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U.S. Environmental Protection Agency
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OPERATING PROCEDURE

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1 General Information

1.1 Purpose

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

1.2 Scope/Application

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

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD 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 SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

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

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version

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

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

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

SESD Operating Procedure for Field pH Measurement, SESDPROC-100, Most Recent Version

SESD Operating Procedure for Field Specific Conductance Measurement, SESDPROC-101, Most Recent Version

SESD Operating Procedure for Field Turbidity Measurement, SESDPROC-103, Most Recent Version

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

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

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

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

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

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

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

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

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

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

1.5 General Precautions

1.5.1 Safety

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

1.5.2 Procedural Precautions

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

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

2 Special Sampling Considerations

2.1 Volatile Organic Compounds (VOC) Analysis

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

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

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

2.2 Special Precautions for Surface Water Sampling

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not 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.
- All background or control samples shall be collected and placed in separate ice chests or shipping containers. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected

contaminated area. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.

- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.
- Samplers must use new, verified and certified-clean disposable or non-disposable equipment cleaned according to procedures contained in SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, or SESD Operating Procedure for Field Cleaning and Decontamination at the FEC, SESDPROC-206, for collection of samples for trace metals or organic compound analyses.

2.3 Sample Handling and Preservation Requirements

1. Surface water samples will typically be collected either by directly filling the container from the surface water body being sampled or by decanting the water from a collection device such as a stainless steel scoop or other device.
2. 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. Place the sample into appropriate, labeled containers. Samples collected for VOC analysis must not have any headspace (see Section 2.1, Volatile Organic Compounds (VOC) Analysis). All other sample containers must be filled with an allowance for ullage.
4. 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 ASB personnel prior to departure for the field investigation. For all other chemical preservatives, SESD will use the appropriate chemical preservative generally stored in an individual single-use vial as described in the SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011). The adequacy of sample preservation will be checked after the addition of the preservative for all samples, except for the samples collected for VOC analysis. If it is determined that a sample is not adequately preserved, additional preservative should be added to achieve adequate preservation. Preservation requirements for surface water samples are found in the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM).
5. All samples preserved using a pH adjustment (except VOCs) must be checked, using pH strips, to ensure that they were adequately preserved. This is done by pouring a small volume of sample over the strip. Do not place the strip in the sample. Samples requiring reduced temperature storage should be placed on ice immediately.

2.4 Quality Control

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

2.5 Records

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

3 General Considerations

3.1 General

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

3.2 Equipment Selection Considerations

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

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

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

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

4 Dipping Using Sample Container

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

5 Scoops

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

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

6 Peristaltic Pumps

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

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

7 Discrete Depth Samplers

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

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

8 Bailers

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

9 Buckets

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

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

10 Submersible Pumps

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

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

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

11 Automatic Samplers

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

12 Trace-Level Mercury Sampling

In order to prevent contamination during sample collection, Region 4 has developed this sampling procedure for trace-level mercury analysis (< 1 part per trillion). This procedure is based on EPA Method 1669.

A vacuum chamber assembly is utilized to collect surface water samples for trace-level mercury analyses. The vacuum chamber assembly consists of the following: 1) an airtight acrylic, cylindrical chamber with an o-ring sealed lid to hold the sample bottle, 2) a Teflon® sample tubing that connects to a centered Teflon® compression fitting on top of the chamber. The other end of the tubing passes through a rigid Teflon® pole for stability and has a modified magnetic screen holder at the intake, and a hand vacuum pump. The chamber is designed to hold a 2-liter sample bottle; however, smaller sample containers may be utilized with a spacer inserted into the chamber. A two inch square of 100 µm Nitex® screen is used on the magnetic screen holder at the intake to prevent large pieces of debris from entering the sample. The screen does not prevent the passage of particulate organic matter which is often prevalent in surface water. The vacuum chamber has a second off-center compression fitting with a 4 inch piece of Teflon® tubing inserted in the fitting. A piece of clear Tygon® tubing approximately 18-24 inches long is placed over the small piece of Teflon®. The Teflon® adds stability to the tubing and keeps it from crimping. The Tygon® is attached to the hand pump and the chamber with electrical tape. The Nitex® screen intake is inserted into the water to be sampled and a vacuum is pulled on the chamber by means of the hand vacuum pump, thus drawing a water sample into a sample container placed directly beneath the intake tubing within the chamber.

