Region 4 U.S. Environmental Protection Agency Laboratory Services & Applied Science Division Athens, Georgia	
Operating Procedure	
Title: Wastewater Sampling	ID: LSASDPROC-306-R6
Issuing Authority: Field Services Branch Supervisor	
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Method Reference: N/A	SOP Author: Chris McHugh

Purpose

The purpose of this procedure is to document both general and specific procedures, methods and considerations to be used and observed when collecting wastewater samples for field screening or laboratory analysis.

Scope and Application

This document describes both general and specific methods to be used by field personnel when collecting and handling wastewater samples in the field. On the occasion that the Laboratory Services and Applied Science Division (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 wastewater sample, the variant procedure will be documented in the field log book, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use by the EPA.

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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Procedural Information

1. Documentation/Verification

1.1. This procedure was prepared by persons deemed technically competent by LSASD management, based on their knowledge, skills and abilities. The procedure 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 LSASD LAN and for maintaining records of review conducted prior to its issuance.

2. General Precautions

2.1. Safety

2.1.1. Proper safety precautions must be observed when collecting wastewater samples. Wastewater can contain microbiological disease agents (pathogens), chemical poisons (toxins), and other biological, chemical, and physical components that may cause human health problems or disturb natural aquatic ecosystems. Waterborne pathogens in the sewer collection system are different, and potentially more antibiotic resistant, than decades ago. Wastewater workers can be exposed to wastewater pathogens and toxins through several pathways:

- Respiratory exposure -face shield and masks protect from droplets and aerosols
- Dermal exposure -gloves and hand hygiene protect from direct contact
- Surface (fomite) exposure - barriers between skin and surfaces protect from wastewater and plant equipment contact

2.1.2. Refer to Centers for Disease Control and Prevention (CDC) Guidance for Controlling Potential Risks to Workers exposed to Class B Biosolids. DHHS (NIOSH) Publication Number 2002-149. Refer to the LSASD Safety, Health and Environmental Management Program 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. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

2.2. Procedural Precautions

2.2.1. The following precautions should be considered when collecting wastewater 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) and/or International Air Transportation Association (IATA) hazardous materials shipping requirements.
- 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 and stored in a secure place.

3. Special Sampling Considerations

3.1. Special Precautions for Wastewater 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 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. 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/control 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.
- Field investigators 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) for collection of samples for trace metals or organic compound analyses.

3.2. Sample Handling and Preservation Requirements

3.2.1. All sample collection and preservation procedures will comply with the requirements outlined in 40 CFR, Part 136.3 (e), Table II, and Table 3-1 of the Region 4 Laboratory Services Branch Laboratory Operations and Quality Assurance Manual (LSB LOQAM), Most Recent Version.

3.2.2. Wastewater samples will typically be collected either by directly filling the sample container or by using an automatic sampler or other device.

3.2.3. 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.

3.2.4. Place the sample into appropriate, labeled containers. Samples collected for VOC analysis must not have any headspace (see Section 8.4, Volatile Organic Compounds). All other sample containers must be filled with an allowance for ullage.

3.2.5. 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 Analytical Support 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 (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.

3.2.6. 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.

3.3. Quality Control

3.3.1. 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 the sampling equipment.

3.4. Records

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

4. General Considerations

4.1. Wastewater Sampling Design

4.1.1. Wastewater sampling studies focus primarily on collecting wastewater samples of the influent or effluent at domestic and non-domestic facilities. Sampling activities are usually conducted for National Pollutant Discharge Elimination System (NPDES) compliance, compliance assistance, civil and criminal investigations, and water quality studies. Collection of wastewater samples is necessary in order to obtain reliable data that can support compliance or enforcement activities.

4.1.2. The main considerations in developing a wastewater sampling strategy are:

- Type of study (Compliance Sampling Inspection (CSI), Diagnostic Evaluation (DE), etc.).
- Regulated or target pollutants in the wastewater stream to be sampled.
- Selection of the projected sampling locations to satisfy the study objectives.
- Quality control criteria of the parameters to be sampled (oil and grease samples need to be collected as grab samples, trip blanks are taken into the field for the collection of samples for volatile organic compound analyses, etc.).

