

Section B3. Sample Handling and Custody Requirements

Proper sample handling procedures for water, sediment, and biological samples are provided in Tables 6 and 7 (on the following pages), as well as in SOP's for Field Sample Collection provided in **Appendix D** (for all sample types except for biological assessment and benthic infaunal community assessment) and **Appendix G** (biological assessment and benthic infaunal community assessment QAPP's). Table 6 provides a summary of recommended sample containers, sample volumes, initial preservation, and maximum storage times for water samples. Table 7 provides the same information for bed sediment, tissue, and biota samples.

In the field, all samples will be packed in wet ice or frozen ice packs during shipment, so that they will be kept at approximately 4°C. Samples will be shipped in insulated containers. All caps and lids will be checked for tightness prior to shipping.

All samples will be handled, prepared, transported and stored in a manner so as to minimize bulk loss, analyte loss, contamination or biological degradation. Sample containers will be clearly labeled with an indelible marker. Where appropriate, samples may be frozen to prevent biological degradation. Water samples will be kept in Teflon™, glass, or polyethylene bottles and kept cool at a temperature of 4°C until analyzed. Maximum holding times for specific analyses are listed in Tables 6 and 7 on the following pages.

Ice chests are sealed with tape before shipping. Samples are placed in the ice chest with enough ice to completely fill the ice chest. RFA forms are placed in an envelope and taped to the top of the ice chest or they may be placed in a plastic bag and taped to the inside of the ice chest lid. It is assumed that samples in tape-sealed ice chests are secure whether being transported by staff vehicle, by common carrier, or by commercial package delivery. The receiving laboratory has a sample custodian who examines the samples for correct documentation, proper preservation and holding times.

Contract laboratories will follow sample custody procedures outlined in their QA plans. Contract laboratory QA plans are on file with the respective laboratory.

All samples remaining after successful completion of analyses will be disposed of properly. It is the responsibility of the personnel of each analytical laboratory to ensure that all applicable regulations are followed in the disposal of samples or related chemicals.

Chain-of-custody procedures require that possession of samples be traceable from the time the samples are collected until completion and submittal of analytical results. A complete chain-of-custody form is to accompany the transfer of samples to the analyzing laboratory.

Table 6. Summary of Sample Container, Volume, Initial Preservation, and Holding Time Recommendations for Water Samples

| Parameters for Analysis in WATER Samples | Recommended Containers (all containers pre-cleaned) | Typical Sample Volume (ml) | Initial Field Preservation | Maximum Holding Time (analysis must start by end of max) |
|---|--|----------------------------|----------------------------|---|
| Conventional Constituents in Water | | | | |
| Alkalinity | Polyethylene bottles (see NOTE ⁽¹⁾ below) | 100 ml | Cool to 4°C, dark | 14 days at 4°C, dark |
| Chloride (Cl), Sulfate (SO ₄) and Fluoride (F) | Polyethylene bottles (see NOTE ⁽¹⁾ below) | 300 ml | Cool to 4°C, dark | 28 days at 4°C, dark |
| Ortho-phosphate (OPO ₄) | Polyethylene bottles (see NOTE ⁽¹⁾ below) | 150 ml | Cool to 4°C, dark | 48 hours at 4°C, dark |
| Nitrate + Nitrite (NO ₃ + NO ₂) | Polyethylene bottles (see NOTE ⁽¹⁾ below) | 150 ml | Cool to 4°C, dark | 48 hours at 4°C, dark |
| Total Kjeldahl Nitrogen (TKN) | Polyethylene bottles (see NOTE ⁽¹⁾ below) | 600 ml | Cool to 4°C, dark | Recommend: 7 days Maximum: 28 days Either one at 4°C, dark |
| Total Dissolved Solids (TDS) | Polyethylene bottles (see NOTE ⁽¹⁾ below) | 1000 ml | Cool to 4°C, dark | 7 days at 4°C, dark |
| Ammonia (NH ₃) | Polyethylene bottles (see NOTE ⁽¹⁾ below) | 500 ml | Cool to 4°C, dark | 48 hours at 4°C dark; if acidify, 28 days at 4°C, dark |
| Total Phosphorus (TPO ₄) | Polyethylene bottles (see NOTE ⁽¹⁾ below) | 300 ml | Cool to 4°C, dark | 28 days at 4°C, dark |
| (1)NOTE: The volume of water necessary to collect in order to analyze for the above constituents is typically combined in four 1-liter polyethylene bottles, which also allows enough volume for possible re-analysis and for conducting lab spike duplicates. This is possible since the same laboratory is conducting all of the above analyses; otherwise, individual volumes apply. | | | | |
| Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC) | 40 ml glass vial | 40 ml (one vial) | Cool to 4°C, dark | 28 days at 4°C, dark |
| Total Suspended Solids (TSS) | 500 ml amber glass jar | 1000 ml (two jars) | Cool to 4°C, dark | 7 days at 4°C, dark |
| Suspended Sediment Concentration (SSC) | 500 ml amber glass jar | 500 ml (one jar) | Cool to 4°C, dark | 7 days at 4°C, dark |
| Chlorophyll <i>a</i> Pheophytin <i>a</i> | 1-L amber polyethylene bottle | 1000 ml (one bottle) | Cool to 4°C, dark | Keep at 4°C, dark, but must filter within 48 hours. Filters may be stored frozen up to 30 days. |

