Montana Department of Environmental Quality

Standard Operating Procedure

Sample Collection for Chemistry Analysis: Water, Sediment, and Biological Tissue



WQDWQPBFM-02, Version 1.0
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Water Quality Planning Bureau

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The Montana Department of Environmental Quality (DEQ) Water Quality Planning Bureau (WQPB) Standard Operating Procedures (SOPs) are adapted from published methods or developed by in-house technical and administrative experts. Their primary purpose is for WQPB internal use, although sampling and administrative SOPs may have a wider utility. Our SOPs do not supplant official published methods. DEQ may provide SOPs to other programs or partners. Distribution of these SOPs does not constitute a requirement to use a particular procedure or method unless stated in other binding communications. This document does not contain regulatory or statutory requirements unless specified.

Any reference to specific equipment, manufacturer, or supplies is for descriptive purposes only and does not constitute an endorsement of a particular product or service by the author or by the DEQ.

Although the WQPB follows this SOP in most cases, there may be situations where an alternative methodology, procedure, or process is used to meet specific project objectives. In such cases, the project manager is responsible for documenting deviations from these procedures in the Quality Assurance Project Plans (QAPPs), Sampling and Analysis Plans (SAPs), and end of project summary reports.

Document Revision and Version History

Revision Date	Version number	Summary of change(s)	Revised sections(s)	Revised by
December 2019	1.0	Initial document	All	Katie Makarowski

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LIST OF FIGURES

ACRONYMS

DEQ	Montana Department of Environmental Quality
GPS	Global Positioning System
H_2SO_4	Sulfuric acid
HAZWOPER	Hazardous Waste Operations and Emergency Response
HCl	Hydrochloric acid
HNO_3	Nitric acid
NaOH	Sodium hydroxide
PCB	Polychlorinated biphenyl
QAPP	Quality Assurance Project Plan
SAP	Sampling and Analysis Plan
SDS	Safety Data Sheet
SOP	Standard Operating Procedure
ULL-Hg	Ultra-low-level mercury
VOA	Volatile organic analysis
WQPB	Water Quality Planning Bureau

1.0 PURPOSE

This document describes the Water Quality Planning Bureau (WQPB) standard operating procedure (SOP) for collecting samples for chemistry analysis. This SOP describes many of the water, sediment, and biological tissue sample collection methods and equipment most commonly used by DEQ.

Each project's Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP) should reference the applicable SOPs in this document that are intended for use during sampling and should also specify for the project:

- Type and quantity of sample media per sample (e.g., water, sediment, tissue)
- List of analytes
- Analytical requirements including analytical method, size and type of sample container, holding time, preservation, and storage
- Reporting requirements (e.g., calculations, wet weight versus dry weight, units, reporting limits, lab reports and electronic data deliverable formats)
- Quantity of samples per analyte
- Quantity and location of sampling sites
- Quantity and timing of sampling events
- Analytical laboratories that will perform chemistry analyses
- Sampling frames or grids
- Sample collection specifications (e.g., filtered, depth-integrated, composited)
- Minimum number of sub-samples for composite samples
- Taxonomic considerations, if applicable (e.g., sorting by order or aquatic versus terrestrial)
- Quality assurance and quality control sample requirements (e.g., duplicates, blanks)

DEQ's Monitoring Parameter Suite table (DEQ, 2019 or most current revision) shows monitoring and analytical specifications applicable to most WQPB monitoring projects.

2.0 APPLICABILITY

The sample collection procedures contained in this SOP are applicable statewide to all surface waterbody types (rivers, streams, lakes, reservoirs, wetlands) unless specified. Selecting the most applicable SOP for chemistry samples is often guided by the analytical requirements for each chemical analyte. For example, unfiltered grab sampling procedures are appropriate if analyzing the total recoverable fraction whereas filtered grab samples are required if analyzing the dissolved fraction. Also, for example, additional decontamination procedures are necessary for organic constituents. The most applicable sample collection method may also depend on site-specific conditions such as depth or wadeability, or project-specific requirements such as the need to composite or ship samples.

3.0 METHOD SUMMARY

This document details standardized procedures for collecting, handling, storing and, in some cases, processing various types of media (i.e., water, sediment, macroinvertebrate and fish tissue) for chemical analyses. Project-appropriate methods and sampling designs should be cited in each project's QAPP or SAP since analytical requirements and data needs are unique for each project.

Chemistry sampling requires measures to avoid contamination and several related considerations are addressed in this document:

- Best practices for preventing contamination
- Decontamination procedures
- Order of operations of field activities to avoid disturbance
- Sample handling and delivery
- Field duplicates and field blanks

Other DEQ SOPs that contain especially relevant information related to this document include:

- Fish Tissue Sampling (WQPBWQS-29) (Mavencamp, 2015)
- Field Equipment Decontamination (WQPBWQM-028) (McCarthy, 2014)

4.0 DEFINITIONS

Ambient monitoring: All forms of monitoring conducted beyond the immediate influence of a discharge pipe or injection well and may include sampling of sediments and living resources (ACWI, 2019).

Benthic: Of, relating to, or occurring at the bottom of a body of water.

Bioaccumulate: The net uptake of a material by an organism from food, water, and (or) respiration that results in elevated internal concentrations (ACWI, 2019).

Blank: A sample of analyte free water used to detect sources of contamination during sampling, transport, storage or analysis of environmental samples (EPA, 2009).

Chain of custody form: A custody record which provides a mechanism for tracking physical samples through sample collection, processing and analysis and document the "chain of custody," the date and person responsible for the various sample handling steps associated with each sample (EPA, 2017).

Decontamination: the neutralization or removal of dangerous substances, radioactivity, or germs from an area, object, or person (dictionary.com, 2019).

Duplicate samples: Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner; used to assess variance of the total method including sampling and analysis (EPA, 2019).

Field blank: A sample of analyte free water poured into the container in the field, preserved and shipped to the laboratory with field samples; used to assess contamination from field conditions during sampling (EPA, 2009).

Holding time: The maximum time allowed between sampling and analysis for results to still be considered valid.

Inorganic compounds: any substance in which two or more chemical elements (usually other than carbon) are combined, nearly always in definite proportions. Compounds of carbon are classified as organic when carbon is bound to hydrogen (Encyclopedia Brittanica, 2019).

Macroinvertebrate: Inverterbate fauna that can be captured by a 500 µm net or sieve (Cushman, date unknown)

Organic compound: Any of a large class of chemical compounds in which one or more atoms of carbon are covalently linked to atoms of other elements, most commonly hydrogen, oxygen, or nitrogen (Encyclopedia Brittanica, 2019).

Parameter: Any of a set of physical properties whose values determine the characteristics or behavior of something (Merriam-Webster Dictionary, 2019)

Quality assurance: An integrated system of management activities involvingplanning, implementation, assessment, reporting, and improvement to ensure that aprocess, item, or service is of the type and quality needed (EPA, 2002).

Quality control: An overall system of technical activities that measures theperformance of a process, item, or service against defined standards to verify that the performance meets the stated requirements (EPA, 2002).

Reporting limit: The minimum value below which data are documente as non-detect (van Buuren, 2017).

Rinsate/Equipment Blank: A sample of analyte freewater poured over or through decontaminated field sampling equipment prior to the collection of environmental samples; used to assess the adequacy of the decontamination process or to assess contamination from the total sampling, sample preparation and measurement process, when decontaminated sampling equipment is used to collect samples (EPA, 2009).

Sample: A finite part of a statistical population whose properties are studied to gain information about the whole (Merriam-Webster Dictionary, 2019).

Toxic: Relating to harmful effects to biota caused by a substance or contaminant (ACWI, 2019).

Trip blank: A clean sample of a matrix that is taken from the laboratory to the sampling site and transported back to the laboratory without having been exposed to sampling procedures; used to assess contamination introduced during shipping and field handling procedures; typically analyzed only for volatile compounds (EPA, 2009).

