

## **Wastewater Sampling**

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## **Preface**

The Watershed Protection Branch (WPB) of the Georgia Environmental Protection Division (GAEPD) has created a series of standard operating procedures (SOP) establishing uniform methods for the field collection of data, document control, quality assurance, laboratory safety, as well as other activities. These protocols were developed to document, and ensure, the validity of measurements, analyses, and the representativeness of samples collected. This is necessary in the event of a dispute with other parties regarding data collection techniques and the resulting quality of field information. Enforcement activities by the Branch require full documentation on particulars of data collection and the equipment used to collect it. All Branch associates who collect samples or field data must be familiar with the measures outlined in the appropriate SOP's.

Requirements pertaining to specifics of sample collection for certain parameters are specified in federal regulations under the authority of the Clean Water Act (CWA) and the National Pollutant Discharge Elimination System (NPDES) permitting program. The most widely applicable guidance at this level is *Title 40 of the Code of Federal Regulations (40 CFR)*. The procedures and techniques given in *40 CFR* are updated periodically by the United States Environmental Protection Agency and field workers are advised to consult the latest revision for proper procedures and new developments. In addition, the SOPs utilized by the Branch should be reviewed annually to certify their concurrence with federal statutes. Other references used in developing each SOP are cited at the conclusion of the individual documents.

The collection protocols in *40 CFR* are in many instances based on the concern for quality assurance. As such, each SOP will contain a section devoted to maintaining and improving the quality of data collected. 'Quality Assurance and Quality Control' sections contained within individual SOPs are not meant to replace the overall Quality Assurance Project Plan documents prepared for the Branch, but rather, are provided as supplemental data for each specific, standardized activity.

This document is dynamic and will be continually revised as new developments warrant. As the Branch assumes more responsibilities for studying and sampling in new investigational areas, it is anticipated that additional SOPs will be required.

## **A. Introduction**

The objective of waste stream sampling is to obtain a representative quantity to analyze for parameters of concern. In some cases, it is desired to accurately represent contents of the waste stream at a given moment; this is accomplished by a grab sample. It may also be advantageous for certain purposes to represent the waste stream characteristics over a period of time; composite sampling does this. Considerations unique to each of these approaches will be discussed below.

Samples collected at wastewater treatment facilities are “split” with the permittee during certain types of water quality inspections. Sample splitting involves providing the facility representative with a set of samples that duplicate those taken by Branch associates to the water quality laboratory for analysis. Comparison of analytical results on such sample splits is an important component of self-monitoring program validation.

The SOP set forth herein was originally developed for the purpose of compliance sampling at various types of wastewater-generating facilities regulated by the Branch. With the passage of time, the Branch has assumed additional responsibilities for environmental protection that go beyond traditional compliance sampling of influent and effluent. Storm water, Combined Sewer Overflows (CSOs), land-use associated runoff and other discharges to the State’s surface waters may be the object of sampling excursions by Branch associates. The guidance and requirements set forth below apply and may be adapted to these additional sampling tasks.

## **B. Purpose and Applicability**

The purpose of this SOP is to establish uniform procedures for sampling wastewater within the State of Georgia. The procedures outlined in this SOP are applicable to all Branch associates who collect, or assist in the collection of, surface water samples in support of water quality and compliance monitoring.

## **C. Health and Safety Warnings**

The health and safety of Branch associates must not be at risk during wastewater sampling. The threats from wastewaters encountered by Branch associates are manifold. Municipal/sanitary wastes pose obvious pathogenic hazards. Process wastewaters from industry may contain harmful concentrations of metals, solvents, or chemicals such as sulfides, cyanide, or chlorine. Some waste streams may be excessively corrosive or caustic. For these reasons, any associates engaged in wastewater sampling should wear protective gloves (and in some cases eye shields). Gloves should be consistent with the level of threat, as well as the nature of the parameters to be analyzed. For example, during metals sampling, latex gloves dusted with talc should not be worn. Industrial settings may require hearing protection and/or steel-toed boots. At a minimum, Branch associates should be familiar with Part III of the DNR Safety Manual prior to going into the field.

