Laboratory Support Section: Standard Operating Procedure for Packing, Shipping and Tracking Sampling Kits

1. INTRODUCTION AND SCOPE

This standard operating procedure details the packing, shipping, and tracking of sampling kits. Laboratory clients or Scientific Services Support Program (SSS) staff request sample analyses and supplies using the Laboratory Information Management System (LIMS) database. The sampling event is assigned a Request ID (RQ) that is reviewed and approved by a designated laboratory manager. Approved RQs are assigned to individual receiving room personnel, who then pack and ship the sampling supplies to the clients.

2. PREPARING AND PACKING SAMPLE KITS

Receiving room personnel will review their assigned backlog of RQs in the LIMS daily. View or print the cooler packing backlog under *Laboratory → Receiving → Cooler Packing,* click *Cooler Packing Backlog*. Select technician's name from the list, RQ statuses 'A' and 'W', and date range. Either open a template or use the arrow key to select fields, then click the *View→ Generate Report* icon. RQ Status Summary Codes are as follows:

T or P – RQ has not been approved by either chemistry, biology or both.

A – RQ is **A**pproved and ready to pack, if kit is requested.

W – Packing list has been printed and kit is **W**aiting to be packed.

S – Kit has been **S**hipped, or is ready for pickup.

R – Samples have been **R**eceived and logged in under the RQ.

2.1. Generate Packing List(s)

Packing Lists provide the inventory of containers and supplies that the clients need for a specific RQ. The packing process is initiated and the RQ status changes from 'A' to 'W' when a packing list is printed. To print a packing list, select *Laboratory→Receiving→Cooler Packing*. Select the *Packing Lists* icon or use the drop-down list for *Print/Packing Lists*. Select the date range for the RQs and appropriate Status. Select one or more RQs. To select multiple RQs hold down the Ctrl key while selecting RQs. Select the *Print Packing List(s)* button to display the packing list(s) and print to a BIZHUB. The Receiving Floor Supervisor may also print packing lists and distribute them to the staff.

2.2 . Collect and Prepare Items to Ship

Collect all necessary supplies that are itemized on a packing list for an RQ. **Review the Comments section of the packing list for special instructions or supplies.**

The LIMS test IDs, container descriptions, and preservation for frequently requested analyses may be found in Appendix 1.

Containers for mercury analyses (e.g., W-CH3Hg, W-HG-H, W-HG-AF) are packed in the metals lab and shipped separately to the customer.

Items included in sampling kits are:

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- Sample containers (labeled and packed appropriately).
- Field sheets, if requested.
- Temperature control bottles (1 per cooler).
- FedEx Return Shipping Labels (excluding SFWMD).
- Packing List.
- Bags for storing bottles (large garbage bags, bubble wrap bags and smaller zip lock bags).
- Bubble wrap packing material.
- Zip ties.
- Sampling accessories, if requested.
- Preservative solutions, if requested. Check with supervisors when shipping preservatives to ensure compliance with DOT shipping regulations.
- DEP chain of custody forms **for criminal cases** or if requested.
- Custody seals **for criminal cases.**

2.2.1. Sample Containers

Collect the required containers as described on the packing list. Save the certification paperwork for each box of containers in the designated location. The DEP Laboratory uses new containers for all analyses except for some biology tests (e.g., MW-FW-QLDC, MI-MN-QNNC, BSY-CD-FCD, BSY-CD-FFS). The Biology Section washes these sampling containers. Vials for VOC analyses are prepped prior to packing in kits (W-VOC-*). Refer to section 4.0 for VOC vial prep procedure.

Scan or type the bottle lot numbers into the LIMS. To scan in the lot number, select *Laboratory→Receiving→Cooler Packing*. Select *Scan / Edit Bottles & Coolers,* and type the RQ. Place cursor in the *Scan Bottle Batch Label* space, select the appropriate number of bottles, and scan in the lot # for every bottle packed. Enter the number of coolers packed and click *Done*. Document the bottle lot numbers on the packing list.

2.2.2. Analyses Labels

The analyses labels for an RQ are generated in the LIMS *Cooler Packing* module. Select the *Test Labels* icon. Highlight the RQ, click OK. The number of labels for each group of tests can be edited by highlighting the correct row and clicking on the "#Labels" column header. Print the labels for the selected RQ using a SATO printer, and adhere the analyses labels to the corresponding bottles.