4.1.3. Complexity of the sampling program will vary with a number of factors. Some primary factors are:

- The number of sampling stations to be monitored. This will be dependent on NPDES permit requirements and the type of study; for example, Toxic CSIs and DEs require a greater amount of sampling stations than a routine CSI.
- Special handling requirements of the target pollutants (sampling equipment for trace organic compounds require special cleaning procedures, etc.).
- Laboratory conducting the analyses (use of a contract laboratory may require shipping from the field, etc.).
- Accessibility to sampling stations.
- Process and operation criteria of the source generator (e.g., batch operation versus continuous discharge).
- Coordination of participating organizations in the study (e.g., state assistance with the sample collection).

- The length of time for sampling activities will dictate logistical considerations (e.g., shipment of samples, additional supplies, etc.).

4.2. Sampling Techniques and Equipment

4.2.1. The wastewater sampling techniques and equipment described in Sections 4 through 9 of this document 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 wastewater should be obtained.

4.2.2. The variety of conditions at different sampling locations requires that considerable judgment be exercised regarding the methodologies and procedures for the collection of representative samples of wastewater. Each sampling location warrants attention commensurate with its complexity. There are, however, basic rules and precautions generally applicable to sample collection. Acceptable procedures are generally those outlined in the NPDES Compliance Inspection Manual, most current version. Some important considerations for obtaining a representative wastewater sample include:

- The sample should be collected where the wastewater is well mixed. Therefore, the sample should be collected near the center of the flow channel, at approximately 40 to 60 percent of the water depth, where the turbulence is at a maximum and the possibility of solids settling is minimized. Skimming the water surface or dragging the channel bottom should be avoided. However, allowances should be made for fluctuations in water depth due to flow variations.
- In sampling from wide conduits, cross-sectional sampling should be considered. Rhodamine WT dye may be used as an aid in determining the most representative sampling locations.
- If manual compositing is employed, the individual sample portions must be thoroughly mixed before pouring the individual aliquots into the composite container. For manual composite sampling, the individual sample aliquots should be preserved at the time of sample collection.

4.3. Site Selection for Wastewater Sampling

4.3.1. Where applicable, wastewater samples should be collected at the location specified in the NPDES permit (if the source has a permit). In some instances the sampling location specified in the permit, or the location chosen by the permittee, may not be adequate for the collection of a representative wastewater sample. In such instances, the investigator is not limited by permit specifications and may collect a sample at a more representative location. When a conflict exists between the permittee and the regulatory agency regarding the most representative sampling location, both sites should be sampled, and the reason for the conflict should be noted in the field notes and the inspection or study report. Recommendations and reasons for a change in sampling locations should be given to the appropriate permitting authority.

4.3.1.1. Influent

4.3.2. Influent wastewaters are preferably sampled at locations of highly turbulent flow in order to ensure good mixing; however, in many instances the most desirable location is not accessible. Preferable influent wastewater sampling locations include: 1) the up flow siphon following a comminutor (in absence of grit chamber); 2) the up flow distribution box following pumping from main plant wet well; 3) aerated grit chamber; 4) flume throat; 5) pump wet well when the pump is operating; or 6) downstream of preliminary screening. When possible, influent samples should be collected upstream from side stream returns.

4.3.2.1. Effluent

4.3.3. Effluent samples should be collected at the site specified in the permit, or if no site is specified in the permit, at the most representative site downstream from all entering wastewater streams prior to discharge into the receiving waters. If a conflict exists between the permittee and inspector regarding the source being sampled or the location of the most representative site, follow the procedures previously described in Section 3.3, Site Selection for Wastewater Sampling.

4.3.4. Pond and Lagoon Sampling

4.3.4.1. Generally, composite effluent wastewater samples should be collected from ponds and lagoons. Even if the ponds or lagoons have long retention times, composite sampling is necessary because ponds and lagoons have the tendency to have flow paths that short circuit, which changes the designed detention time.