Protective equipment serves another purpose; that of ensuring the integrity of the sample collected. For the collection of samples to be analyzed for volatile organics, for example, a clean pair of Latex gloves must be worn. Various products such as hand creams, cologne, or hair preparations may contain chemicals that could be detected in a worst-case contamination scenario. Branch sampling associates should use good judgment when approaching each unique field assignment with these considerations in mind.

#### **D. Personnel Qualifications**

All Branch associates who collect wastewater samples or field data must be familiar with the procedures outlined in this document. In all aspects of water quality planning and field assessment activities, safety is to be addressed and treated as a critical element. The Georgia DNR Safety Manual is to be consulted and its policies, protocols, and procedures are to be incorporated and implemented in WPB field activities.

#### **E. Standard Operating Procedures for All Sampling Activities**

##### **1. Sampling Methods, Sampling Equipment & Sample Containers**

Analytical parameters determine wastewater sampling protocols. That is, sampling methods, equipment and sample containers must be consistent with the analytes of concern. Parameter-specific procedures are provided in the following sections. All wastewater sampling equipment must be clean and free of contamination that may have occurred during previous sampling, storage, or transportation to the site. “Clean” in the sense of suitability for conventional parameter sampling (oxygen demand, solids, nutrients, et cetera) means:

- New equipment from a closed container
- Equipment that has been washed with laboratory grade phosphorous-free detergent and rinsed with potable water.

Sample bottles must be appropriate for analytical parameters. The definitive reference and guidance for determination of sample containers, sample types (grab versus composite), required preservation techniques, maximum holding times and parameter-specific sampling considerations is *Title 40 of the Code of Federal Regulations, Part 136*. Samples collected for NPDES reporting by the regulated community must adhere to the requirements of *40 CFR Part 136*; Branch associates must also follow these procedures for sampling results to be valid for enforcement purposes. A complete reiteration of the Federal guidelines is beyond the scope of this document. The Code is continually updated and revised by publications of notices in the Federal Register. In the event of a conflict regarding a method or technique, the most recent guidance appending the Code will rule.

Sample bottles used by Branch associates are provided by GAEPD’s Laboratory. The correct types and amounts of preservatives are added to the containers by laboratory associates. All bottles and preservatives are consistent with the requirements of *40 CFR*

*Part 136.* Samples for certain parameters are required to be collected as grab samples only. Other samples must be collected directly into the bottle containing the preservative (examples include oil & grease, volatile organics, and bacteriological samples).

## **2. Sample Identification & Sampling Records**

Sample bottles must be labeled in the field either by writing with an indelible, waterproof marker directly on the bottle or by securing a special waterproof label to the bottle that has been inscribed with the necessary information. Bottle labeling information shall include, at a minimum, the following items:

- Name of facility (or locational identifier) where sample was collected
- Nature of the sampled substance (influent, effluent, activated sludge, etc.).
- The date of sample collection
- The time or time interval represented by the sample
- The name of the sampling team leader
- Any special sample characteristics that could affect analysis for the parameters requested (facility dechlorinates with sulfur-dioxide, sample suspected of high concentrations of certain pollutants, etc.)

Records of samples collected are made at the time of collection in bound field books. This is to ensure sample analysis results are “traceable” from the moment of sample collection to laboratory report preparation. The records required include all sample label information, as well as the identity of the sample collector and other team members present. Additional observations of sampling concerns, unusual waste stream characteristics and environmental conditions, must also be noted in the field books. If an automatic sampler is used, its serial number or DNR Property Control number must be recorded. Primary responsibility for field book entry falls on the sampling team leader but may be delegated as the leader deems appropriate to expedite operations. Documentation of photographs taken, diagrams of sampling locations or treatment vessel layout and field data information must also be recorded in the field book. Calibration data for field instruments may be entered in the field book or separate QA/QC forms designed and used by individual work units.

Various specialized evaluation forms have been designed and implemented by Branch work units. Forms corresponding to the different types of inspections, investigations, audits, overviews, and reconnaissance should be filled out on site with the necessary field observations as appropriate.