Place preservative indicator stickers or colored "dots" on the container labels as appropriate. Some analyses may generate additional labels, such as "Fill Jar ½ Full". Adhere these labels to the appropriate containers.

2.2.3. Preservatives

Determine if preservatives are required for analyses or requested in the Comments section of the packing list. If shipping snap vials of acid, wrap each vial in absorbent material, label with preservative sticker, and insert all vials in a sealable bag labeled with the acid expiration date. See Floor Supervisor for instructions on packing bulk acids. Record the preservative type, expiration date, and lot # on the packing list; include the company name if using snap vials. Any coolers containing bulk acids must display the label, "This package conforms to 49 CFR 173.4 for domestic highway or rail transport only."

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2.2.4. Field Sheets

Some customers maintain their own field sheets (e.g., Ambient, SFWMD). It is not necessary to print field sheets for these customers. To print field sheets for other customers, select *Cooler Packing*, then select the *Field Sheets* icon. Type the RQ and click *OK.* Select a BIZHUB printer and configure to print double-sided to capture information printed on the backs of the field sheets. If requested a separate field sheet for Department of Health customers is available electronically.

For criminal cases: Include DEP Chain of Custody Forms if the RQ is a criminal case. Check the top right-hand corner of the Packing List. If it is marked "Criminal" then COC forms must be packed along with the field sheets.

2.3. Packing the Coolers

Select appropriately sized coolers for the kit. Most kits will require ice so be sure the cooler has enough space for both the ice and containers. Pad the cooler with protective bubble wrap as needed for glass containers and place all glass bottles and jars in bubble wrap bags to prevent breakage. Pack plastic bottles in zip lock bags (one bag per sampling site - e.g. place the metals and nutrients bottles for 1 site into a zip lock bag).

For Criminal Cases: Sign and date custody seals and place them across the bag closures or on the container caps. Place them so that the bag or cap cannot be opened without damaging the seal. Do not place custody seals on the outside of the coolers since they can be easily damaged during shipment.

Record the number of coolers associated with the RQ on the designated section of the packing list**. Date and sign the packing list**. This is very important for custody documentation for the containers/samples.

Scan the completed packing list to the Packed RQs folder for a year, and place the original packing list, field sheets, and a pre-printed FedEx return shipping label and mailing pouch for each cooler in the request into a zip lock bag. For new customers, include a copy of the sample shipping instructions (Appendix 2). Send FedEx shipping labels to all *interna*l customers except Ambient; their shipping labels are in the drawer labeled "Ambient". Some customers may include a comment indicating that no shipping label is needed.

Place one temperature control bottle in each cooler. A temperature control bottle is a 125mL plastic bottle filled with tap water and labeled "Temperature Control". Place one large heavy-duty plastic bag and zip-tie in each cooler for the customer's use.

Add bubble wrap to all coolers that contain glass containers or pre-preserved bottles and pack securely so that the bottles will not be broken during transport. Place the zip lock bag with the paperwork on top of the bottles in the cooler. If shipping multiple coolers for an RQ, place the paperwork in the cooler labeled 1 of X.

2.4. Shipping Address and RQ Labels

Adhere a "Send Coolers To", a FedEx shipping, and an RQ label to each cooler.

To print a "Send Coolers To" label, select *Laboratory→Receiving→Cooler Packing*, and select the *Address Labels* icon. Highlight the RQ and click OK. Check the address. Enter the number of address labels needed (1 per cooler) and print to a SATO printer.

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Generate and print a FedEx shipping label for each RQ using the receiving FedEx account. Cooler weight is required to print FedEx labels so weigh cooler prior to opening the FedEx account screen. All coolers are shipped FedEx -Ground, unless a supervisor approves an express method.

Use the shipping bar code from the FedEx label to generate corresponding RQ labels. To print an RQ label, select *Cooler Packing*, then select the technician's name from the "Packed By" drop down list. Click the *RQ Labels* icon and select the RQ. Enter the number of labels needed (1 per cooler), then scan the FedEx bar code on the shipping label. You may scan more than one bar code if there are multiple coolers in the request. Click *Done* when finished and print to a SATO printer. Printing the RQ label sets the shipping date and time in LIMS and changes the status of the RQ from 'W' to "S". An email is automatically generated and sent to the customer with their FedEx tracking number.