5.0 HEALTH AND SAFETY WARNINGS

Field personnel should be aware of job hazards associated with collecting samples that could result in personal injury or loss of life. Driving, boating, wading, and chemical safety hazards are especially pertinent to this SOP. Consult the WQPB Job Hazard Analysis form and DEQ's Waterborne Operations Procedure (DEQ, 2016). Personnel must be familiar with health hazards associated with fixing or preserving agents such as dry ice or acids, and must always use caution when using them. Dry ice produces carbon dioxide that may displace oxygen in confined spaces. Acids can cause eye, sinus, or skin damage, and may also ruin clothing, backpacks or other materials. Safety Data Sheets (SDS) for all chemicals used must be available to sampling team members. Personal protective equipment should be worn or used as needed, including gloves, eye protection, respirators, or fume hoods. If sampling will be performed in an exclusion or contaminant reduction zone of a hazardous waste site, sampling personnel are required to have Hazardous Waste Operations and Emergency Response (HAZWOPER) training.

6.0 CAUTIONS

This section contains information pertaining to activities that could result in degradation of samples, equipment damage, or possible invalidation of results.

6.1 PREVENTING CONTAMINATION

The following best practices will help prevent contamination of chemistry samples (USGS, 2006):

- Use new or properly acid-washed sample containers
- Store and transport sample containers with the lids secured
- Avoid hand contact with the lip and interior of containers and lids
- Avoid hand contact with contaminating surfaces (e.g., equipment, coins, food)
- Do not smoke or use tobacco products prior to or during sampling
- Avoid atmospheric exposure (if required) by opening, filling and closing sample bottles underwater, or collecting samples in enclosed chambers
- Cap bottles immediately after sample collection
- Cap bottles of deionized water while preparing field blanks to minimize exposure to atmosphere, dust, etc.
- Adequately clean or decontaminate equipment between use at different sites
- Perform field rinsing (e.g., triple-rinsing of sample containers)
- Use clean hands/dirty hands technique for parts-per-billion trace element sampling
- Store samples upright
- Drain sample storage coolers frequently so sample containers are not submerged in water
- Use sample containers and equipment constructed of materials that are relatively inert with respect to the analytes targeted for the study (high-density polyethylene containers for metals, glass for volatiles, etc.)
- Wear gloves (latex or nitrile, powder-free), as needed, when collecting especially sensitive samples or to avoid contact with the water when you suspect it is contaminated
- Follow appropriate sample handling procedures and minimize sample handling steps
- Ensure adequate training for field personnel
- Follow approved field procedures
- Collect sufficient blanks, duplicates, or other quality control samples
- Collect samples from downstream to upstream to minimize disturbance
- Collect samples upstream from any previous disturbance
- Sample at sites with least contamination or lowest likely chemical concentrations first (e.g., upstream or upgradient from known or suspected sources of contamination)

6.2 EQUIPMENT USE AND MAINTENANCE

Proper use, maintenance, and storage of all equipment or instruments associated with procedures described in this SOP is the responsibility of the field personnel using it. Adhere to user manuals, clean

and calibrate as needed, inspect prior to use, and store in a secure location when not in use. DEQ may provide SOPs and field guides for specific pieces of equipment. Report any problems to DEQ staff responsible for inventorying and maintaining equipment and supplies.

6.3 SAMPLE HANDLING AND DELIVERY

6.3.1 Sample Containers

All water samples should be collected in new bottles or vials provided by the analytical laboratory. The type and size of sample containers for each analyte should be specified in a project SAP. The sample container material depends on the analyte being targeted (e.g., high-density polyethylene, clear or amber glass). Sample container size depends on the volume required by the analytical lab for sample analysis and quality control samples. Sample container lids may be color-coded to indicate the preservative added (e.g., yellow signifies sulfuric acid, red signifies nitric acid, white signifies no preservative).

6.3.2 Sample Storage and Holding Time

Refer to the project SAP for quality control requirements pertaining to sample storage for each analyte.

- Store samples that must be kept cold (< 6°C) in a cooler on regular ice (in the field) or in a refrigerator (in the lab).
- Store samples that must be frozen in a well-insulated cooler on dry ice (in the field) or in the freezer (in the lab).
- Maintain sufficient ice in coolers to adequately surround samples.
- Regularly drain coolers to prevent sample bottles from becoming submerged in water.
- Wrap light-sensitive samples in aluminum foil and/or store in an opaque container/cooler/box.
- Take precautions storing samples containing preservatives; consider chemical safety, ventilation, and flammability.

Holding times are the maximum times allowed between sample collection and sample analysis for results to still be considered valid. Holding times vary depending on sample preservation methods and should be specified per analyte in each project SAP. Samples must be delivered to the lab as soon as possible following collection and within holding time requirements, and samples should be analyzed as soon as possible after being received by the lab. When maximum holding times cannot be met, resampling may be requested. If samples exceed the holding time, laboratory staff may contact project managers prior to analysis to confirm whether or not the lab should proceed with analysis. If samples are analyzed out of holding time, data is qualified with an “H” flag in the database.

6.3.3 Sample Delivery and Shipping

Hand delivery to the lab is preferred:

- Indicate “hand” delivery method on the chain-of-custody form
- Confirm with the receiving lab that proper temperatures were achieved
- Confirm all samples are relinquished and received

If samples must be shipped to the lab:

- Ship samples same-day or overnight
- Accompany samples with proper documentation (e.g., chain-of-custody forms)

- Ensure samples will arrive at the lab during business hours (e.g., Monday through Friday, not weekends or holidays) and within holding times specified in the project SAP
- Specify on chain-of-custody forms which shipping method was used (i.e., United States Postal Service, FedEx, UPS)
- Fill out shipping labels carefully and completely to minimize risk of loss
- Place custody seals over opening of cooler or shipping container and secure with tape
- Use package tracking to confirm delivery status
- Notify the lab to expect a delivery
- Ship samples according to packaging instructions provided by the laboratory
- Keep samples cold using bagged ice (in sealable zipper bags) and/or frozen icepacks; pack additional insulating material into open spaces in the cooler
- Comply with Department of Transportation Hazardous Materials Regulations (49 CFR Part 172)

NOTE: When using DEQ Site Visit Forms in lieu of laboratory-provided chain-of-custody forms, the lab keeps photocopies of site visit forms and DEQ retains the original forms. If samples are shipped, the original forms are shipped along with the samples to maintain chain-of-custody; in this case, Project Managers should retain a photocopy in case forms are lost and must request the lab return the originals with all necessary chain-of-custody signatures.

7.0 INTERFERENCES

This section describes components of the process that may interfere with the accuracy of final results.

7.1 ENVIRONMENTAL INTERFERENCES

Project SAPs contain specific guidance to address environmental interferences.

Natural Variability
Sampling designs should account for factors that influence natural variability of environmental samples such as climate, geology, landforms, soils, vegetation, moisture, solar radiation and hydrology.

Spatial Variability
Sampling designs should account for factors that influence spatial variability, including the number of sampling sites, their location, and the spacing between them. Sampling frames are often selected to represent larger portions of a system. Longitudinal or vertical profiles are often used to represent change throughout multiple dimensions.

Temporal Variability
Factors such as time of day, diurnal fluctuations, seasonality, and the amount of time between sampling events are factors to consider when accounting for temporal variability of environmental samples.

7.2 SAMPLING LOCATIONS AND SAMPLING FRAMES

Each sampling site needs to be selected and sampled in a manner that minimizes bias caused by the collection process and that best represents the intended environmental conditions at the time of

sampling (USGS, 2006). Samples are often collected at multiple locations and at multiple depths to capture variability in physical and chemical properties.

Sampling site locations are selected based on a project's monitoring objectives. Latitude and longitude coordinates of proposed sites, as well as the rationale for site selection, are specified in each project's SAP. For example, a SAP may describe stream sites near the headwaters, mouth, stream gage station, or tributary confluence, or perhaps lake sites near the deepest point, inlet or outlet of the lake. Proposed sites are often subject to change pending landowner permission, access, safety or other site-specific considerations. Always consult with the project manager before changing site locations to ensure that alternative sites align with project objectives. Landowner permission to access sites must be confirmed prior to any data collection on private lands.