| Parameters for Analysis in WATER Samples | Recommended Containers (all containers pre-cleaned) | Typical Sample Volume (ml) | Initial Field Preservation | Maximum Holding Time (analysis must start by end of max) |
|---|---|--|---|---|
| Non-Routine Compounds in Water Samples | | | | |
| OIL AND GREASE | 1-L glass jar with Teflon lid-liner, rinsed with hexane or methylene chloride | 1000 ml (one jar) | Add 2 ml conc. H ₂ SO ₄ to pH <2; cool to 4°C, dark. | 28 days at 4°C, dark |
| PHENOLS | 1-L glass jar with Teflon lid-liner | 1000 ml (one jar) | Add 2 ml conc. H ₂ SO ₄ to pH <2; cool to 4°C, dark. | 28 days at 4°C, dark |
| CYANIDE | 1-L cubitainer | 1000 ml (one cubitainer) | Add 2 ml 1:1 NaOH to make pH > 12; Add 0.6 g ascorbic acid if residual Cl present. Cool to 4°C, dark. | 14 days at 4°C, dark |
| BIOCHEMICAL OXYGEN DEMAND (BOD) | 4-L cubitainer | 4000 ml (one cubitainer) | Cool to 4°C, dark. Add 1g FAS crystals per liter, if residual Cl present. | 48 hours at 4°C, dark |
| CHEMICAL OXYGEN DEMAND (COD) | 1-L cubitainer | 110 ml (one cubitainer) | Add 2 ml conc. H ₂ SO ₄ to make pH <2. Cool to 4°C, dark. | 28 days at 4°C, dark |
| Trace Metals in Water Samples | | | | |
| DISSOLVED METALS (except Dissolved Mercury) | 60 ml polyethylene bottle, pre-cleaned in lab using HNO ₃ | 60 ml (one bottle) if salinity <0.5 ppt 180 ml (three bottles) if salinity >0.5 ppt | Filter at sample site using 0.45 micron in-line filter, or syringe filter. Cool to 4°C, dark. Acidify in lab, within 48 hrs, using pre-acidified container (ultra-pure HNO ₃) for pH<2. | Once sample is filtered and acidified, can store up to 6 months at room temperature |
| DISSOLVED MERCURY | 250 ml glass or Teflon bottle, pre-cleaned in lab using HNO ₃ | 250 ml (one bottle) | Cool to 4°C, dark. Filter in lab within 48 hours, using bench top Hg filtration apparatus. Acidify in lab within 48 hrs, with pre-tested HCL to 0.5%. | Once sample is filtered and acidified, can store up to 6 months at room temperature |