Adhere the "Send Coolers To" label(s) to the top left-hand corner of each cooler lid. Adhere the RQ label(s) to the top right-hand corner of each cooler lid and record the number of coolers on each RQ label (e.g. 1 of 3, 2 of 3 etc.). Verify that the address on the FedEx bill is the same as the address on the LIMS packing list and "Send Coolers To" label. Place the FedEx shipping label in a plastic pouch, attach the pouch to the coolers (match the label to the cooler by the weight), and seal the pouch.

Tape the cooler lid(s) closed with packing tape to ensure that there is no spillage during shipment.

Transport the coolers to the loading dock and place in the designated pick-up area for FedEx Ground (or Express, if approved). If FedEx has already come then leave the coolers in the receiving room until the next day.

3. PACKING EXTRA SUPPLIES FOR SPECIFIC ANALYSES

3.1. Volatiles and Fumigants in All Matrices, (W-VOC-* S-VOC-*, WAS-VOC-*, W-FUMGNT-A, W-FUM)

Add one Trip Blank for every 10 samples, or one trip blank for each cooler shipped with any of these analyses, i.e., any cooler with volatiles or fumigants requires a trip blank.

3.2. Cyanide in Water (W-CN-*)

Assemble the test kit using the supplies listed below.

- One copy of Appendix 3 – instructions for field preservation.
- Pre-prepared, 30 mL vial of lead carbonate.
- Two sodium thiosulfate tablets per sample, to be used only if needed.
- Two Sen Safe chlorine test strips per sample.
- One strip of the lead acetate test paper per sample.
- Sodium hydroxide solution or vials (one vial per sample) – see Floor Supervisor regarding shipping under the DOT regulations or if there are many samples.
- One copy of the MSDS for Lead Carbonate.

3.3. 1,4-Dioxane in Water (W-1-4-DIOX)

Adhere a green dot to the sampling container as indicated on the packing list. Assemble the kit using the supplies listed below:

- One copy of Appendix 4 – field preservation instructions.
- Pre-prepared container(s) of sodium sulfite.

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- Pre-prepared container(s) of sodium bisulfate
- One ¼ teaspoon – only if requested in the packing list comment.

3.4. Sulfide in water (OV-SULFD-W)

- Include zinc acetate /sodium hydroxide vials to preserve samples.
- One copy of Appendix 4 – field preservation instructions

3.5. Analyses requiring Chlorine testing

Adhere a blue dot to the sampling container as indicated on the packing list.

- Include two Sen Safe chlorine test strips and two sodium thiosulfate tablets per sample, if requested.
- One copy of Appendix 4, field preservation instructions

4. PREPARING VOLATILE ORGANIC (VOC) VIALS FOR SAMPLING KITS

The preparation of Volatile Organic (VOC) sampling kits must be conducted in a clean and controlled work environment due to the ease of introducing contamination into the kits. Volatile contaminates are easily introduced through the air, on skin or clothing, and from other activities occurring within the work space. Consequently, effort and attention must be taken in the preparation and handling of VOC sampling kits to avoid contamination. The vials must be prepared in the Volatiles (VOC) laboratory within the DEP Central Lab.

Wear a clean, dedicated lab coat in the VOC lab to avoid transfer of contamination from clothing. Avoid wearing cologne, perfume, or heavily scented lotions. If you have recently entered one of the semi-volatile laboratories, do not immediately come into the VOC laboratory since solvent vapors may linger on your clothing.

Before opening any vials for preservation or trip blank preparation verify that the VOC staff are not working with anything that could contaminate the VOC sampling kits. Be aware of your work environment and do not hesitate to ask staff or the supervisor about the possibility of cross contamination from other activities within the workspace.

When working with VOC vials, dispose of any damaged vials or septa. Look for chips or defects on the sealing glass surface or small cracks in the vials.

4.1. Acid Preserved Vials

Add 130 uL of 1:1 hydrochloric acid to the vials using a repeater pipet for the preserved tests (W-VOC-MS-A or W-VOC-A-R and Trip Blanks).