In addition to latitude and longitude coordinates, project SAPs may include guidance for determining exact location(s) within a site where to physically collect samples for chemistry analysis. For example, SAPs may specify:

- Depth(s) where samples will be collected
- Allowable distance from the site coordinates
- Preferred locations in the water column
- Sampling frames, grids, transects or profiles
- Whether samples are composited and, if so, minimum number of sub-samples per composite

Upon arrival at each site, field personnel should consult the SAP for guidance and use site-specific considerations when selecting exact locations to collect samples. The following considerations may help guide this selection, though considerations may differ for flowing-water sites (i.e., streams and rivers, canals, ditches, and flumes, or to any other surface feature in which water moves unidirectionally) and still-water sites (i.e., all sizes and shapes of lakes, reservoirs, ponds, swamps, marshes, riverine backwaters, lagoons or any other body of surface water where water generally does not move unidirectionally) (USGS, 2006):

- Near the latitude/longitude coordinates of the proposed site in the project SAP
- In the main channel (i.e., the channel with a majority of flow) if channel is braided or split
- In a straight reach where water column is well-mixed with laminar, unidirectional flow (near the thalweg, and avoid stagnant pools, eddies, high turbulence, backwater, side channels, tributary inflows, etc.)
- In a place where field personnel can safely access, wade, and stand
- Upstream from recent disturbances to the substrate or water column
- Upstream from bridges, other structures, roads, crossings, or other on-site disturbances
- Where the water is sufficiently deep so water sample bottles can be fully submerged below the water surface and so the mouth of the bottle is elevated away from the bottom substrate
- Where chemical characteristics appear most likely to represent true/typical environmental conditions
- If lake is stratified, consider collecting samples from both the epilimnion and hypolimnion (**Section 10.2.7**)
- Where samples can be collected consistently throughout the period of the study regardless of discharge, stage, or other conditions

- Avoid mixing zones of tributaries or point source discharges, structures (e.g., bridges, harbors, boat ramps, piers, fuel docks), etc., unless the SAP specifically targets these areas.

8.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

Training
All field personnel conducting sampling must be familiar with proper sampling techniques, sample handling, safety procedures, and record keeping. New staff and student interns will be provided written SOPs and in-person training at the start of their employment and must be trained and accompanied in the field by experienced staff until competence is demonstrated and verified.

Responsibilities and Field Preparations
Project managers are responsible for:

- Developing SAPs and ensuring they are approved prior to the first sampling event
- Distributing SAPs and SOPs to field personnel
- Ensuring that analytical requirements are communicated to the lab(s)
- Ensuring site access permission is granted
- Ensuring field personnel have received training and demonstrate proficiency in all sampling protocols employed during the project
- Providing detailed instructions for individual sampling trips and reviewing them with field personnel, including sufficiently detailed site access instructions, landowner contact information, sites to be visited, allowable deviations from sampling plans, etc.
- Communicating to ensure samples are delivered within holding times
- Updating site lists, landowner contact records, and/or sampling plans based on feedback received from field personnel prior to subsequent sampling events

Field personnel are responsible for:

- Their safety
- The quality of the work performed
- Familiarity with study objectives and the purpose for each type of data being collected
- Understanding the system that each sample is striving to represent
- Packing necessary equipment and supplies
- Following sampling and decontamination protocols
- Completing all required documentation and field forms
- Following site access instructions and not trespassing
- Sampling equipment and supplies, including calibration, pre-field checks, use and maintenance
- Sample handling and storage
- Communicating to project manager (or designated appointee) regarding sample delivery
- Communicating with project manager and supervisor regarding evening safety check-ins and completion of sampling event
- Reporting vehicle maintenance issues

- Collecting the required number of quality control samples (blanks and duplicates)

9.0 EQUIPMENT AND SUPPLIES

NOTE: This section contains a general list of equipment and supplies associated with each procedure; additional items may be needed depending on project- and site-specific plans.

General

- Site Visit Forms
- Site Visit Codes
- Chain-of-custody forms (if separate from Site Visit Forms)
- Sample labels
- Clear tape
- Clipboard
- Pencil
- Permanent fine line marker
- Digital camera with spare batteries and/or battery charger
- GPS unit with spare batteries
- DI water for blanks
- Bubble wrap & sleeves or tape for protecting sample bottles in cooler (if glass)
- Sample bottle(s)
- Sample cooler(s)
- Ice (regular or dry depending on analysis)

Unfiltered Grab Samples

- Sample bottle(s)

Filtered Grab Samples

- Sample bottle(s)
- 60-cc syringe(s)
- 0.45 µm filter(s)

Clean Hands/Dirty Hands

- Sample bottle pre-preserved with HCl and double-bagged
- Sample cooler
- Regular ice
- Gallon-size resealable bags for bagging ice in cooler
- Two plastic garbage bags for lining cooler
- Two small cooler totes – one each for Dirty and clean hands kits
- Latex gloves in sealed bag (dirty hands)
- Latex gloves in sealed bag (clean hands)
- Forearm-length rubber gloves in sealed bag (clean hands)
- Paper towels/napkins in sealed bag
- Sample label
- Clear tape strips

Extension (Telescopic) Pole Sampler

- Sample bottle(s)
- Telescopic extension pole with clasp
- Reusable collection bottle
- Decontamination supplies

Depth-Integrated DH-48 Sampler

- Sample bottle(s)
- DH-48 sampler

Van Dorn or Kemmerer Water Sampler

- Sample bottle(s)
- Van Dorn or Kemmerer water sampler
- Messenger weight
- Rope or cable
- Secure knot or clips to connect ponar to rope
- Gloves for handling rope during ponar deployment/retrieval
- Winch (optional)
- Pulley boom (optional)
- Depth-finder (optional)
- Weights with strap to attach to water sampler
- Cable with depths marked, often in 0.1 or 0.5-meter increments

Two-meter Integrated Sampler

- Sample bottle(s)
- Two-meter integrated Sampler with a stopper for each end

Buchner Funnel

- Sample bottle(s)
- Buchner funnel (2 parts)
- Teflon mesh (fine, 60-micron)
- Plastic spoon
- Turkey baster

Ekman or Ponar Sediment Sampler

- Sample bottle(s)
- Ekman or ponar grab sampler with pin
- Rope or cable
- Secure knot or clips to connect ponar to rope
- Gloves for handling rope during ponar deployment/retrieval
- Bucket for spooling rope into while raising/lowering
- Winch (optional)
- Pulley boom (optional)
- Broad-bottomed shallow pan to put the grab sampler into once retrieved (stainless steel if organics)
- Spoons for scooping sediment out of instrument (stainless steel if organics)
- Bowl for compositing (stainless steel if organics)

- Sample cooler
- Ice (regular or dry depending on analysis)
- Decontamination supplies

Sediment Corer

- Sample bottle(s)
- Sediment corer
- Rope or cable
- Secure knot or clips to connect corer to rope
- Gloves for handling rope during ponar deployment/retrieval
- Bucket for spooling rope into while raising/lowering
- Winch (optional)
- Pulley boom (optional)
- Broad-bottomed shallow pan to put the grab sampler into once retrieved (stainless steel if organics)
- Spoons for scooping sediment out of instrument (stainless steel if organics)
- Bowl for compositing (stainless steel if organics)
- Sample cooler
- Ice (regular or dry depending on analysis)
- Decontamination supplies

Macroinvertebrate Tissue

- Sample bottle(s)
- D-frame dip net(s) & handle(s) (benthic sampling, wadeable)
- Benthic dredge or grab sampler (benthic sampling, unwadeable)
- Neuston net (surface sampling)
- Shallow-bottomed white trays for viewing, sorting (plastic or metal)
- Small pail for temporarily holding extra-large macroinvertebrates or collecting rinse water
- 500 µm sieve
- Sample cooler(s)
- Ice (regular or dry depending on analysis)
- Forceps
- Battery-powered field balance (scale) with spare batteries
- Macroinvertebrate identification key/guide book

- **Decontamination**
- 5-gallon bucket with lid for garbage
- Tarp/liner for boat surface where decon will take place
- Tub to capture detergent rinse water while cleaning
- Scrub brush, toothbrushes and thin scrubbing pads for cleaning
- Phosphate-free Alconox or Liquinox Soap (1% solution)
- Squirt bottles for Liquinox solution
- Laboratory-grade DI water for rinsing (in 1 L HDPE bottles or larger carboys)
- Squirt bottle for DI water
- Acid, if inorganics (dilute (5%) nitric or hydrochloric)
- Squirt bottle for 5% acid

- Solvents, if organics (certified ACS HPLC grade)
- Squirt bottle for solvents
- Container for storing solvent wastewater (e.g., new, empty paint cans)
- Latex gloves for use during decontamination
- Paper towels
- Plastic tubs with lids for storing PCB equipment and Hg equipment during transport
- Material Safety Data Sheets for acids/solvents

10.0 PROCEDURAL STEPS

This section describes procedural steps for several water, sediment and biological tissue sample collection procedures.