| Parameters for Analysis in WATER Samples | Recommended Containers (all containers pre-cleaned) | Typical Sample Volume (ml) | Initial Field Preservation | Maximum Holding Time (analysis must start by end of max) |
|---|--|--|---|---|
| Trace Metals in Water Samples | | | | |
| TOTAL METALS (except Total Mercury) | 60 ml polyethylene bottle, pre-cleaned in lab using HNO ₃ | 60 ml (one bottle) if salinity <0.5 ppt 180 ml (three bottles) if salinity >0.5 ppt | Cool to 4°C, dark. Acidify in lab within 48 hrs, with pre-acidified container (ultra-pure HNO ₃), for pH<2. | Once sample is acidified, can store up to 6 months at room temperature |
| TOTAL MERCURY | 250 ml glass or Teflon bottle, pre-cleaned in lab using HNO ₃ | 250 ml (one bottle) | Cool to 4°C, dark. Acidify in lab within 48 hrs, with pre-tested HCL to 0.5%. | Once sample is acidified, can store up to 6 months at room temperature. |
| HEXAVALENT CHROMIUM (filtered) | 600 ml plastic or glass bottle | 600 ml (one bottle) | Cool to 4°C, dark No acid | Keep at 4°C, dark for up to 24 hours; must notify lab in advance. |
| HARDNESS | 200 ml polyethylene or glass bottle | 200 ml (one bottle) | Cool to 4°C, dark OR Filter and add 2 ml conc. H ₂ SO ₄ or HNO ₃ to pH < 2; Cool to 4°C, dark. | 48 hours at 4°C, dark 6 months at 4°C, dark |
| Synthetic Organic Compounds in Water Samples | | | | |
| VOLATILE ORGANIC ANALYTES (VOA's) including VOC, MTBE and BTEX | 40 ml VOA vials | 120 ml (three VOA vials) | All vials are pre-acidified (50% HCl or H ₂ SO ₄) at lab before sampling. Cool to 4°C, dark | 14 days at 4°C, dark |
| PESTICIDES & HERBICIDES* <input type="checkbox"/> Organophosphate Pesticides <input type="checkbox"/> Organochlorine Pesticides <input type="checkbox"/> Chlorinated Herbicides SEMI-VOLATILE ORGANICS* POLYCHLORINATED* BIPHENYL AND AROCHLOR COMPOUNDS TPH, PAH, PCP/TCP* | 1-L I-Chem 200-series amber glass bottle, with Teflon lid-liner (per each sample type) | 1000 ml (one container) *Each sample type requires 1000 ml in a separate container | Cool to 4°C, dark If chlorine is present, add 0.1g sodium thiosulfate | Keep at 4°C, dark, up to 7 days. Extraction must be performed within the 7 days; analysis must be conducted within 40 days. |