When capping the vials, ensure the septa's Teflon side is facing down towards the vial's sealing surface. Place 2, 3, or 4 vials in a small bubble bag with one small packet of absorbent. Place the 2, 3, and 4 vial sets into separate, large zip lock bags labeled with:

- Vial lot number, expiration date, and the suffix "-P" (e.g. 007940350-01-11-2013-P).
- Lot number and expiration date of the acid used to pre-preserve the vials

4.2. Trip Blanks

After acidifying the vials, fill with de-ionized (DI) water until meniscus is just over the top. Do not overfill or the acid will be diluted. Only use the DI water from the VOC Lab's Solution 2000 DI water unit. Do

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not use the main lab's DI water supply. Consult the VOC staff if there is any uncertainty regarding the appropriate water supply to use.

After capping, turn the vial over to ensure that there are no air bubbles.

Place 2 trip blank vials into a small bubble bag with one small packet of absorbent. Place vial sets into a large outer bag and label with:

- Vial lot number, expiration date, and the suffix "-TB" (e.g. 007940350-01-12-2013-TB).
- Lot number and expiration date of the acid used to pre-preserve the vials

4.3 Unpreserved Vials

Acid is NOT added to the unpreserved analysis vials (W-VOC-MS or W-VOC-R). Place 2 or 3 vials in a small bubble bag with one small packet of absorbent. Place the vial sets in a large zip lock bag and label with:

- Vial lot number, expiration date, and the suffix "-U" (e.g. 007940350-01-11-2013-U).

4.4. Packing VOCs

For customers SIS and OER, pack the 3 vial sets, one for each sample. For all other customers, pack the 2 vial sets, one for each sample. Lab QC for VOCs is 10%, so include one bag of 4 vial sets for every 10 samples.

5.0 PACKING ROOM INVENTORY

Each person who uses the packing room and supplies is responsible for ensuring that the supplies are adequately stocked. If an item is depleted during packing, a cache of frequently used packing supplies is available in storage room B329. Supply availability and requests are monitored by a designated supply attendant. If the necessary items are not available in either location, a request should be submitted to the supply attendant. All requests for packing supplies should be emailed directly to the supply attendant.

Receiving staff may use the Bottle Count feature in the Cooler Packing module to determine the supplies they will need for the upcoming week.

Tickets received by email between 8:00 a.m. and 12:00 noon will be delivered to the stockroom at noon and filled on the same day.

Tickets received by email between 12:00 and 5:00 will be delivered to the stockroom by 5:00 p.m. and filled by 9:00 a.m. the following day.

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Appendix 1- page 1 of 4

DEP Laboratory Sampling Containers, Preservation, and Holding Times

Table 1: Frequently Requested Inorganic Analyses in Aqueous Matrices

Tests listed within one row can share the same container. All test codes ending with "–R" are for EPA SW846 methods (RCRA, Waste). All other tests are for the Clean Water Act methods.

Test Description	Bottle Type	Preservative
Metals		
W-ICP, W-ICPMS, W-HG-H, W-ICP-R, W-ICPMS-R	500 mL to 1L clear plastic bottle. Use 1L if more than 2 tests are requested.	Nitric acid[1]
Field Filtered Metals (without mercury): W-ICP-F, W-ICPMS-F	125 mL to 250 mL clear plastic bottle. Use 250 mL if more than 1 test is requested.	Nitric acid[1]
Field Filtered Metals with Mercury: W-HG-H-F, W-ICP-F, W-ICPMS-F	500 mL clear plastic bottle	Nitric acid[1]
Nutrients and Major Anions		
Acid Preserved Nutrients: W-NH3, W-TKN, W-NO2NO3, W-TP, W-TOC	Clear plastic bottle[2] 500 mL for 4 or more tests	Sulfuric acid[3] and ice
Acid Preserved, Field Filtered Nutrients: W-NH3-F, W-NO2NO3-F, W-TKN-F, W-TP-F, W-DOC	Clear plastic bottle[2] 500 mL for 4 or more tests	Sulfuric acid[3] and ice
Unpreserved Nutrients and Major Anions: W-ALK, TURBIDITY, W-COLOR, W-NO3-IC, W- SO4-IC, W-CL-IC, W-TDS, W-COND, W-F, W-TSS, W-PO4, W-NO2 etc.	Clear plastic bottle[2] 125 mL and up to 1 L	Ice
Unpreserved, Field Filtered Nutrients and Major Anions: W-ALK-F, W-SO4-IC-F, W-CL-IC-F, W-PO4-F, W-F-F, W-SI-ICP-F, W-NO2-F etc.	Clear plastic bottle[2] 125 mL and up to 1 L	Ice