10.1 ORDER OF OPERATIONS

Field personnel should be aware of how the order of operations of field activities may effect the quality of samples. Samples for chemistry analysis should be collected in an order that minimizes risk of disturbance to the surrounding water column and substrate:

- Collect samples in order of most sensitive to least sensitive to disturbance
- Collect samples away and upstream from recent disturbances
- Chemistry samples should be collected before other biological or physical procedures

10.2 WATER SAMPLE COLLECTION FOR CHEMISTRY ANALYSIS

Samples should be collected directly into the laboratory-supplied sample containers whenever possible.

10.2.1 Unfiltered Grab Samples

The following protocol is used to collect unfiltered grab samples:

1. Carry the sample bottle to a suitable sampling location (**Section 7.2**).
2. Triple-rinse the bottle and lid: face upstream into the direction of the flow (if flowing water), collect a small volume of water in the bottle, replace the lid, and shake gently. Discard the rinse water downstream from the sampling location. Repeat two more times to triple-rinse the bottle.

NOTE: Do not rinse bottles that have preservatives added to the sample bottle prior to sampling (e.g., sample bottles for *E. coli*, mercury, and dissolved organic carbon are often pre-preserved).

3. Collect the sample: face upstream, submerge the bottle so the mouth is below the water surface to prevent particulates floating on the water surface from entering the bottle and above the bottom to prevent substrate from entering, and fill. For lakes that are less than two meters deep, surface samples may be collected using a grab technique by submerging the bottle until the field personnel's elbow is at the water surface (Egge, *et al*., 2018).

NOTE: If the bottle is pre-preserved, quickly remove the bottle from the water column once filled to minimize loss of preservative.

4. Leave appropriate headspace:

a. For most samples, fill the bottle to the shoulder or line that denotes the target volume, leaving a small amount of head space (especially if space is needed for preservative).

b. If samples are to be frozen, leave sufficient head space to allow for sample expansion without damaging the bottle.

c. If samples require zero headspace such as volatile organic analysis (VOA) or ultra-low-level mercury (ULL-Hg), fill the container completely. Submerge the bottle or vial and, while submerged, open the lid, fill, and secure the lid. Confirm no head space or air bubbles remain once the cap is secured. If necessary, use the lid to add a small amount of water to the top of the bottle or vial to form a convex meniscus before resecuring the lid.

NOTE: Minimize risk of contamination by keeping sample bottles closed whenever possible and avoid touching the inside of the bottle lid or lip.

5. Add preservative (if applicable): put on gloves, carefully unscrew the lid, pour the entire contents of the preservative vial into the sample bottle, replace the lid, and gently invert the sample bottle three times to mix the preservative into the sample. Discard the empty preservative vial.
6. Securely tighten the lid.
7. Store samples per sample preservation and storage requirements until delivery to the analytical laboratory within required holding times (e.g., upright in a cooler on regular ice at ≤6°C or frozen on dry ice).

10.2.2 Filtered Grab Samples

The following protocol is used to collect filtered grab samples:

1. Carry the sample bottle, a 60-cc syringe (**Figure 1**), and at least two 0.45 µm filters (**Figure 2**) to a suitable sampling location (**Section 7.2**).
2. Triple rinse the syringe: open the package and remove the new 60-cc syringe and discard the packaging. Rinse the syringe by drawing ambient water into the syringe, gently shaking, and compressing the syringe to force the water out. Repeat this process three times to triple-rinse.

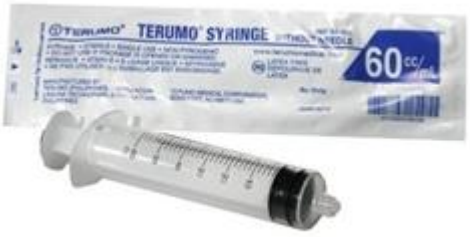

Figure 1: 60 cc syringe used to collect filtered water samples
Photo credit: EMS Professionals

3. Face upstream into the direction of the flow (if flowing water), submerge the tip of the syringe below the water surface and fill the syringe with ambient water upstream from any previous disturbance.
4. Apply and prepare the filter: open a new 0.45 µm filter package by gripping the outer ring and peeling the cover open. Without touching the filter, screw the filter onto the syringe and discard the packaging. Waste a small amount of water through the filter.

Figure 2: 0.45 µm disposable filters used to collect filtered water samples
Photo credit: Sigma Aldrich

NOTE: Avoid contaminating the filter before and during sample collection by not touching the filter anywhere besides the outer (blue) ring.

5. Triple-rinse the sample bottle with filtered water: plunge a small amount of water (approximately 10-20 ml) from the syringe through the filter into the sample bottle, replace the lid, and shake gently. Discard the rinse water downstream or away from the sampling location. Repeat this process three times to triple-rinse with filtered water. Discard the filter used for rinsing.
6. Collect the sample: refill the syringe with ambient water upstream from any previous disturbance, open and attach a new filter, and waste a small amount of water through the new filter. Fill the bottle with filtered water to the required volume.
    a. When the syringe is empty, carefully unscrew the filter touching only the outer ring. Refill the syringe, reattach the filter, and continue filling the sample bottle.
    b. If the filter clogs mid-way through filtering, unscrew and discard the clogged filter, refill the syringe, attach a new filter, waste a small amount of water through the new filter, and continue filtering. Repeat this process as needed until the sample bottle contains the required volume.

NOTE: Filters may break if excessive pressure is applied; do not apply excessive pressure on the syringe when filtering samples and replace the filter when it clogs rather than attempting to force water through.

NOTE: Consider performing this sample collection process at the water's edge to minimize risk of dropping, spilling or contaminating the sample bottle.

7. Leave appropriate headspace:
    a. For most samples, fill the bottle to the "shoulder" or line that denotes the target volume, leaving a small amount of head space (particularly if preservatives are to be added).
    b. If samples are to be frozen, leave sufficient head space to allow for sample expansion without damaging the bottle.
    c. For samples that require zero headspace such as volatile organic analysis (VOA) or ultra-low-level mercury (ULL-Hg), fill the bottle or vial completely and confirm no head space or air bubbles are visible once the cap is secured. Consider first submerging the sample container under water, opening the lid while submerged, filling, and securing the lid while still submerged. Or, use the lid to add a small amount of additional water to the top of the bottle or vial to form a convex meniscus prior to securing the lid.
8. Add preservative (if applicable): put on gloves, carefully unscrew the lid, pour the entire contents of the preservative vial into the sample bottle, replace the bottle lid, discard the empty preservative vial, and gently invert the sample bottle three times to mix the preservative into the sample.
9. Securely tighten the lid.

10. Store samples per sample preservation and storage requirements until delivery to the analytical laboratory within required holding times (e.g., upright in a cooler on regular ice at ≤6°C or frozen on dry ice).