## **3. Parameter-Specific Sampling Procedures**

### **A. Metals Sampling**

Sampling for metals (at other than “trace metal” levels) necessitates a further stage of equipment cleaning. Any component of the sampling equipment train that contacts the

sample must meet these guidelines. Sampling equipment must be plastic (polyethylene, etc.), Teflon, Tygon, Silastic, PVC or similar substances. Metal containers or intermediate collection devices are not acceptable for metals sampling (brass, cast iron, aluminum, copper, or galvanized containers, etc.). Although acceptable for conventional parameter sampling, the potential for leaching of metals into the sample makes these items unsuitable for metals sampling.

Metals sampling equipment must be either new or acid rinsed. The procedure for acid rinsing is as follows:

- Prepare 10% strength dilute acid-rinsing solution with laboratory-grade nitric acid and de-ionized water
- Rinse the equipment with dilute acid solution three (3) times
- Rinse the equipment with de-ionized water three (3) times
- Allow equipment to air dry then cover with laboratory-grade plastic wrap

Metals samples may be collected as grab or composite samples. When using an automatic sampler to collect a composite sample for metals analyses each individual aliquot bottle in a sequential sampler must be acid-rinsed in addition to the sample strainer, sample intake tubing and sample pump tubing. Alternatively, disposable Low-Density Polyethylene bottle liners (ISCO “ProPak” or equivalent) may be used, obviating the need for acid-rinsing each aliquot bottle.

Whether collected as a grab or composite sample, care must be taken not to overfill the laboratory-supplied sample bottle or the acid preservative will be lost. Due to QA/QC requirements by the GAEPD Laboratory, the metals sample bottles must be almost completely filled.

Trace-metal detection sampling involves yet another level of equipment preparation, QA/QC documentation and anti-contamination measures. Although the specialized methodologies are beyond the scope of this document, EPA has developed guidance for trace-metal sampling that is used on those occasions when Branch associates must sample for trace level metals (United States Environmental Protection Agency, Office of Water Engineering and Analysis Division, Method 1669 Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels, July 1996).

## **B. Organics Sampling**

Sampling for those analytes generally termed “organics” (volatiles, purgeables, PCBs, pesticides, base neutrals, etc.) also requires specialized containers and sampling equipment. Acceptable materials include glass, stainless steel, and Teflon. Cleaning between sampling events must include a solvent rinse (laboratory-grade hexane or methanol) in addition to conventional cleaning, followed by a de-ionized water rinse. The equipment should then be covered with aluminum foil or clean plastic sheeting.

Sampling for Volatile Organic Compounds (VOCs) requires special attention. One set of VOCs consists of three (3) 40-milliliter septum vials with screw caps and a Teflon coated cap insert. An amount of concentrated hydrochloric acid is placed in each vial at the laboratory as a preservative.

The procedure for filling organics bottles (including VOC vials) is as follows:

- The associate filling organics bottles must don a clean pair of Latex gloves.
- VOC vials must be filled first. If possible, collect the sample directly into each vial without running the vial over and losing the acid preservative. Allow sample to fill the vial by running down the side with a minimum of turbulence. Continue to fill each vial until there is a “positive meniscus” of wastewater sample held above the level of the vial rim by surface tension. Gently place the cap with Teflon insert on the vial top and engage the threads by clockwise rotation (as viewed from above). As the cap is tightened, water displaced from the positive meniscus should be observed running down the outside of the vial; this is an indication of a completely filled vial. Verify the vial is completely filled by inverting and looking for air bubbles. If an air bubble is observed, the final stage of vial filling must be repeated (With some industrial wastewater gas will come out of solution after the vial is capped. If this is the case the vial does not have to be refilled and recapped).
- If it is not possible to collect the VOC sample directly into the vials a one-liter amber glass bottle (as provided by the laboratory for purgeables/extractables, PCBs, and pesticides) may be used as an intermediate sampling vessel. Collect the bottle half-full and decant into VOC vials as specified above.
- If the wastewater to be sampled for VOCs contains chlorine, the sample aliquot must be dechlorinated with sodium thiosulfate before placement in the septum vials. The vessel used for dichlorination must meet the requirements for organics sampling equipment. (A one-liter amber glass bottle as discussed in the previous paragraph may be used). Five-hundred mL (500 mL) of sample may be dechlorinated with eight (8) drops of a 10% sodium-thiosulfate solution. The sample being treated should be gently swirled to avoid volatilization as the dechlorinating agent is mixed. The sample may then be transferred to the 40-milliliter vials as discussed above.
- The one-liter amber glass bottles should be filled with sample directly from the wastewater stream. If this is not possible any intermediate sample container must meet the organics sampling equipment requirements. Although the one-liter bottles do not have to be collected and capped without air bubbles, the GAEPD Laboratory requires that they be full well up into the narrow neck of the bottle.