5. Sample Types

5.1. General

5.1.1. For NPDES sampling, two types of sampling techniques are used: grab and composite. For these procedures, the NPDES permit specifies the appropriate sample type. A complete description of all NPDES sampling procedures and techniques is presented in the NPDES Compliance Inspection Manual, most current version.

5.2. Grab Samples

5.2.1. Grab samples consist of either a single discrete sample or individual samples collected over a period of time not to exceed 15 minutes. The grab sample should be representative of the wastewater conditions at the time of sample collection. The sample volume depends on the type and number of analyses to be performed.

5.3. Composite Samples

5.3.1. Composite samples are collected over time, either by continuous sampling or by mixing discrete samples. A composite sample represents the average wastewater characteristics during the compositing period. Various methods for compositing are available and are based on either time or flow proportioning. The choice of a flow proportional or time composite sampling scheme depends on the permit requirements, variability of the wastewater flow or concentration of pollutants, equipment availability and sampling location. The investigator must know each of these criteria before a sampling program can be initiated. Generally, a time composite is acceptable. However, in enforcement cases where strict adherence to permit requirements are necessary, a flow proportional sample is preferable, if possible.

5.3.2. A time composite sample consists of equal volume discrete sample aliquots collected at constant time intervals into one container. A time composite sample can be collected either manually or with an automatic sampler.

5.3.3. A flow proportional composite sample can be collected using one of two methods. One method consists of collecting a constant sample volume at varying time intervals proportional to the wastewater flow. For the other method, the sample is collected by varying the volume of each individual aliquot proportional to the flow, while maintaining a constant time interval between the aliquots.

5.3.4. Flow proportional samples can be collected directly with an automatic sampler that is connected to a compatible flow measuring device. An automatic sampler can also be used to collect discrete samples. At the end of the compositing period, the discrete samples are composited by volume versus flow chart readings. Field personnel can use the facility's primary flow device and flow measurement system when their accuracy can be verified. Prior to collecting flow proportional samples, the facility's flow measuring system should be examined for proper installation and accuracy according to LSASD Operating Procedure for Wastewater Flow Measurement (LSASDPROC-109). If the facility's primary flow measuring device does not meet standard conditions specified in LSASDPROC-109 (Most Recent Version), or is in an unsafe or inaccessible location, then the investigator may choose to collect time composite samples or install a portable primary flow device. If the flow measurement system is acceptable, samples should be collected using the appropriate flow proportioning methods.

6. Automatic Samplers

6.1. General

6.1.1. Automatic samplers may be used to collect composite or grab samples when several aliquots are to be collected at frequent intervals or when a continuous sample is required. For composite sampling applications, the automatic samplers may be used to collect time composite or flow proportional samples. In the flow proportional mode, the samplers are activated and paced by a compatible flow meter. Flow proportional samples can also be collected using an automatic sampler equipped with multiple containers and manually compositing the individual sample portions proportional to the flow.

6.1.2. Automatic samplers must meet the following requirements:

- Sampling equipment must be properly cleaned to avoid cross-contamination which could result from prior use per the LSASD Operating Procedure for Field Equipment Cleaning and Decontamination (LSASDPROC-205).
- No plastic or metal parts of the sampler shall come in contact with the water or wastewater stream when parameters to be analyzed could be impacted by these materials.
- The automatic sampler must be capable of providing adequate refrigeration during the sampling period. This can be accomplished in the field by using ice.
- The automatic sampler must be able to collect a large enough sample for all parameter analyses.
- The individual sample aliquot must be at least 100 mL if the sampler uses a peristaltic pump.
- The automatic sampler should be capable of providing a lift of at least 20 feet and the sample volume should be adjustable since the volume is a function of the pumping head.
- The pumping velocity must be at least two (2) ft. /sec to transport solids and not allow solids to settle.
- The intake line leading to the pump must be purged before each sample is collected.
- The minimum inside diameter of the intake line should be 1/4 inch.
- An adequate power source should be available to operate the sampler for the time required to complete the project. Facility electrical outlets may be used if available.
- Facility automatic samplers should only be used if 1) field conditions do not allow for the installation of EPA sampling equipment, and 2) the facility sampling equipment meets all of the requirements detailed above.