NOTE: If filtering multiple samples at a single site, use a new, separate set of filters to fill each sample bottle to avoid cross-contamination, especially when filtering into pre-preserved bottles.

NOTE: It is highly preferable to filter in the field at the time of sampling. However, if the water is too turbid to filter in the field at the time of sampling (as indicated by a filter clogging nearly immediately), field personnel may collect water in a clean, acid-washed secondary container and allow the water to settle for a short time until it is able to pass through a filter. The secondary container must first be triple-rinsed with ambient water. Once the container has been rinsed and a grab sample has been collected, the lid should be secured and it should be stored upright in a cooler surrounded with ice. Filtering the final sample should happen as soon as possible and not more than 24 hours should pass between collection and filtering. Preservatives are added only after filtration.

10.2.3 Clean Hands/Dirty Hands Method

This procedure requires two people: one person is designated as “clean hands” and the second person is designated as “dirty hands.” “Clean hands” is responsible for all activities that involve direct contact with the sample bottle and water whereas “dirty hands” is responsible for all activities that do not involve direct contact with the sample bottle and water. This method is used to collect samples for ultra-low-level mercury analysis and other trace element analytes with parts-per-billion detection limits.

Minimize contamination risk:

- Procure fresh ice specifically for packing the sample cooler.
- Do not pack bags of ice for the sample cooler at gas stations or other industrial areas.
- “Clean hands” must not smoke cigarettes during a sampling day as cigarette smoke can contain mercury and other heavy metals.
- Use hand wipes regularly to clean hands and surfaces such as the vehicle steering wheel.
- Store empty sample bottles double-bagged, closed, and in a box or cooler until use.
- Store extra sampling supplies (e.g., paper towels, glove sets) in clean bags in a box or cooler.
- Whenever possible, approach the sampling site from downwind and downstream to reduce the risk of contamination.
- Periodically clean sample kits and discard used disposable supplies.

Prepare the sample cooler with ice:

1. Put on gloves.
2. Place a large, clean garbage bag in the cooler to line the cooler.
3. Pack ice into sealable gallon-size plastic bags and place the bags of ice inside the garbage bag inside the cooler. Line the bottom and the sides of the cooler with bags of ice to form a “nest” in which sample bottles will be cradled and kept cold; approximately 6-8 bags are needed for an average size cooler.

4. Place a second large, clean garbage bag inside the first garbage bag; this inner bag provides an additional barrier between sample bottles and bags of ice, and is the bag in which completed samples are placed.
5. Close both inner and outer garbage bags and close the cooler.

Prepare "clean hands" and "dirty hands" sample kits:

- Clean hands kit: Sample bottle (double-bagged and pre-preserved with hydrochloric acid (HCl) or other appropriate preservative), clean hands glove kits (sealed plastic bag containing one pair latex gloves and one pair forearm-length rubber gloves)
- Dirty hands kit: Dirty hand glove kits (sealed plastic bag containing one pair latex gloves), sample label already filled out, clear tape for affixing label to bottle, clean paper towels or napkins in a bag

Prepare to collect the sample and put on gloves:

6. Both people find an appropriate sample location (**Section 7.2**) along the water's edge and place the sample kits in a stable and accessible location.
7. "Clean hands" enters the water and stands near the water's edge in an area that allows for stable footing.
8. "Dirty hands" opens the "dirty" sample kit, removes the "dirty hands" latex gloves from the bag and puts them on. "Dirty hands" opens the "clean" sample kit, removes the bag containing the two sets of "clean hands" gloves, opens the outer sealed plastic bag containing the gloves without touching the inner bag, and holds the outer bag open within reach of the "clean hands" person.
9. "Clean hands" reaches into the outer bag, opens the inner bag containing the latex gloves, and puts the latex gloves on their hands.
10. "Clean hands" reaches again into the outer bag, opens the inner bag containing the long [yellow] rubber gloves, and puts this second pair of gloves over the first pair on their hands.

NOTE: Once "clean hands" is gloved, they must not touch anything except the inner bag containing the sample bottle, the sample bottle and cap, ambient (stream or lake) water, paper towels, the sample label and tape strips. If "clean hands" mistakenly touches any surface such as waders, streambanks, boats, vegetation, skin, etc., they should immediately remove the outer pair of gloves and continue the sampling procedure wearing just the inner pair of gloves.

Collect the sample:

11. "Dirty hands" opens the "clean" sample kit, removes the bag containing the double-bagged, pre-preserved sample bottle, opens the outer sealed plastic bag without touching the inner bag, and holds the outer bag open within reach of the "clean hands" person.
12. "Clean hands" reaches into the outer bag, opens the inner bag containing the bottle, and pulls the sample bottle out.

NOTE: The bottle is glass so extra care must be taken not to break it.

13. "Dirty hands" reseals the outer bag and returns it to the "clean" sample kit for later use.
14. "Clean hands" carries the bottle to an appropriate sample location (**Section 7.2**). If the water is not flowing, wade carefully to avoid disturbing the bottom sediments and water column. Facing upstream (if flowing), submerge the bottle completely beneath the water surface, taking care not to disturb the substrate. Once the bottle is completely submerged, "clean hands" unscrews the cap underwater and allows the bottle to fill with water. While the bottle is filling, the bottle and cap must remain fully submerged. Once full, secure the cap onto bottle while still submerged.

NOTE: Do not rinse the bottle as it is pre-preserved and also should not be exposed to the atmosphere.

NOTE: Fill the bottle and secure the cap as quickly as possible to minimize loss of preservative.

HINT: To ensure zero headspace, once the bottle is nearly full, quickly tip the bottle back toward you so it is completely upright to release the remaining bubble that often gets trapped at the bottle's shoulder.

15. "Clean hands" removes the full bottle from the water column and tips the bottle upside down to confirm there is no air trapped inside (i.e., zero headspace). ***There must be zero headspace in the bottle.*** If air is trapped, re-submerge and quickly re-open the bottle to release the air and fill completely with water, then replace the cap and remove the full bottle from the water column and verify again that there is zero headspace.

NOTE: If water depth is insufficient to fully submerge the bottle, a clean beaker that has been rinsed with dilute hydrochloric acid (HCl) then triple rinsed with deionized water may be used to fill the bottle; a note should be made on the site visit form indicating this modification was used.

Dry and label the bottle:

16. "Dirty hands" opens the "dirty" sample kit, removes the sealed bag of paper towels/napkins, opens it and extends it within reach of the "clean hands" person.
17. "Clean hands" reaches into the bag, removes a paper towel or napkin, and uses it to dry the bottle.
18. "Dirty hands" opens the "dirty" sample kit, removes the sample label, removes the label backing, and hands the label to "clean hands"; "clean hands" affixes the label to the bottle.
19. "Dirty hands" opens the "dirty" sample kit, removes a tape strip, removes the backing from the tape strip, and hands it to the "clean hands" person; "clean hands" affixes the tape over the sample label.

Store and transport the sample:

20. "Dirty hands" opens the "clean" sample kit, removes the empty double-bag that initially contained the sample bottle, opens the outer bag without touching the inner bag, and extends it within reach of the "clean hands" person. "Clean hands" reaches into the outer bag, opens the inner bag, and carefully places the sample bottle into the inner bag. "Clean hands" reseals the inner bag. "Dirty hands" reseals the outer bag.

HINT: By working together, both people can assist with squeezing the excess air out of the bags containing the sample bottle before they are sealed, as long as "clean hands" touches only the inner bag and "dirty hands" touches only the outer bag. Minimizing air in the bags will help ensure that the bottles can be as close as possible to the ice in the cooler despite everything being double-bagged.

21. "Dirty hands" places the double-bagged, full sample bottle into the "clean" sample kit for temporary storage and transport to the ice cooler.
22. Gloves may now be removed.
23. Upon returning to the ice cooler, one person should put on a clean pair of gloves, open the sample cooler, open the inner garbage bag, remove the double-bagged sample bottle from the "clean" sample kit, and carefully place the double-bagged sample bottle inside the inner garbage bag; attempt to place the sample bottle such that it is near ice so it stays sufficiently cold during storage.