### **C. Bacteriological Sampling**

Bacteriological samples must be collected directly into sterile containers provided by the GAEPD Laboratory. The bottles have an appropriate amount of sodium-thiosulfate to



neutralize chlorine. (Presence of sodium-thiosulfate does not negatively affect the sample if chlorine is not present). The procedure for collecting bacteria samples is as follows:

- Remove the Kraft paper-covered cap only when ready to dip the sample from the waste stream. Do not touch the inside of the cap or allow other objects to contact it. Do not lay the cap on the ground or other surface while collecting the sample.
- Any intermediate container used for bacteria sampling must be sterile.
- During sample collection the bottle should be inserted into the waste stream with the neck partially below the surface and tilted slightly upward with the mouth directed against the current. The bottle should be filled to within approximately one inch of the top to provide adequate mixing space. If chlorine is present, do not allow the bottle to overfill or the sodium-thiosulfate dechlorinating agent will be lost.
- Replace the bottle cap immediately after filling the bottle with sample to the proper level.
- Bacteriological samples must be stored at 10°C or below.

#### **D. Oil & Grease / Immiscible Liquids Sampling**

Oil and grease may be present as a surface film, a solution, an emulsion, or a combination of these forms. The area of greatest mixing in the wastewater stream is the preferred sampling location. Oil and grease may only be collected as a grab sample and must be collected directly into the bottle provided by the GAEPD Laboratory. With the mouth of the bottle facing upstream, it should be filled as completely as possible without losing the preservative, which is an amount of sulfuric acid sufficient to depress the pH to less than 2 standard units.

#### **E. Cyanide Sampling**

Cyanide (Total or Amenable) may only be collected as a grab sample; however, conventionally cleaned intermediate containers are permissible. The cyanide sample bottles have a number of sodium-hydroxide pellets added sufficient to raise the sample pH above 12 standard units. Since the sample is preserved, the sample bottle should not be overfilled, but it must be full to near the bottle mouth.

#### **F. PFAS Sampling**

In depth PFAS sampling guidance can be obtained from the GAEPD Laboratory. Grab sampling is the preferred method for PFAS wastewater collection. Due to an increased risk for cross-contamination, automatic composite sampling should only be used if a grab sample is not viable. If a direct sample into the sample bottle is not possible, make sure automatic sampler tubing is vinyl or another PFAS free tubing. Teflon tubing should not be used. Care must be taken to make sure sampling equipment is PFAS-free. Approved containers should be PFAS-free HDPE and placed in PFAS-free LDPE bags (e.g. Ziploc storage bags). Clean, new nitrile, -gloves must be worn during sampling. It is important to sample equipment wash blanks for any equipment that could contaminate the wastewater sample. Every collected sample should be in duplicate and trip blanks, as well as field

blanks, and matrix spike/ matrix spike duplicate (MS/MSD) samples are also required. Samples may have added preservative. Samples are shipped on ice at  $\leq 6$  °C. Once the samples are at the lab, the PFAS is extracted with methanol for analysis.

