

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