NOTE: Samples bottles should remain double-bagged and inside the inner garbage bag until they are delivered to the analytical laboratory. If samples must be unloaded from the ice cooler, for example, into a refrigerator for storage, the person unloading the samples should put on at least one pair of clean gloves, lift the entire inner garbage bag out of the ice cooler, tie the bag closed, and gently place the entire bag of samples into the refrigerator without breaking them.

NOTE: The ice supply in the cooler should be monitored closely to keep samples sufficiently cold. It may be necessary to replenish ice in the cooler. To do this, one person should put on a pair of clean gloves, carefully remove the entire inner garbage bag full of double-bagged sample and place it temporarily in a clean, safe place to avoid breaking or contaminating samples. Remove each sealed bag of ice, pour out water, replace with clean ice, reseal, and replace bags of ice into the outer garbage bag lining the ice cooler. Once all sealed ice bags have been replenished, carefully replace the inner garbage bag containing sample bottles and organize as needed to ensure sample bottles are upright and in contact with bagged ice so they stay sufficiently cold.

NOTE: An entirely new set of gloves and other supplies should be used for each routine sample, field duplicate, and field blank.

10.2.4 Composite Samples

Project SAPs will specify whether samples will be composited. For example, where a lake thermocline exists samples may be collected at multiple depths from the epilimnion and hypolimnion. Alternately, multiple samples may be collected from within a sampling grid or sampling frame. Sub-samples (replicates) may be submitted separately for analysis or may be composited, depending on study objectives.

10.2.5 Extension (Telescopic) Pole Sampler

Direct collection into the sample container being submitted to the laboratory for analysis is always preferred. However, if water samples must be collected from a distance because, for example, samples cannot be safely collected via wading, access to the water's edge is limited, or wading will cause excessive disturbance, an extension (telescopic) pole sampler may be used to collect samples (**Figure 3**).

A new, clean site- and analyte-specific collection bottle may be used with the extension pole sampler. Alternately, if the same collection bottle that is attached to the extension pole will be used at multiple site locations, it must be cleaned and decontaminated between uses using approved decontamination procedures (**Section 12.5;** McCarthy, 2014). Project SAPs should specify decontamination requirements.

1. Decontaminate the sample collection bottle (if necessary) (**Section 12.5**).
2. Attach the clean/decontaminated collection bottle to the telescopic rod by securing the ring clasp.
3. Identify a suitable sample location (**Section 7.2**).
4. Triple-rinse the collection bottle with ambient water: extend the pole into a well-mixed portion of the water column, submerge the bottle into the water column below the water surface, fill the bottle, remove from the water column, and shake gently to rinse. Discard rinse water downstream from the sampling location. Repeat this process two more times to triple-rinse the bottle.
5. Collect the water sample: face upstream into the direction of the flow (if flowing), extend the pole into a well-mixed portion of the water column so the mouth is below the water surface to prevent particulates floating on the water surface from entering the bottle and above the bottom to prevent substrate from entering the sample bottle, and fill the bottle.
6. Return the bottle to a stable location on the bank, shore or in the vessel, carefully unfasten the ring clasp and remove the collection bottle from the telescopic pole.
7. Transfer water from the collection bottle into laboratory-provided sample bottles: follow unfiltered grab samples procedure (**Section 10.2.1**) or filtered grab samples procedure (**Section 10.2.2**), as applicable, to rinse sample bottles and collect samples.

Figure 3: Extension (telescopic) pole sampler
Photo credits: Fondreist Environmental Products (left), Dynamic Aqua-Supply (right)

10.2.6 Depth-Integrated DH-48 Sampler

The DH-48 (**Figure 4**) is a lightweight hand-held depth-integrating sampler used for the collection of water samples in wadeable streams, especially those being analyzed for suspended sediment. The sampler is drawn through a vertical profile of the water column, often multiple times at evenly-spaced transects along a cross-section. The DH-48 sampler can be used in velocities that range from 1 to 9 ft/sec and to a depth of up to 9 feet. The DH-48 can sample to within 3.5 inches of the streambed; this un-sampled zone is due to the distance between the nozzle and the bottom of the sampler (FISP, date unknown).

NOTE: If the sample bottle is not pre-preserved, triple-rinse it first before using the DH-48 to collect the sample. Face upstream into the direction of the flow (if flowing water), collect a small volume of water in the bottle, replace the lid, and shake gently. Discard the rinse water downstream from the sampling location. Repeat two more times to triple-rinse the bottle.

1. Insert the sample bottle into the sampler head and turn the spring-tension bottle retainer at the rear of the sampler to hold the bottle in place and seal the opening against the neoprene gasket inside the sampler head.
2. Hold the DH-48 by the handle vertically and extended upstream as far away from the body as possible with the nozzle pointing upstream.
3. Using a constant transit rate (FISP, date unknown), lower the sampler through the water until it reaches the bottom. Use care to avoid breaking the sample bottle as it touches the bottom and to prevent stirring up bottom sediments that could bias the sample. Once the sampler touches the bottom, immediately reverse the direction and raise the sampler, using the same transit rate, until it clears the surface of the water.
4. Verify the desired volume is achieved, remove the sample bottle from the DH-48 and secure the lid.

NOTE: If the volume is insufficient, remove and empty the sample bottle, adjust the transit rate, and repeat the sample collection steps.

NOTE: DH-48 samplers may have metal or plastic components; select the material that is most suitable for the target analytes (e.g., a metal DH-48 should not be used when collecting trace metals).

Figure 4: DH-48 Integrated Grab Sampler

10.2.7 Stratified Lakes (Locating the Thermocline)

Cold water is denser than warm water and therefore sinks to the bottom, thereby causing many lakes to be stratified into three distinct layers (**Figure 5**):

1. Epilimnion (warmer top layer)
2. Thermocline (middle layer where rate of temperature change is the greatest)
3. Hypolimnion (colder bottom layer)

Due to stratification, chemical properties of water may vary among the layers. Therefore, projects that involve lake sampling often specify in the project SAP where along a vertical profile of the water column samples will be collected. For example, if a lake is not stratified it may be sufficient to sample just below the water surface only. However, in a stratified system, samples may be collected from both above the thermocline (epilimnion) (e.g., just below the water surface) and below the thermocline (hypolimnion) (e.g., 1 meter above the lakebed); samples from each layer may then be composited together or may be submitted separately to the lab depending on project objectives.

The thermocline is defined as the depth range where the rate of temperature change is greatest in a vertical temperature profile; this equates to at least a 1^{o}C drop with each 1-meter depth increase (Cole, 1983). Therefore, to determine if a lake is stratified:

1. Attach a field meter with a temperature probe to a cable/rope.
2. Mark depths on the cable in one meter or smaller increments.
3. Measure and record temperature just below the water surface.
4. Gradually lower the probe vertically down through the water column and measure and record temperature at least every one meter until reaching the lake bed; record a final temperature measurement just above the lake bed.
5. Identify the one meter depth increment where there is a decrease in temperature of at least 1^{o}C. This depth marks the thermocline.