#### **4. Process Control Sampling**

Branch associates perform Technical Evaluations at treatment works of particular concern. Process control tests are run on activated sludge system component streams such as aeration basin mixed liquor, return activated sludge, settled sludge, and primary and secondary effluents. Due to the very high solids concentrations of some of these fluids, obtaining a representative sample is especially critical. Recommended sampling sites include return sumps or pits, junction boxes, aeration basin overflow weirs, and near mechanical aerators or compressed air diffusers because flow at these locations is typically very turbulent, providing a well-mixed sample. In the case of very large aeration basins that are not uniformly mixed or aerated, an areal composite may be necessary. Several samples from widely dispersed locations in the basin are mixed to form a composite basin sample.

Most of these samples will be manual grab samples (although in some cases an automatically collected time-composite may be required). For certain substances of extremely high solids content (digester contents, dewatered solids, and return sludge) the GAEPD Laboratory may provide special containers.

Although procedures on process control testing and troubleshooting are beyond the scope of this document, the reader is referred to the EPA's Activated Sludge Process Control Testing handbook and their Process Control Manual: Aerobic Biological Treatment Facilities. The California State University at Sacramento's Operation of Wastewater Treatment Plants (Volumes I, II, and III) also has valuable practical information.

#### **5. Sampling Site Selection**

Additional consideration must be given to selection of the sampling location. Branch associates encounter a wide variety of conditions and wastewater conduit configurations during field work. It would be impossible to design guidance that would address every possible sampling situation. On site judgment must be exercised on each occasion to assure that the samples collected satisfy the purpose and intent of the sampling effort. General guidelines may be detailed, as follows:

- Samples should be taken where the wastestream is well mixed, near the center of the channel.
- Care should be taken to select a location that will not unduly bias any parameter positively or negatively.
- Note that the sampling location defined in the facility's permit (if a permit has been issued) is not necessarily the optimum location for sampling.

- In the case of influent sampling at a wastewater treatment works, it is desirable to sample upstream of any “return” flows such as filtrates, centrates, washdown water, return activated sludge, supernatants, etc., to the extent possible.
- When sampling wastewater treatment effluents, the final effluent is often specified as the point of application of discharge limitations; however, there are many exceptions to this relevant to chemical addition (chlorine and dechlorinating agents, defoamer, supplemental oxygen, etc.). In the event of conflicting protocols, more than one sample may be collected.

Additional guidance may be found in EPA’s NPDES Compliance Sampling Inspection Manual, which has been adopted by the Branch as its main reference for wastewater sampling standard procedures.

## **6. Grab Sampling & Manual Sampling**

Grab samples consist of a single sample or individual samples collected over a period of fifteen minutes or less. The quantity collected is determined by the type and number of analytical parameters. Grab sampling is useful when the waste stream is not continuous, such as the cases of batch discharges and intermittent flows. Grab sampling may also be appropriate when the characteristics of the waste stream are known to be constant through time. Samples for certain parameters are required to be collected as grab samples. By collecting a series of grab samples on a continuous discharge, valuable data on maximum and minimum concentrations may be obtained that would be missed by the compositing process.

As discussed above, sampling equipment and preparation must be consistent with any special requirements of the parameters. In the case of sampling for a broad suite of analytes, multiple protocols may need to be followed, necessitating a variety of sampling devices of different materials.

Grab samples are most often collected manually. In-situ field determinations and on-site analyses are also performed on manually collected grab samples. When grab sampling for laboratory parameters, it is desirable to collect the sample directly in the bottle(s) provided by the laboratory. In the case of bottles containing preservatives, care must be taken not to overfill the bottle and lose the preservative. When an intermediate sample container must be used for reasons of safety or accessibility, it must meet the material and cleaning requirements for the parameter(s) being sampled. Samples collected for analysis of oil & grease, bacteria, and volatile organics must always be collected directly into the laboratory sample bottle.

Sometimes it is advantageous to collect grab samples by use of a pump. If a pump is used, the same considerations apply as for an intermediate sample container. All internal pump components that contact the sample must meet the appropriate guidelines for the parameters being analyzed.

Certain grab samples must be collected from a tap, valve, or another outlet as opposed to an open channel. When this is necessary, allow sufficient fluid to run out of the source to purge the piping of stagnant material prior to sampling.