[1]Preserve to pH < 2.0. Add 1 mL of concentrated nitric acid for up to 500 mL of sample volume.
[2]Bottle size may vary depending on the number and types of tests requested.
[3]Preserve to pH < 2.0. Add 1 mL of concentrate sulfuric acid for up to 500 mL of sample volume. Adjust the amount of acid for smaller sample volume. Excess acid may cause interference with some nutrients analyses.

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Appendix 1- page 2 of 4

Table 2: Frequently Requested Organic Analyses in Aqueous Matrices

Tests listed within one row can share the same container. All test codes ending with "–R" are for EPA SW846 methods (RCRA, Waste). All other tests are for the Clean Water Act methods.

Test Description	Bottle Type	Preservative
Pesticides / Herbicides		
W-PESTNP-R, W-PST-CL-R, W-PCB-R, W-PSCL-TQ, W-PSNP-TQ	1L amber glass bottle	Ice
W-PC-AA-SF, W-NP-AA-SF	1L amber glass bottle	Ice
W-AHB-AA-R, W-AHERB-AA, W-UHB-AA, W-UHERB-AA and W-SUC-AA-RR	1L amber glass bottle	Ice
W-PDQUAT	250 mL brown plastic	1 mL sulfuric acid[1] and ice
W-AHB-AA-R or W-AHERB-AA	1L amber glass bottle	Ice
W-CARB[2]	1L amber glass bottle	Monochloroacetic acid
W-FUM	40 mL amber glass vial	Ice
W-GLYPH	40 mL amber glass vial	Ice
Base Neutral, Acid Extractable and Petroleum Products		
W-BNA, W-PAH or W-BNA-R	1L amber glass bottle	Ice
W-FL-PRO	1L amber glass bottle	Sulfuric acid[1] and Ice
W-1-4-DIOX (1,4-Dioxane)[3]	1L amber glass bottle	Sodium Bisulfate
Purgeable		
W-VOC-MS, W-8260B-HP, W-FUMGNT, W-624-AA, or W-VOC-R	40 mL glass vial: fill vial to top (no air in vial)	Ice[4]
W-VOC-MS-A, W-FUMGNT-A, W-8015c, or W-VOC-A-R	40 mL glass vial: fill vial to top (no air in vial)	Hydrochloric acid[5] and ice

[1]Preserve to pH < 2.0. Add 1 mL of concentrated sulfuric acid per 250 mL and 2 ml concentrated sulfuric acid per 1 liter of sample volume.
[2]Sample containers are pre-preserved with monochloroacetic acid solution by DEP Lab staff
[3]Refer to Appendix 4 for details on W-1,4 Dioxane collection and preservation.
[4]Add up to 3 mg of sodium thiosulfate per 40 mL of sample volume when residual chlorine is present.
[5]Sample containers are pre-preserved with hydrochloric acid by DEP Lab staff.

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Appendix 1- page 3 of 4

Table 3: Inorganic and Organic Soil/Sediment/Waste Samples

Tests listed within one row can share the same container. All test codes ending with "–R" are for EPA SW846 methods (RCRA, Waste). All other tests are for the Clean Water Act methods.