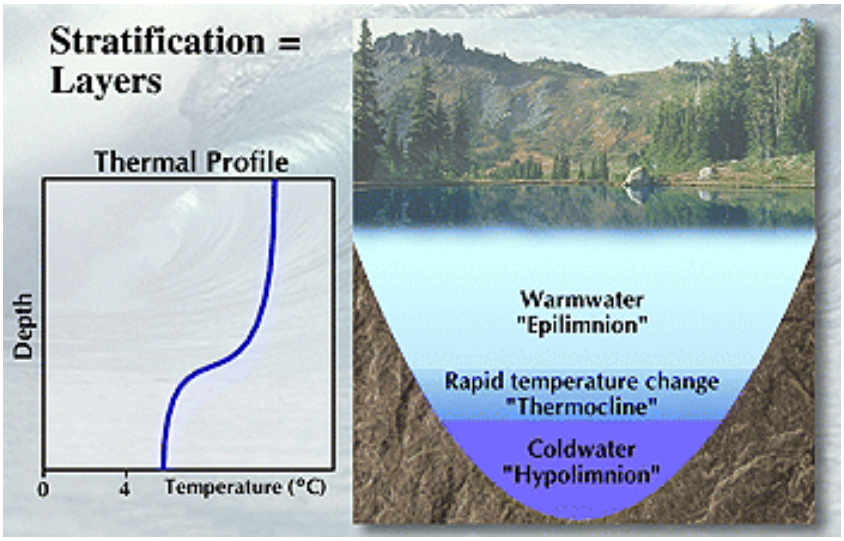

Figure 5: Thermocline and thermal profile of a lake
Photo credit: Hebert, 2002

10.2.8 Van Dorn Water Sampler

Van Dorn samplers (**Figure 6**) allow water to be collected from a known depth.

1. Determine desired depth(s) of sampling. Consult with sampling plan to determine whether samples will be composited.
2. Securely attach the Van Dorn to a sturdy line (e.g., rope, chain, cable); use of a winch is optional.
3. Open the Van Dorn: each end of the cylinder is fitted with a rubber cover; pull the rubber end seals out and back to open, and secure in place using the cables.

NOTE: Consider adding weights around the exterior of the Van Dorn to assist in lowering the instrument.

NOTE: Consider attaching a cable to the instrument to track depths while lowering.

4. Attach the metal weight called a “messenger” to the line.
5. Hold the messenger above the water surface and lower the Van Dorn to the desired depth by hand, with a winch, with a pulley boom, etc.
6. Once the desired depth is reached, hold in place and drop the “messenger” down the line to trigger the rubber end seals to release and snap shut. Raise the Van Dorn to the surface.
7. Use the drain valves to release water from the Van Dorn to perform rinsing and sample collection; follow the unfiltered grab samples procedure (**Section 10.2.1**) or filtered grab samples procedure (**Section 10.2.2**), as applicable.

Figure 6: Van Dorn Water Samplers
Photo credits: eco environmental (left), Dynamic Aqua-Supply (right)

10.2.9 Kemmerer Water Sampler

Kemmerer samplers (**Figure 7**) allow water to be collected from a known depth.

1. Determine desired depth(s) of sampling. Consult with sampling plan to determine whether samples will be composited.
2. Securely attach the Kemmerer to a sturdy line (e.g., rope, chain, cable); use of a winch is optional.
3. Open the Kemmerer: each end of the cylinder is fitted with a rubber cover; hold one side firmly and pull the other side to open until it clicks to remain open.

NOTE: Consider adding weights around the exterior of the Kemmerer to assist in lowering.

NOTE: Consider attaching a cable to the instrument to track depths while lowering.

4. Attach the metal weight called a “messenger” to the line.
5. Hold the messenger above the water surface and lower the Kemmerer to the desired depth by hand, with a winch, with a pulley boom, etc.
6. Once the desired depth is reached, hold in place and drop the “messenger” down the line to trigger the rubber end seal to snap shut. Raise the Kemmerer to the surface.
7. Use the drain valves to release water from the Kemmerer to perform rinsing and sample collection: follow the unfiltered grab samples procedure (**Section 10.2.1**) or filtered grab samples procedure (**Section 10.2.2**), as applicable.

Figure 7: Kemmerer Water Sampler
Photo credits: environmental equipment & supply (left), BioWeb (right)

10.2.10 Two-meter Integrated Sampler

For lakes that are two meters or more deep, surface samples may be collected using a two-meter integrated sampler. A 2-meter integrated sampler (**Figure 8**) enables the sampler to collect an integrated sample of the top two meters of the water column of a lake. It is simply a 2-meter PVC pipe with a rubber stopper on each end. It is helpful to have two people working together when using this instrument.

NOTE: Inspect the integrated sampler to ensure it is clean prior to use. Rinse the integrated sampler with tap water at the end of a sampling day, allow to dry completely, and store with the stoppers closed. To clean the sampler, mix one cup baking soda and one-gallon water in a plastic jug; plug one end, pour in half the solution, plug the other end, shake the sampler, and pour out the solution. Repeat with the second half of the solution, then rinse with tap water (RMB Environmental Laboratories, Inc., 2018).

1. Remove the stoppers at both ends to open the sampler and triple-rinse the integrated sampler on the opposite side of the boat from where the sample(s) will be collected.
2. Lower the integrated sampler straight down into the water column until the top is about 6 inches above the water surface (**Figure 8**).
3. Secure the top rubber stopper in place while the top 6 inches of the instrument is protruding out of the water.
4. Raise the integrated sampler straight upward slowly out of the water column; before pulling the sampler completely out of the water, while the bottom of the sampler is still submerged, replace the bottom stopper to prevent water loss.
5. Invert the tube slowly to mix the water three times; do this slowly so you can hear it slosh from end-to-end which is ensuring proper mixing of the water.
6. Place the end of the sampler over a sample collection container, remove the bottom stopper and pour out the entire contents. Samples may be collected directly into laboratory-provided sample bottles. Alternately, to facilitate triple-rinsing and/or filtering, water may be poured into an intermediate sample collection container (e.g., a clean 2-Liter bottle) from which the final sample is collected following unfiltered grab sample procedures (**Section 10.2.1**) or filtered grab sample procedures (**Section 10.2.2**).

NOTE: Remove the stopper from the bottom in an upward motion to avoid pouring water over the sampler's hands before it goes into the sample container which could contaminate the sample.

NOTE: Consider filling sample bottles over a bucket to contain spillage if working in a boat.

Figure 8: **Using a 2-meter Integrated Sampler**
Photo credit: RMB Environmental Laboratories, Inc., 2018

10.3 BENTHIC SEDIMENT SAMPLE COLLECTION FOR CHEMISTRY ANALYSIS

Benthic sediment samples may be collected from rivers, streams, lakes, or reservoirs for various chemical analyses. For example, benthic sediment samples are commonly analyzed for total recoverable metals as an indicator of the likelihood that heavy metals will become entrained in the water column during high flows. Benthic sediment is also often targeted when analyzing organic compounds such as PCBs. Chemical parameters such as redox potential (Eh) may also be measured in benthic sediment.

10.3.1 Benthic Sediment Sampling Strategy

The sample collection strategy for benthic sediment focuses on obtaining samples of fine-grained surficial sediments from natural depositional zones during low-flow conditions and on compositing samples from several depositional zones; this strategy is designed to yield a representative sample of fine-grained surficial bed sediments (USGS, 1994).

Depositional zones are locations where the energy regime is low and fine-grained particles accumulate. For example, depositional zones in streams/rivers are often found in shallow areas along the water's edge, along the inside of meander bends, and downstream of boulders, logjams, or other obstructions, and depositional zones in lakes are often found near tributary inflows or at the deepest point. Depositional zones can cover large areas at some sites and small pockets at other sites.

The goal when sampling benthic sediment in streams/rivers or lakes is to select multiple depositional zones that represent upstream influences and various flow regimes (e.g., left bank, right bank, center channel, and different depths of water). This will ensure that the sediment sample represents depositional patterns from various flow regimes and sources within the reach (USGS, 1994).

At each sampling site, it is recommended to collect sub-samples of benthic sediment from at least five depositional zones and to composite the sub-samples into a single sample unless otherwise noted in the project SAP. Generally, five sub-samples will adequately provide a physical average of surface sediment concentrations over a reasonable area and provide enough material for analysis (Washington DOE, 2003; Washington DOE, 2014; USGS, 1994; ORSANCO, 2002). Compositing will smooth the local scale variability and represent the average contaminant levels present at the site (USGS, 1994). Approximately equal volumes of sediment should be collected from each sub-sampled depositional zone.

Benthic sediment should be collected from the top 2-5 centimeters of the bed surface (USGS, 1994; ORSANCO, 2002; Washington DOE, 2003, 2007), representing the ongoing fish exposure medium (Washington DOE, 2003).