Whatever method is chosen, the preferred location for grab sampling is one that is well mixed so a representative sample is obtained. The accepted procedure is to dip the sampling vessel in the wastestream so the mouth of the container faces upstream.

## **7. Composite Sampling**

A composite sample consists of a number of individual sample aliquots collected over a period of time. The purpose of composite sampling is to accurately reflect the average characteristics of the waste stream over this time period. Composite sampling is most appropriate when the waste stream flow is continuous (or nearly so). Concentration data from composite samples allow pollutant mass loadings to be calculated. Composite samples may be collected manually, but the most common method used by Branch associates involves automated sampling equipment.

As with all sampling methods, the equipment used and its preparation must be consistent with the corresponding requirements of the parameters to be analyzed. For automatic sampling devices, this includes sample intake lines, strainers, internal pump components, sample aliquot bottles, and any other mechanical components that contact the sample. Teflon sample tubing is available for compositing organics (except for VOCs that must always be collected as grab samples). Sample strainers of various materials are in use, including Teflon, plastic, and stainless-steel models. Internal sample aliquot bottles made of glass and plastic are on hand. Disposable aliquot containers and dedicated holders are available to minimize the amount of acid-rinsing that must be performed for metals sample compositing. All internal sampler components must also be cleaned according to the corresponding parameter protocol. For example, when sampling for metals, dilute nitric acid must be circulated through the pump mechanism and sample intake line. The Branch sampling equipment distributes sample directly to the aliquot bottles by way of a flexible tube that is part of the pump assembly itself. This greatly reduces the amount of material that must be properly prepared.

Most parameters require samples to be kept “cool” (six degrees Centigrade or below) during the compositing period. The refrigerated Glacier ISCO sampler should be used in feasible locations where on-site electrical power can be utilized. All other automatic samplers in use by the Branch have internal chambers for the placement of ice to meet this requirement. In the case of high ambient temperatures during summer months or locations inside factories, replenishment of the ice during the compositing interval is necessary.

Additional concerns pertaining to the use of automatic sampling equipment include:

- The pumping mechanism must be able to lift fluid a vertical distance of at least twenty (20) feet at a velocity of at least two (2) feet per second.

- Aliquot volume must be adjustable; aliquot volume should be verified at the start of the compositing period with a graduated cylinder (100 mL minimum aliquot volume).
- The sample intake line must be purged prior to collection of each aliquot.
- Clean or new sample intake tubing must be used for each installation.
- The minimum inside diameter of the sample intake line must be 1/4 inch.
- Care must be taken to ensure suitable mixing when decanting sample from one container to another. This applies to the compositing of individual aliquot bottles as well as the filling of laboratory sample bottles.
- When using an automatic sampler to composite samples for analysis of metals, an equipment blank must be collected. The complete sample collection train (including strainer, sample intake line, pump tubing, and a number of aliquot bottles) must be rinsed with organic/analyte free water and a portion directed into a metals sample bottle for analysis by the GAEPD Laboratory.
- If the sample intake tubing must be attached to metal pipe for proper positioning in the waste stream, the tubing should be mounted upstream of and away from the pipe or conduit. If possible, the submerged portion of the pipe should be wrapped with protective material.
- When using an automatic sampler for organic, PCB or pesticide sample collection, internal glass bottles must be used that have been solvent-rinsed. The sample intake line and strainer must be Teflon. An equipment blank must be collected and analyzed for the same parameters as the sample itself. New pump tubing must be installed.
- The power source must be capable of running the equipment for the entire compositing period, plus a reserve capacity for grab sample pumping (if necessary). Facility electrical outlets may be used. For locations where power is not available, nickel-cadmium or lead-acid batteries may be used. For extended sampling investigations, deep-cycle marine batteries may be used.

In some situations, it may not be possible to install a Branch automatic sampler. The desired sample point may be within a force-main or may be inaccessible for safety or other reasons. In such cases the facility often has installed specialized sampling devices to accommodate the unusual condition. It is permissible to use a facility's sampler if it meets the protocols required for the parameters to be analyzed.