6.1.3. Specific operating instructions, capabilities, capacities, and other pertinent information for automatic samplers are included in the respective operating manuals.

6.2. Conventional Sampling (Inorganic Parameters)

6.2.1. Conventional sampling includes all inorganic parameters (e.g., BOD5, TSS, COD, nutrients) that can be collected using an automatic sampler.

6.2.2. New tubing (Silastic ®, or equal, in the pump and either Teflon ® or Tygon ®, or equal, in the sample train) will be used for each sampler installation.

6.2.3. Installation procedures for installing tubing on a sampler include cutting the proper length of tubing, positioning it in the wastewater stream, and sampler programming. Protective gloves should be worn to reduce exposure and to maintain the integrity of the sample.

6.2.4. For a time composite sample, the sampler should be programmed to collect sufficiently sized aliquots (at least 100-milliliter if using a peristaltic pump) at a frequency that provides a representative sample and enough sample volume to conduct all required analyses.

6.2.5. For a flow proportional sample, the sampler should be programmed to collect a minimum of 100 -milliliters for each sample aliquot with the interval predetermined based on the flow of the monitored stream.

6.2.6. At the end of the compositing period, the sample collected should be properly mixed and transferred into the respective containers, followed by immediate preservation, if required. For routine inspections, the permittee should be offered a split sample.

6.3. Metals

6.3.1. When an automatic sampler is used for collecting samples for metals analyses, the entire sample collection system is rinsed with organic-free water and an equipment rinse blank is collected. The equipment rinse blank is taken to ensure that metals contamination is not occurring from the sampling equipment, and to check the effectiveness of the decontamination procedures. To collect an equipment rinse blank approximately one-half gallon of rinse water should be pumped through the sample tubing into the composite container and discarded. After the purge, another one-half gallon of rinse water is pumped through the sample tubing, into the composite container, and collected as an equipment rinse blank. Once the equipment rinse blank sample is collected, it must be properly preserved with Nitric acid. The automatic sampler may then be positioned in the appropriate location and the sampler program initiated.

6.3.2. If the automatic sampler tubing is attached to a metal conduit pipe, the intake tubing should be carefully installed upstream and away from the conduit to prevent metals contamination. This can be accomplished by clamping the tubing upstream of the conduit using laboratory clamps and wrapping the submerged portion of conduit pipe with a protective barrier (e.g., duct tape).

6.4. Extractable Organic Compounds, Pesticides and PCBs

6.4.1. When an automatic sampler is used for collecting samples for the analyses of extractable organic compounds, pesticides and/or PCBs, the installation procedures include cutting the proper length of new Teflon tubing, rinsing of the entire sampler collection system with organic-free water and collection of appropriate equipment blanks for organic compounds analysis. For the organic-free water rinse, approximately one-half gallons is initially pumped into the composite sample container and discarded. An additional one and one-half gallons (approximate) are then pumped into the composite sample container for distribution into the appropriate blank container. Finally, the collection tubing should be positioned in the wastewater stream and the sampler programmed and initiated.

6.5. Automatic Sampler Security

6.5.1. Field investigators should take whatever steps are necessary to prevent tampering with EPA equipment. A lock or custody seal may be placed on the sampler to detect tampering. However, this does not prevent tampering with the sample collection tubing. If necessary, seals may be placed on the sampling pole and tubing line to further reduce tampering possibilities.

6.6. Automatic Sampler Maintenance, Calibration and Quality Control

6.6.1. To ensure proper operation of automatic samplers, and thus the collection of representative samples, the following maintenance and calibration procedures should be used and any deviations should be documented in the field logbook.

6.6.2. Prior to being used, the sampler operation should be checked by the field investigator or Field Equipment Center personnel to ensure proper operation. This includes operation (forward, reverse, and automatic) of at least one purge-pump-purge cycle; checking desiccant and replacing if necessary; checking the 12-volt batteries to be used with the sampler; and repairing any item if necessary.