Test Description	Bottle Type	Preservative
For ALL soil/sediment/waste tests except for VOC analyses and overflow lab analyses (i.e., S-VOC-MS, S-8260B-AA, TCLP-VOC, SPLP-VOC, WAS-VOC-MS and tests starting with "OV").	Glass jar (fill half full). Jar size may vary depending on how many tests are scheduled. For metals or TOC only, a plastic jar may be used.	Ice
S-VOC-MS, S-8260B-AA (Soil/Sediment tests).	One set contains one 25g EnCore sampler and a 250-mL glass jar with septa. Fill jar completely, and eliminate as much air space as possible.	Ice[1]
TCLP-VOC, SPLP-VOC, WAS-VOC-MS	250 mL glass jars with septa lid. Fill jars completely and eliminate as much air space as possible.	Ice
OV-: Collect samples for tests beginning with "OV- "in a separate jar. These samples will be analyzed by an outside laboratory.	Glass jar (fill half full). The glass jar varies in size depending on how many tests are scheduled.	Ice

[1]Store EnCore samples on ice and freeze within 48 hours of collection. Wipe the outer edge of the jar before closing to ensure a tight seal. Refer to SW 846, EPA Method 5035 for additional details on sampling procedures.

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Appendix 1- page 4 of 4

Table 4: Frequently Requested Biology Analyses

Test Description	Bottle Type	Preservative
Chlorophyll, BOD, Microbiology		
CHLSUITE- or CHL-CORR-	1L brown plastic bottle	Ice
BOD-INHB (=CBOD) or BOD-UNIN	1L clear plastic bottle	Ice
Fecal and Total (non-chlorinated water)	120 mL plastic cup with orange screw-cap	Ice
Fecal, Total, Enterococci (chlorinated water)	120 mL plastic cup with white screw cap and thiosulfate pill for de-chlorination	Ice
Fecal, Total, Enterococci (Overflow)	120 mL plastic cup with white screw cap and thiosulfate pill for de-chlorination	Ice
E. coli and Enterococci	250 mL plastic bottle	Ice
Taxonomy		
MI-FW-QLDC [freshwater macroinvertebrate taxa, 20 dipnet composite (AKA a Stream Condition Index (SCI) sample)]	2 L plastic jar	10% formalin
ALGAL-ID or BLOOM-ID	Any, often 50 mL plastic tube	Ice
PKD-QN-C and DTY-QN-C	1 L brown plastic bottle	Lugols or ice then preserve with Lugols within 24 hours

Table 5: Holding Times for Frequently Requested Analyses in Water

Analysis	Holding Time
BOD, CBOD, Chlorophyll	48 hours
Turbidity, Color, Nitrate, Ortho-Phosphate, Nitrite	48 hours
TDS, TSS, Sulfide	7 days
Alkalinity, Cyanide	14 days
NH3, TKN, TP, NO2NO3, TOC	28 days
SO4, Chloride, Fluoride, Conductivity, Silica	28 days
Mercury	28 days
Metals (except mercury and Cr VI)	6 months
Organics (Pesticides, BNA, Herbicides, TRPH)	7 days
Volatiles (preserved on ice)	7 days
Volatiles (preserved on ice and with acid)	14 days

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Appendix 2

Instructions for Packing and Shipping Samples to the DEP Central Lab

- Place plastic sample bottles inside the Ziploc bags provided.
- Place glass containers in the bubble wrap bags provided.
- Place all sample containers and ice inside the large heavy-duty plastic bag provided and close securely with a knot or zip tie to prevent melting ice leaking from coolers. Samples may also exceed the required temperature ranges if they are not in the bag with the ice.
- Place all paperwork in the zip lock bag provided and tape to the inside of the cooler lid. This helps prevent the papers from getting wet.
- Tape the cooler closed so that the lid will not open during shipment.
- Ship the cooler(s) to the lab using the FedEx shipping bills provided or another priority overnight service for next morning delivery. Ship the coolers to the following address:

FL Department of Environmental Protection
Central Laboratory
2600 Blair Stone Rd.
Tallahassee, FL 32399-2400
Attn: Sample Receiving

Important! The lab is not open on Saturdays, Sundays, or state holidays. Please do not ship coolers on a Friday or the day before a holiday.

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Appendix 3

Field Preservation for Cyanide in Liquid Samples

There are 2 tests performed prior to preservation with NaOH. Both the tests and the preservation need to be done in the field.