The Branch's automatic samplers are assumed to be in the custody of the facility while they are on their property. This assumption should be presented to the facility representatives when equipment is left unattended. Samplers may be locked in such a way that the composite sample bottles and the sampler's controls cannot be accessed, however, this does not prevent tampering with the sample intake line and strainer. Seals may be placed on the intake line to reduce the possibility of tampering.

Specific operating instructions for each type of automatic sampler are beyond the scope of this document. The reader is referred to the user manuals that accompany the equipment.

These manuals also provide valuable data on pump performance, battery life and maintenance requirements.

Automatic samplers must be kept clean and maintained to ensure proper performance and avoid sample contamination. Each unit should have a log maintained of its maintenance and repair history. The log should identify the samplers by their manufacturer's serial number, model number and DNR Property Control number. Entries should be made in the log at least quarterly denoting the status of the sampler (operable, inoperable, sent off for repair, et cetera). The following procedures should be performed at the frequency indicated:

- Prior To Field Trip - Sample pump operation; forward, reverse, and automatic modes tested. Desiccant, fuses, and batteries checked; batteries charged.
- After Each Field Trip - Outside of sampler cleaned; inside of sample bottle tub rinsed. Used sample bottles replaced with clean ones. Distributor retention nut tightened. Pump tubing cleaned. Sampler body checked for cracks and leaks and repairs made.
- Quarterly - Verify forward, reverse, and automatic pump operation modes. Tighten control box mounting bolts. Examine internal pump hose for splits and cracks, replace if worn. Lubricate pump hose with silicone. Lubricate pump impeller drive spindle with light machine oil.

The following procedure should be followed to denote the status of sampler bottle tubs and mechanical condition:

- The wide latch on the bottle tub should be tagged "Acid Rinsed" when bottles prepared for Metals sampling are installed. The tag should be removed when the sampler is placed in use in the field. If the sample bottles have been prepared for Organics sampling, the tub should be tagged "Solvent Rinsed."
- After use in the field, the wide latch of the bottle tub should be flagged with brightly colored surveyor's tape to indicate that the bottles require cleaning prior to use again.
- A sampler that suffers a mechanical problem should have one of the carrying handles on the sampler body tagged with bright tape and a sample tag inscribed with a brief description of the malfunction.

Composite samples may be "flow-proportioned" or "composited according to flow" to reflect the nature of the waste stream more accurately over time. Periods of greater discharge are represented in the composite by a correspondingly larger volume of sample. This procedure is a requirement of many Water Quality permits issued by the Branch. All Branch associates engaged in composite sampling should be familiar with flow proportioning, not only to enhance the representativeness of their own samples but to be able to demonstrate the procedure to members of the regulated community on request.

There are two procedures for flow proportioning a composite sample:

1. Constant-interval/variable volume
2. Variable-interval/constant volume

The first of these, constant-interval/variable volume consists of individual aliquots collected at uniform time intervals. At the end of the compositing period, varying amounts of each aliquot are transferred to a separate vessel depending on flow data for the respective aliquot collection time. For example, aliquots of 800 mL could be collected at one-hour intervals. If the maximum flow rate during the compositing period was 4.0 MGD, then the amount of sample to be used from each aliquot could be calculated by multiplying the flow (in MGD) by a “proportioning factor” of 200. Thus, for periods of maximum flow, all of the aliquot would be used, intervals of flow rate 2.0 MGD would be represented by 400 mL per aliquot, etc. This procedure is subject to certain assumptions and requirements:

1. The sampler must be equipped with a sufficient number of individual aliquot bottles.
2. Reliable flow information for the aliquot collection times must be available.
3. The amount of sample obtained after flow proportioning must be adequate for all parameters to be analyzed, plus provide for sample “splitting” with the facility.
4. Volume of the smallest aliquot portion composited must be  $\geq 100$  mL.
5. The vessel used for aliquot measurement (graduated cylinder) must be cleaned in accordance with the protocols for all sampling parameters.