6.6.3. During each field trip, prior to initiating the automatic sampler, the rinse and purge-pump-purge cycle shall be checked at least once. The pumping volume should be checked at least twice using a graduated cylinder or other calibrated container prior to initiating the sampler. For flow proportional sampling, the flow meter that activates the sampler should be checked to ensure that it operates properly.

6.6.4. Upon returning from a field trip, the structural integrity of the sampler should be examined and repaired, if necessary. The desiccant will be checked and replaced if appropriate. The operation (forward, reverse, automatic, etc.) will be checked and required repairs will be made and documented. The sampler will then be cleaned as outlined in LSASD Operating Procedures for Field Equipment Cleaning and Decontamination (LSASDPROC-205).

6.6.5. The automatic sampler should be checked against the manufacturer's specifications and documented whenever one or more of the sampler functions appear to be operating improperly.

7. Manual Sampling

7.1. Manual sampling is normally used for collecting grab samples and/or for immediate in-situ field analyses. However, it can also be used in lieu of automatic equipment over extended periods of time for composite sampling, especially when it is necessary to evaluate unusual waste stream conditions.

7.2. The best method to manually collect a sample is to use the actual sample container which will be used to transport the sample to the laboratory. This eliminates the possibility of contaminating the sample with intermediate collection containers. If the water or wastewater stream cannot be physically reached by the sampling personnel or it is not safe to reach for the sample, an intermediate collection container may be used, from which the sample can be redistributed to other containers. If this is done, however, the container used to collect the sample must be properly cleaned according to the LSASD Operating Procedure for Field Equipment Cleaning and Decontamination (LSASDPROC-205) and must be made of a material that meets the requirements of the parameter(s) being investigated. Samples for oil and grease, bacteria, and most volatile compounds (both organic and inorganic; see Section 7.4 for specific requirements) must always be collected directly into the sample container. Sampling guidance for PFAS included in section 7.5

7.3. In some cases, it may be best to use a pump, either power or hand operated, to withdraw a sample from the water or wastewater stream. If a pump is used, it is imperative that all components of the pump that come in contact with the sample are properly cleaned according to the LSASD Operating Procedure for Field Equipment Cleaning and Decontamination (LSASDPROC-205) to ensure the integrity of the sample.

7.4. In general, samples are manually collected by first selecting a location in the Wastestream that is well mixed then dipping the container in the water or wastewater stream, so the mouth of the container faces upstream. The container should not be overfilled if preservatives are present in the container.

8. Special Sample Collection Procedures

8.1. Organic Compounds and Metals

8.1.1. Trace organic compounds and metals detection limits are usually in the parts per billion or parts per trillion ranges, so extreme care must be exercised to ensure sample integrity.

8.1.2. All containers, composite bottles, tubing, etc, used for sample collection for trace organic compounds and metals analyses should be prepared as described in the LSASD Standard Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (LSASDPROC-206).

8.1.3. When possible, the sample should be collected directly into the appropriate sample container. If the material to be sampled cannot be physically reached, an intermediate collection device may be used. This should be a Teflon®, glass or stainless steel (for non-metals only) vessel on a pole or rope, or Teflon® tubing via a peristaltic type pump and a Teflon® vacuum container attachment, which converts a sample container into a vacuum container. The device used should be cleaned as described in the LSASD Operating Procedure for Field Equipment Cleaning and Decontamination (LSASDPROC-205).

8.1.4. Sample collection for trace-level mercury analysis will be conducted in accordance with the LSASD Operating Procedure for Surface Water Sampling (LSASDPROC-201). This procedure is based on EPA Method 1669.

8.2. Bacteriological

8.2.1. Samples for bacteriological analyses must always be collected directly into the prepared glass or plastic sample container. The sample container should be kept unopened until it is to be filled. When the cap is removed, care should be taken not to contaminate the cap or the inside of the bottle. The bottle should be held near the base and filled to within about one inch of the top without rinsing and recapped immediately. During sample collection, the sample container should be plunged with the neck partially below the surface and slightly upward. The mouth should be directed against the current. Preservation procedures and holding times are found in the LSB LOQAM.

