type="checkbox"/> Temporary Canopy/Tarp <input checked="" type="checkbox"/> Vehicle with A/C <input type="checkbox"/> Other:		
High Heat Procedures	<i>Required when temperatures are expected to exceed 95° F. If possible, limit strenuous tasks to morning or late afternoon hours. Rest breaks in shade must be provided at least 15 minutes every 2 hours (or more if needed). Effective means of communication, observation, and monitoring for signs of heat illness are required at all times. Pre-operations meeting required.</i> <input checked="" type="checkbox"/> Direct supervision <input checked="" type="checkbox"/> Buddy system <input type="checkbox"/> Reliable cell or radio contact <input type="checkbox"/> Other:		
Emergency Services and Contact Information			
Client Contact:	Rob Redfield rredfi@ascendmaterials.com	Montrose Contact:	Alex Testoff. 443-745-6247 atestoff@montrose-env.com
Nearest Emergency Department (ED)	Emergency Care at Self Regional Medical Center, 864.725.4111 1325 Spring Street, Greenwood, SC 29646		
Non-Emergency Medical Support	Axiom Medical – 1-877-502-9466 Please notify your PM and/or People Manager after calling to give them an update of the situation.		
Cell Phone Coverage	Device carried? <input checked="" type="checkbox"/> yes <input type="checkbox"/> no Type: Multiple Coverage: Multiple Nearest location with coverage: Multiple	Satellite device (phone or locator)	Device carried? <input type="checkbox"/> yes <input checked="" type="checkbox"/> no Type: Coverage: Nearest location with coverage:
Nearby Facilities	Restrooms are available at the facility.		
Participant Information			
Field Team/ Participants	<i>Primary Field Team Leader: Alex Testoff, 443.745.6247 Secondary Field Team Leader: Joe Terry, 813.943.8633</i> <input checked="" type="checkbox"/> Field Team/Participant list is attached as training documentation <i>Is anyone working alone?</i> <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No		
Physical Demands	Walking and standing for long periods of time. When applicable, strength to carry 70lbs of free weight, wading into water.		
First Aid Training	First aid training is required for at least one Montrose representative on-site.		
Equipment and Activities			

Fieldwork Transportation	Field vehicle non-commercial (DOT requirements are not applicable).
Project Activities (Attach AHA's to this document for tasks requiring them)	Mobilizing to and from site; direct push sampling; monitoring well sampling; surface water sampling; porewater sampling; disposal of IDW onsite.
Project Hazards	Potential hazards include heat illness, work in/near water, wildlife attack in the form of small animals, insects or snake bites. Repetitive motion (standing/certain equipment). Electrical hazards from sampling equipment.
Required Tools	Hand tools may be required for miscellaneous tasks.
Personal Protective Equipment	Required —Safety toed boots, safety glasses, hardhat (if overhead hazard exists), safety vest (ANSI Class II), flotation devices; cut resistant gloves when handling glassware or handtools Recommended —Permethrin treated clothing
Safety Equipment	Required – First aid kit; fire extinguisher Recommended – NA

First Aid Reference – Signs & Symptoms of Heat Illness		
Signs & Symptoms	Treatment	Response Action:
HEAT EXHAUSTION <ul style="list-style-type: none"> • Dizziness, headache • Rapid heart rate • Pale, cool, clammy, or flushed skin • Nausea and/or vomiting • Fatigue, thirst, muscle cramps 	<ol style="list-style-type: none"> 1. Stop all exertion. 2. Move to a cool shaded place. 3. Hydrate with cool water. 	Heat exhaustion is the most common type of heat illness. Initiate treatment. If no improvement, call Axiom at 1-877-502-9466 and seek medical help. Do not return to work in the sun. Heat exhaustion can progress to heat stroke.
HEAT STROKE <ul style="list-style-type: none"> • Disoriented, irritable, combative, unconscious • Hallucinations, seizures, poor balance • Rapid heart rate • Hot, dry, and red skin • Fever, body temperature above 104 °F 	<ol style="list-style-type: none"> 1. Move (gently) to a cooler spot in shade. 2. Loosen clothing and spray clothes and exposed skin with water and fan. 3. Cool by placing ice or cold packs along neck, chest, armpits, and groin (Do not place ice directly on skin) 	Call 911 or seek medical help immediately. Heat stroke is a life-threatening medical emergency. A victim can die within minutes if not properly treated. Efforts to reduce body temperature must begin immediately!