The other flow-proportioning method, variable-interval/ constant volume entails interfacing the automatic sampling device with a flow measurement system. Collecting a uniform volume aliquot per set discharge amount proportions the composite. For example, 200 mL of sample could be composited for every 50,000 gallons of discharge. It may be seen that during intervals of greater discharge rates, the aliquots will be collected more frequently, representing such periods with proportionally greater sample volume. The advantages of this approach include:

1. Compositing is done automatically, reducing time required for this task.
2. Any internal sampler bottle configuration may be used (including a single large vessel, again reducing time requirements).
3. Sample aliquot handling is reduced, avoiding a possible contamination vector.
4. The minimum composite aliquot volume requirement of 100 mL is never compromised.

Possible disadvantages of the variable-interval/constant volume procedure may include:

1. An appropriate flow measurement system may not be available or may not be accurate.
2. The flow pattern during the compositing period may not be as anticipated resulting in an insufficient volume of sample being collected (or too much sample may be collected causing overflow or shutdown of the sampler before the compositing interval is complete).

If neither flow-proportioning method is possible, sample aliquots will have to be composited on a constant-volume/uniform interval basis. In some cases, samples collected in this manner may not be acceptable for enforcement purposes. The flow proportioning requirement does not apply to those instances where it can be shown that the instantaneous flow rate did not vary by more than 15% during the compositing interval.

## **8. Groundwater Sampling**

Certain types of wastewater treatment facilities (most notably Land Application Systems and Overland Flow facilities) pose a threat to subsurface waters. Monitoring wells are mandated by the Water Quality permits for such facilities. Sampling of subsurface waters via these constructed wells is required of the permittees at varying frequencies for different parameters.

Occasions arise when Branch associates are called upon to sample groundwater monitoring wells. EPA has operating procedures for Groundwater Sampling Monitoring (2013), which contains standard operating procedures that assure integrity and representativeness of samples collected from the subsurface. When sampling fluids from monitoring wells, the guidelines of the operating procedures document must be combined with guidance from this document regarding appropriateness of sample containers, preservatives, and other considerations for the parameters to be analyzed.

## **F. Sample Handling**

After collection, all sample handling should be minimized. Investigators should use extreme care to ensure that samples are not contaminated. If samples are placed in a cooler, investigators should ensure that melted ice cannot cause the sample containers to become submerged, as this may result in sample cross-contamination. Plastic bags, such as Zip-Lock® bags or similar sealable plastic bags should be used when small sample containers (e.g., VOC vials or bacterial samples) are placed in coolers to prevent cross-contamination. Trace metals sampling is to be conducted according to the EPA document, United States Environmental Protection Agency, Office of Water Engineering and Analysis Division, Method 1669 Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels, July 1996.



## **G. Data and Records Management**

Data and records will be managed according to the policies outlined in the GAEPD SOP# EPD-WPMP-1. Any deviation from the policies outlined in the SOP should obtain prior permission from first the Unit manager, and then the Program manager, and be documented accordingly.

## **H. References**

California State University at Sacramento, Department of Civil Engineering, Operation of Wastewater Treatment Plants, Volumes I, II and III, 8<sup>th</sup> Edition, 2019.

Georgia Department of Natural Resources, Safety Manual, 2018.

United States Environmental Protection Agency, ESD, Water Compliance Unit, Activated Sludge Process Control Testing, 2000.

United States Environmental Protection Agency (Region IV), Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, May 1996.

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United States Environmental Protection Agency, Office of Water Engineering and Analysis Division, Method 1669 Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels, July 1996.

United States Environmental Protection Agency, Office of Water, Process Control Manual: Aerobic Biological Treatment Facilities MD-14, 1977.

United States Environmental Protection Agency (Region IV), Science and Ecosystem Support Division, SESDPROC-306-R3, Wastewater Testing, February 2013.

United States Environmental Protection Agency (Region IV), Science and Ecosystem Support Division, SESDPROC-301-R3, Groundwater Sampling, March 2013.

United States Government Printing Office, Code of Federal Regulations, Title 40, 2020.