| Parameters for Analysis in WATER Samples | Recommended Containers (all containers pre-cleaned) | Typical Sample Volume (ml) | Initial Field Preservation | Maximum Holding Time (analysis must start by end of max) |
|--|--|--|--|--|
| Toxicity Testing Water Samples | | | | |
| TOXICITY IN WATER | Four 2.25 L amber glass bottles | 9000 ml | Cool to 4°C, dark | 48 hrs at 4°C, dark |
| Bacteria and Pathogens in Water Samples | | | | |
| <i>E. Coli</i> | Factory-sealed, pre-sterilized, disposable Whirlpak® bags or 125 ml sterile plastic (high density polyethylene or polypropylene) container | 100 ml volume sufficient for both <i>E. coli</i> <u>and</u> <i>Enterococcus</i> analyses | Sodium thiosulfate is pre-added to the containers in the laboratory (chlorine elimination). Cool to 4°C; dark. | STAT: 6 hours at 4°C, dark if data for regulatory purposes; otherwise, 24 hrs at 4C, dark if non-regulatory purpose. |
| <i>Enterococcus</i> | Factory-sealed, pre-sterilized, disposable Whirlpak® bags or 125 ml sterile plastic (high density polyethylene or polypropylene) container | 100 ml volume sufficient for both <i>E. coli</i> <u>and</u> <i>Enterococcus</i> analyses | Sodium thiosulfate is pre-added to the containers in the laboratory (chlorine elimination). Cool to 4°C; dark. | STAT: 6 hours at 4°C, dark if data for regulatory purposes; otherwise, 24 hrs at 4C, dark if non-regulatory purpose. |
| FECAL COLIFORM | Factory-sealed, pre-sterilized, disposable Whirlpak® bags or 125 ml sterile plastic (high density polyethylene or polypropylene) container | 100 ml volume sufficient for both fecal <u>and</u> total coliform analyses | Sodium thiosulfate is pre-added to the containers in the laboratory (chlorine elimination). Cool to 4°C; dark. | STAT: 6 hours at 4°C, dark if data for regulatory purposes; otherwise, 24 hrs at 4C, dark if non-regulatory purpose. |
| TOTAL COLIFORM | Factory-sealed, pre-sterilized, disposable Whirlpak® bags or 125 ml sterile plastic (high density polyethylene or polypropylene) container | 100 ml volume sufficient for both fecal <u>and</u> total coliform analyses | Sodium thiosulfate is pre-added to the containers in the laboratory (chlorine elimination). Cool to 4°C; dark. | STAT: 6 hours at 4°C, dark if data for regulatory purposes; otherwise, 24 hrs at 4C, dark if non-regulatory purpose. |

Table 7. Summary of Sample Container, Volume, Preservation, and Storage Requirements for SWAMP Bed Sediment, Biota, and Tissue Samples (for contaminant analysis)

| Parameters for Analysis | Recommended Containers | Typical Sample Volume (ml) | Initial Field Preservation | Maximum Holding Time |
|---|---|---------------------------------------|----------------------------------|--|
| Bed Sediment Samples | | | | |
| Trace Metals, including Hg and As (except for Se--see below) | 60 ml I-Chem 300-series clear glass jar with Teflon lid-liner; Pre-cleaned | 60 ml (one jar) | Cool to 4°C, dark, up to 14 days | 12 months ⁽¹⁾ (-20°C) |
| Selenium (separate container required) | 60 ml I-Chem 300-series clear glass jar with Teflon lid-liner; Pre-cleaned | 60 ml (one jar) | Cool to 4°C, dark, up to 14 days | 12 months ⁽¹⁾ (-20°C) |
| Synthetic Organic Compounds | 250 ml I-Chem 300-series amber glass jar with Teflon lid-liner; Pre-cleaned | 500 ml (two jars) | Cool to 4°C, dark, up to 14 days | 12 months ⁽¹⁾ (-20°C) |
| Sediment TOC | 125 ml ⁽³⁾ clear glass jar; Pre-cleaned | 125 ml (one jar) | Cool to 4°C, dark, up to 28 days | 12 months ⁽²⁾ (-20°C) |
| Sediment Grain Size | 125 ml ⁽³⁾ clear glass jar; Pre-cleaned | 125 ml (one jar) | Cool to 4°C, dark, up to 28 days | 28 days (4°C) <i>Do not freeze</i> |
| Sediment Toxicity Testing | 1-Liter I-Chem wide-mouth polyethylene jar with Teflon lid-liner; Pre-cleaned | 2-Liters (two jars filled completely) | Cool to 4°C, dark, up to 14 days | 14 days (4°C) <i>Do not freeze</i> |
| <p>(1) Sediment samples for parameters noted with one asterisk (*) may be refrigerated at 4°C for up to 14-days maximum, but analysis <u>must</u> start within the 14-day period, or the sediment sample <u>must</u> be stored frozen at minus (-) 20°C for up to 12 months maximum.</p> <p>(2) Sediment samples for sediment TOC analysis can be held at 4°C for up to 28 days, and <u>should</u> be analyzed within this 28 day period, but can be frozen at any time during the initial 28 days, for up to 12 months maximum at minus (-) 20°C.</p> <p>(3) Sediment samples for TOC AND grain size analysis can be combined in one 250 ml clear glass jar, and sub-sampled at the laboratory in order to utilize holding time differences for the two analyses. If this is done, the 250 ml combined sediment sample must be refrigerated only (<u>not frozen</u>) at 4°C for up to 28 days, during which time the sub-samples must be aliquoted in order to comply with separate storage requirements (as shown above).</p> | | | | |