Teflon® bottles or 300-Series glass bottles with single use Teflon®-lined caps may be used for sample collection. All sample containers used for collection of trace-level mercury water samples must be pre-cleaned in a laboratory as described in EPA Method 1631. Teflon® containers should also be etched on the outside of the bottle with a unique identification number for QA purposes. All bottles for trace-level sampling must be double bagged in re-sealable bags. Water samples collected for total, inorganic, methyl or ethyl mercury analyses are pumped into appropriately cleaned bottles. Preservation should be done in a clean room laboratory that has been specifically prepared for the preparation of trace level samples (positive pressure ventilation, sticky floor mats, etc.). Preservation must occur within 48 hours of sample collection, sooner if possible. Region 4 utilizes laboratory preservation of trace-level mercury samples in order to minimize the potential for contamination, and if split samples are required, they must be split in a trace-level clean room laboratory.

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

- bottle blanks

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

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

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

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

Duplicate samples are discrete samples collected at the same site and time to measure variability of collected samples and to assess sample collection consistency. Sample splits are aliquots of a minimum 500 ml poured from a single ambient sample. They must be split in a trace-level clean room laboratory.

In order to prevent cross contamination in samples analyzed for trace-level mercury in ambient surface waters, clean sampling protocols must be employed throughout the

sampling effort. For each sampling event, one sampling team member is designated as “clean hands” and one as “dirty hands” (see below). All operations involving contact with the sample bottle and transfer of the sample from the sample collection device to the sample bottle are handled by the individual designated as “clean hands.” “Dirty hands” is responsible for preparation of the sampling device (except the sample container) and for all other activities that do not involve direct contact with the sample.

Prior to sample collection with the vacuum chamber assembly, the Teflon® line is cleaned at each station by rinsing with ambient water as follows: A 2-liter poly bottle is placed into the chamber and filled half full with ambient water. The bottle is swirled to rinse it and the water is discarded downstream of sampling area. The same 2-liter poly bottle can be used at each station. Additional cleaning measures are not recommended as long as the chamber assembly is only used to collect ambient surface water samples. Detergent washes and acid rinses are not conducted due to potential mercury contamination from these solutions. If applicable, samples for other analyses can be collected in a poly bottle with the vacuum chamber assembly but should be collected before the trace-level sample as an additional means of flushing the sampling line prior to collection of the trace-level samples. It is not necessary to implement the “clean hands”/ “dirty hands” method for collection of non-mercury samples, but latex or vinyl gloves should be worn when any samples are collected.

Following are procedures for cleaning the vacuum chamber tubing and collection of ancillary water quality samples, if applicable:

1. Carefully approach the sampling station from downstream and downwind if possible.
2. While wearing latex or vinyl gloves, place an uncapped 2-liter poly bottle into the chamber and secure the chamber lid by attaching the spring-loaded clamps.
3. Place a new square of 100 µm Nitex® screen in the magnetic screen holder. Place the intake beneath the surface of the water (mid-depth or six inches, whichever is less) and hold firmly in place. Care should be taken not to disturb sediment particles in very shallow waters (< 4 inches deep).
4. Squeeze the hand pump until liquid starts to fill the bottle in the chamber. When the bottle is approximately half full, release the vacuum on the chamber, remove the bottle, swirl the contents and discard the water downstream. Repeat this rinse. If ancillary water quality samples are to be collected, return the 2-liter poly bottle to the chamber and pump the required volume of water to fill the appropriate ancillary sample containers. Remove the 2-liter bottle from the chamber and cap. Fill the ancillary sample bottles upon completion of the mercury sample collection.

Water samples for trace level mercury analyses should be collected immediately after the ancillary water samples have been collected according to the following procedures:

1. “Clean hands” should put on a pair of latex or vinyl gloves, then a pair of shoulder length polyethylene gloves.
2. “Dirty hands” should put on a pair of latex or vinyl gloves, retrieve the double bagged trace level sample bottle from the cooler, and open the outer bag. “Clean hands” should open the inner bag and remove the pre-cleaned Teflon® or glass bottle.
3. “Dirty hands” should open the lid on the chamber. “Clean hands” should place the sample bottle in the chamber, remove the bottle top and place it inside the chamber with the bottle.
4. “Dirty hands” should close and secure the chamber lid and using the hand pump, fill the container. The sample container should be filled to overflowing. “Dirty hands” should then release the vacuum and open the lid on the chamber.
5. “Clean hands” should place the top on the sample bottle, remove it from the chamber and place it in the inner bag and seal the bag. “Dirty hands” should seal the outer bag and place the sample in the black bag in the dark cooler. Only coolers dedicated to storage and transport of trace-level mercury samples should be used.