8.2.2. When the sample container must be lowered into the waste stream, either because of safety or impracticality (manhole, slippery effluent area, etc.), care must be taken to avoid contamination.

8.3. Immiscible Liquids/Oil and Grease

8.3.1. Oil and grease may be present in wastewater as a surface film, an emulsion, a solution or as a combination of these forms. Since it is very difficult to collect a representative sample for oil and grease analysis, the inspector must carefully evaluate the location of the sampling location. The most desirable sampling location is the area of greatest mixing. Quiescent areas should be avoided. The sample container should be plunged into the wastewater using a swooping motion with the mouth facing upstream. Care should be taken to ensure that the bottle does not over fill during sample collection.

8.3.2. Because losses of oil and grease will occur on sampling equipment, an automatic sampler should not be used to collect samples for oil and grease analysis. Individual portions collected at prescribed time intervals must be analyzed separately to obtain the average concentrations over an extended period.

8.4. Volatile Organic Compounds

8.4.1. Samples to be analyzed for volatile organic compounds (VOCs) should be collected in 40-mL septum vials with screw caps with a Teflon® -lined silicone disk (septum) in the cap to prevent contamination of the sample by the cap. Samples for VOC analysis must be collected using either stainless steel or Teflon® equipment.

8.4.2. When sampling for VOCs, triplicate samples should always be collected from each location. The investigator should determine if the water to be sampled contains chlorine. If the water contains no chlorine, three pre-preserved 40-ml vials should be filled with the sample. The samples may be held for up to 14 days before analysis. When preservation is not feasible, samples can be held up to seven (7) days before analysis. In the great 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 wastewater 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.

8.4.3. If the water contains chlorine, collect the sample in an 8-ounce sampling container with two (2) drops of a 25% ascorbic acid solution (the jar with acid should be obtained from the LSASD laboratory prior to sample collection). Cap and mix thoroughly but gently by swirling to eliminate residual chlorine. Transfer the sample to three pre-preserved 40-ml vials. The ascorbic acid and preservative must be added in this order and in two separate steps.

8.4.4. The 40-mL vials should be completely filled to prevent volatilization, and extreme caution should be exercised when filling each vial to prevent 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 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.

8.5. Per- and Polyfluoroalkyl Substances (PFAS)

8.5.1. 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, PFAS may be concentrated in the WWTPs biosolids.

8.5.2. For wastewater or process wastewater samples undergoing PFAS analyses, guidance documents recommend sampling equipment be made of high-density polyethylene (HDPE), polypropylene, stainless steel and/or silicone. 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. If the wastewater or process 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.

8.5.3. 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 or other potential contaminants, they need to be changed for new gloves. Nitrile gloves are recommended for PFAS sampling investigations. Also, sample containers along with sampling gloves should be kept covered in original packaging or in Whirl Paks® until ready for use.

8.5.4. For wastewater or process wastewater samples undergoing PFAS analyses, it extremely important that quality control samples (equipment rinse blanks) be collected as part of the investigation to demonstrate the PFAS contribution, if any, 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 wastewater sampling and sample handling activities of the investigation.

9. Special Process Control Samples and Tests

9.1. During diagnostic evaluations, process control tests may be conducted to evaluate and troubleshoot the performance of the biological treatment processes of a municipal or industrial wastewater treatment facility. The EPA Activated Sludge Process Control Manual is the standard reference used by EPA inspectors for activated sludge process control testing. The manual includes a complete description of the step-by-step procedures for each test and the interpretation of the results. The six basic activated sludge process control tests are:

- Sludge settleability (settlometer)
- Centrifuge spins
- Aeration basin Dissolved Oxygen (DO) profiles
- Oxygen uptake rate (OUR) measurements
- Mixed liquor microscopic examinations
- Sludge blanket depth (SBD) measurements

9.2. Additional references are available that provide a more comprehensive evaluation of the methods used to conduct a diagnostic evaluation.