Table 7 (continued). Summary of Sample Container, Volume, Preservation, and Storage Requirements for SWAMP Bed Sediment, Tissue (for contaminant analysis), and Biota Samples

| Parameters for Analysis | Recommended Containers | Typical Sample Volume (ml) | Initial Field Preservation | Maximum Holding Time |
|---|--|----------------------------|--|----------------------|
| Tissue samples | | | | |
| Fish, crab, and shellfish tissue (for contaminant analysis) | Polyethylene bags (Teflon™ sheets in Ziplock™ bags); or glass (with Teflon™ lid); or polyethylene jar for trace metals sample only | 200g | Freeze until processing | 12 months (-20°C) |
| Biota (Benthic macroinvertebrates) | | | | |
| FRESHWATER | plastic or glass | variable | 90% ethyl alcohol OR 70% isopropyl alcohol OR Add formalin to produce a 5-10% formalin solution Store in dark and away from extremes of hot and cold. | 5 years |
| MARINE | plastic or glass | variable | Add formalin buffered with borax to create a 10% formalin solution. After 2 weeks, sort sample and preserve with 70% ethanol | 5 years |
| Netplankton | amber plastic or glass (Lugol's solution will permeate plastic cubitainers and stain materials in contact with cubitainer) | variable | Rinse net bucket with 3-5% buffered formalin OR If net bucket rinsed with tap water, preserve sample with 1 ml of modified Lugol's solution per 100 ml of sample. Store in dark and away from extremes of hot and cold. | 5 years |

| Parameters for Analysis | Recommended Containers | Typical Sample Volume (ml) | Initial Field Preservation | Maximum Holding Time |
|-------------------------|--|----------------------------|--|----------------------|
| Nannoplankton | amber plastic or glass (Lugol's solution will permeate plastic cubitainers and stain materials in contact with cubitainer) | 500 g | 1 ml of modified Lugol's solution per 100 ml of sample. Store in dark and away from extremes of hot and cold. | 5 years |
| Nekton | plastic or glass | variable | Fix in a 10% formalin solution. After about 1 week thoroughly wash and preserve in 40 % ethyl alcohol Store in dark and away from extremes of hot and cold. | 5 years |

A sample is considered under custody if:

- it is in actual possession;
- it is in view after in physical possession;
- it is placed in a secure area (accessible by or under the scrutiny of authorized personnel only after in possession)

Field Log

Field crews shall be required to keep a field log for each sampling event. The following items should be recorded in the field log for each sampling event:

- date and time of sample collection, name of sampler(s), waterbody name
- sample ID numbers, including etched bottle ID numbers for Teflon™ mercury sample containers and unique IDs for any replicate or blank samples;
- the results of any field measurements (temperature, D.O., pH, conductivity, turbidity) and the time that measurements were made;
- qualitative descriptions of relevant water conditions (e.g. color, flow level, clarity) or weather (e.g. wind, rain) at the time of sample collection;
- a description of any unusual occurrences associated with the sampling event, particularly those that may affect sample or data quality.

The field crews shall have custody of samples during field sampling. Chain of custody forms will accompany all samples during shipment to contract laboratories. All water quality samples will be transported to the analytical laboratory directly by the field crew or by overnight courier.

Laboratory Custody Log

Laboratories shall maintain custody logs sufficient to track each sample submitted and to analyze or preserve each sample within specified holding times.