1. Test 1

Chlorine decomposes cyanides. If the sample is suspected of containing chlorine then perform the following test:

> Follow the instructions with Sen Safe Free Chlorine Water Check test strips to determine if free chlorine is present in sample. If chlorine is present then treatment is needed. If there is no color change then go to Test 2.
>
> Sample treatment for chlorine when using 500 mL sample bottles: Add a tablet (10 mg) of sodium thiosulfate to the sample and swirl to dissolve. Retest with test strips. Continue adding one tablet at a time and retesting until the test strip no longer indicates free chlorine is present.

2. Test 2

Sulfide adversely affects the cyanide analysis. If sulfide is present the sample must be treated in the field. To check for sulfide, perform the following test:

> Place a drop of sample on lead acetate paper. If the paper turns dark then sulfide is present and the sample needs to be treated in the field. If no sulfide is present then the sample is ready to preserve.
>
> **Read the $PbCO_3$ MSDS sheet, included with kit, for handling and disposal instructions. Avoid contact with skin (wear gloves), inhaling dust and ingestion.**
>
> Sample treatment for sulfide: If analysis of particulates is needed filter the particulates and save the filter for recombination with the liquid sample. Add small amounts of lead carbonate to the liquid sample until the test paper no longer turns dark. Filter the precipitate (PbS). See below for disposal of PbS filter. Add the sample particulates, from the initial filtration, back to the liquid sample. Sample is ready to preserve. Proceed to step 3.
>
> *Disposal of PbS filter: PbS is a hazardous waste; please dispose of the filter properly. Ship the PbCO3 bottle back to the lab.*

3. Preservation

Add 10 M NaOH to the sample to bring the pH to between 10 and 12 (add approximately 1 mL per liter of sample). **Ice preservation also required.**

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Appendix 4

Field Preservation Instructions and Chlorine Testing

1,4 Dioxane in water

Sodium sulfite reduces free and combined chlorine in samples that have been disinfected with chlorine and/or chloramine. Approximately 50 mg/L of sodium sulfite is required. De-chlorination must be performed prior to the addition of the sodium bisulfate preservative.

The sodium bisulfate (anhydrous, technical grade "dry" acid) is added to each sample to reduce to pH < 4.0 to act as a microbial inhibitor. Approximately 1.0-1.5 gram/L of sodium bisulfate, or **no more than 1/2 a capful is required**, but it should be checked and additional reagent added if necessary.

Sample Collection (Refer to EPA Method 522)

Allow system to flush until the water temperature has stabilized. Collect samples from the flowing system. Fill sample bottles, taking care not to flush out the sample dichlorination reagent. Samples do not need to be collected headspace free. Cap the bottle and agitate by hand until the sodium sulfite is dissolved. Add enough sodium bisulfate such that the final concentration will be 1 g/L. Cap the bottle and mix until dissolved. Unless field verification of pH is to be performed, keep the sample sealed until just prior to extraction.

Samples must be chilled (on ice) during shipment and must not exceed 10 degrees C during the first 48 hours after collection.

Sulfide in water

Add **one** vial of zinc acetate/sodium hydroxide solution (provided in the sampling kit) to each empty 250 mL sampling container. Fill the sample container with minimal aeration and check that the pH is above 9. If using bulk acid, dd 14 to 20 drops (0.9 mL) of 2 M zinc acetate solution to the empty sample container, collect the sample with minimum aeration, and then add 6 to 8 drops of 10 M sodium hydroxide solution to bring the pH above 9.

Ice preservation also required.

Chlorine testing for select analyses (containers with Blue Dots adhered to labels)

1. The initial chlorine test can be performed by allowing the sample to flow from the source spigot over the test strip to find out the amount of chlorine, if any.
2. If the test strip indicates 0 ppm Total Chlorine, then dichlorination with sodium thiosulfate tablets is not necessary. You may fill the sample container.
3. If the test strip indicates 0.5 ppm or more Total Chlorine, then dechlorinate with sodium thiosulfate tablets
4. Fill the sample container.
5. Drop 1 tablet into the container and shake gently to dissolve.
6. Test for chlorine by pouring a small amount of sample into the cap. Dip a test strip in the cap and keep it in contact with the sample for 30 seconds, moving it back and forth. Check the color

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against the legend included with test strip. Do not pour the water from the cap back into the container.

7. If additional dichlorination is needed, repeat steps 5 and 6 until the test strip indicates 0.4 ppm Total Chlorine or less.

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