10.Supplementary Data Collection

10.1. While conducting wastewater sampling, the following information will be obtained, if applicable:

- Field measurements -- pH, dissolved oxygen, total residual chlorine, conductivity and temperature (LSASDPROC-100,101,102,106,112 most recent version).
- Flows associated with the samples collected -- continuous flows with composite samples and instantaneous flows with grab samples (LSASDPROC-019 most recent version).
- Photographs of pertinent wastewater associated equipment, such as flow measuring devices, treatment units, etc. (keep photolog as specified in the LSASD Operating Procedure for Logbooks (LSASDPROC-010 most recent version)).
- Global Positioning System (GPS) data point of the location sampled (LSASDPROC-110 most recent version)
- Diagrams and/or written descriptions of the wastewater treatment systems (if available).
- Process control information on the wastewater treatment process (if applicable).
- Completion of applicable forms required during specific investigations.

10.2. All observations, measurements, diagrams, etc., will be entered in bound field logbooks or as specified in the LSASD Operating Procedure for Logbooks (LSASDPROC-010 most recent version).

References

California State University – Sacramento. Operation of Wastewater Treatment Plants - Volumes I, II, III. Sacramento, California.

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

Metcalf and Eddy, Inc. 2003. Wastewater Engineering: Treatment, Disposal, and Reuse. 4th Edition, McGraw-Hill Book Co., New York, NY

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

LSASD Operating Procedure for Equipment Inventory and Management (LSASDPROC-104, 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 Field pH Measurement, LSASDPROC-100, Most Recent Version

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

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

LSASD Operating Procedure for Field Temperature Measurement, LSASDPROC-102, Most Recent Version

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

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

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

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

LSASD Operating Procedure for Wastewater Flow Measurement, LSASDPROC-109, Most Recent Version

Title 40 Code of Federal Regulations (CFR), Part 136.3, Table II, Most Recent Version

US EPA. 1977. Process Control Manual: Aerobic Biological Treatment Facilities MD-14. EPA 430/09-77-006, Office of Water, Washington, D.C.

US EPA. 2000. Activated Sludge Process Control Testing. ESD, Water Compliance Unit, Athens, GA

US EPA. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. 2004. National Pollutant Discharge Elimination System (NPDES) Compliance Inspection Manual

US EPA. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4 LSASD, Athens, GA, Most Recent Version

US EPA. April 13, 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)

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

Water Environment Federation and ASCE. 1998. Design of Municipal Wastewater Treatment Plants. Manual of Practice No. 8, Fourth Edition.

Water Environment Federation. 1996. Operation of Municipal Wastewater Treatment Plants. Manual of Practice No.11, Fifth Edition

Interstate Technology & Regulatory Council(ITRC),2020.PFAS Technical and Regulatory Guidance Document and Fact Sheets PFAS-1. Washington, DC.: Interstate Technology & Regulatory Council, PFAS Team. https://pfas-1.itrcweb.org/.

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	Review Date
Replaced Chief with Supervisor _____ LSASDPROC-306-R5, Wastewater Sampling, replaces LSASDPROC-306-R3 **General:** Corrected any typographical, grammatical and/or editorial errors. Replace the Division name from SESD to LSASD. Operating procedure documents where not changed except for this document. **Title Page:** Field Services Branch changed to Applied Sciences Branch, and Supervisor changed from Danny France to John Deatrick. Changed Field Quality Manager from to Jeff Hendel Section 7.5: Added language to increase awareness of sampling considerations for PFAS Section 1.5.1: Added language to increase awareness of pathogen and exposure pathways. **Section 5.1.2:** Added language to discuss the collection of an equipment rinse blank from an automatic sampler. **Section 9.0:** Added language concerning the collection of a GPS data point at each sample location.	April 22, 2023 _____ February 02, 2021
LSASDPROC-306-R3, Wastewater Sampling, replaces LSASDPROC-306-R2	February 28, 2013
LSASDPROC-306-R2, Wastewater Sampling, replaces LSASDPROC-306-R1	February 19, 2010
LSASDPROC-306-R1, Wastewater Sampling, replaces LSASDPROC-306-R0	November 1, 2007
LSASDPROC-306-R0, Wastewater Sampling, Original Issue	February 05, 2007