Benthic sediment samples should be collected during low flow/baseflow conditions to provide maximum direct access to the stream bed and to minimize seasonal streamflow variability (USGS, 1994). Unusually high flows can wash out, redistribute, or bury substantial parts of sediment deposits; therefore, sampling should be delayed following major discharge to allow fresh sediment to deposit.

10.3.2 Benthic Sediment Sampling Frames and Grids

Project SAPs may specify a sampling frame or sampling grid scheme to guide sediment collection. If a project-specific sediment frame or grid is not specified in a SAP, sampling should adhere to the following sampling frames for stream/river sites or sampling grids for lake sites:

Sampling Frames for Streams/River Sites

1. Sample depositional zones that are located within a distance upstream and downstream from the sampling site coordinates. Identify a sampling frame that meets these specifications:

- Is relatively homogenous.
- Encompasses approximately 50 meters up- and downstream from the sampling site specified in the SAP (i.e., ~100 m total around the site latitude/longitude coordinates). A longer sampling frame is acceptable if depositional zones cannot be easily located as long as access permits (USGS, 1994; USFWS, 2010).

2. Identify five depositional zones within the sampling frame. Include depositional zones along left bank, right bank and channel center if access allows; one bank is acceptable if other portions of the channel are unwadeable or otherwise not accessible.
3. Collect one benthic sediment grab sample from each depositional zone; be sure to grab submerged deposited benthic sediment, not bank material; composite these sub-samples into a single container to produce the final sample.

Sampling Grids for Lakes/Reservoir Sites

A systematic random grid sampling design may be used to guide lakebed sediment sample collection in depositional zones of interest; a grid is overlaid on a depositional area and plots within the grid are randomly selected for sampling (EPA, 2002; USFWS, 2014).

NOTE: This approach requires pre-field planning so maps and random number generators can be used.

1. Overlay a grid on the depositional zone so the center of the grid is positioned at the sampling site coordinates specified in the SAP. The size of the grid should be scaled to encompass all or most of the targeted depositional area and therefore some grids may be *larger* to accommodate large depositional areas from high order tributaries or the deepest portion of the lake, or *smaller* if tributaries are smaller or in narrow bays (**Figure 9**).
2. Divide the total area of the grid into 25 plots of equal area (**Figure 10**) and assign each plot a number (e.g., 1 through 25, left to right, top to bottom).
3. Use a random number generator to randomly select five plots to sample (**Figure 10**); exclude plots whose center falls entirely or mostly on land and are therefore not immersed during the sampling season. Include the latitude and longitude of each plot in the project SAP.

Figure 9: Example of small and large grid placement and plot selection

1	2	3	4	5
6	7	8	9	10
11	12	13	14	15
16	17	18	19	20
21	22	23	24	25

Figure 10: Example of numbering scheme and random number generation to identify grid sample plots

4. Navigate to the latitude and longitude of each of the five plots randomly-selected for sampling. Collect one benthic sediment grab sample (**Section 10.3.3**) from each plot and composite these sub-samples into a single container to produce the final sample.

10.3.3 Acquiring Benthic Sediment Samples with Grab Samplers (Ekman, Ponar)

Ekman and Ponar grab samplers (**Figure 11**) are used to collect benthic sediment samples from unwadeable zones or extreme depths such as in lakes, reservoirs, or large rivers. The Ponar is heavier than the Ekman and bites more effectively into hard substrates, whereas the Ekman probably creates less of a shock wave and can penetrate farther into soft sediments (Howmiller, 1971).

At each sampling location, collect a surficial sediment sample using a standard stainless steel Ekman or Ponar clamshell-style grab sampler:

1. Navigate via boat to the sampling location and deploy an anchor or the anchor setting on a trolling motor. Check the GPS occasionally to ensure the boat remains reasonably near the designated sampling location given GPS accuracy, wind, waves, and currents.
2. Securely attach the rope to the Ekman or Ponar, following these recommendations:
    - Use braided nylon climbing rope on a spool with more-than-sufficient weight rating.
    - Use a carabiner to attach the instrument to the rope so it can be more readily attached and removed.
    - Use a boom with a rope pulley attached for smooth deployment and retrieval.
    - Use a Capstan winch to assist with retrieval.

Figure 11: Ekman (left) and Ponar (right) Samplers
Photo credits: Cole-Parmer (left), eco environmental (right)

3. Secure the jaws of the Ekman or Ponar open and fit the spring-loaded pin in place.
4. Steadily lower the Ekman or Ponar until it contacts the lake bed sediments to trigger jaw closure.

NOTE: Lower the Ekman or Ponar at a moderate and steady pace with enough speed to penetrate the benthic sediments. Avoid lowering and raising too fast to avoid losing fine sediment due to "blow-out" when the dredge strikes the bottom and "wash-out" during dredge retrieval (ORSANCO, 2002). To avoid premature release of the spring-loaded pin, especially when sampling at very deep depths, 1) avoid jerking motions, and 2) consider using a depth finder and slowly lowering the instrument until it is within approximately 30 meters from the bottom before releasing the rope for free-fall. Repeat as needed until an acceptable sample is collected.

5. Gradually raise the instrument, bring it into the boat, detach it from the rope, and place it on a stable surface in a broad, shallow pan; use a stainless steel pan if analyzing organics.
6. Open the instrument (e.g., remove the stainless-steel screens from the Ponar or pull the Ekman open) and inspect the sample to determine if it is acceptable.

NOTE: A grab sample from a Ponar or Ekman will be considered acceptable if it is not underfilled, overlaying water is present but is not overly turbid, the sediment surface appears intact, and the grab reached the desired sediment depth (Ohio EPA, 2001). The Ekman or Ponar should penetrate 6-10 centimeters. Sediment grab samples for toxics or metals analyses will often target the 0- to 10-centimeter depth considered to be the biologically active zone (Ohio EPA, 2001).

7. Once a grab is deemed acceptable, pour overlying water off the sample.
8. If necessary, record a brief description of the sample (e.g., color, texture, odor, organic matter content, presence of biota or foreign matter) on the Site Visit Form.
9. Collect a sub-sample from the grab sampler for inorganic analysis (**Sections 10.3.5**) or organic analysis (**Section 10.3.6**).

10.3.4 Acquiring Benthic Sediment Samples with Sediment Corers

Sediment corers are usually inexpensive sampling devices and can be as simple as homemade tubes of steel, plastic or glass (Ohio EPA, 2001). Sediment corers can collect samples at depth, can maintain a more representative vertical profile of the sediment stratigraphy, create less disturbance by shock waves and can collect more highly consolidated deposits (Ohio EPA, 2001). Gravity sediment corers are released at the water surface and allowed to free fall and penetrate the sediment under their own

weight (Ohio EPA, 2011). There are many types of corers so project SAPs should specify instrument-specific instructions and field personnel should reference instrument-specific user manuals, as needed.

1. Navigate via boat to the sampling location and deploy an anchor or the anchor setting on a trolling motor. Check the GPS occasionally to ensure the boat remains reasonably near the designated sampling location given GPS accuracy, wind, waves, and currents.
2. Securely attach the rope or cable to the corer, following these recommendations:
    - Use braided nylon climbing rope on a spool with more-than-sufficient weight rating.
    - Use a carabiner to attach the instrument to the rope so it can be more readily attached and removed.
    - Use a boom with a rope pulley attached for smooth deployment and retrieval.
    - Use a Capstan winch to assist with retrieval.

NOTE: Braided nylon climbing rope on a spool with more-than-sufficient weight rating is recommended.

3. Deploy the corer (e.g., allow the gravity corer to steadily free-fall so the weight of th instrument drives itself vertically down into the lake bed sediments. Some lake sediments are softer than others, it may take multiple attempts to collect the desired amount of sediment without completely immersing the instrument and going deeper into the substrate than the length of tube.
4. Raise the instrument gradually (by hand or winch), bring it to the boat, (before taking the instrument completely out of the water place a hand or cap at the open end of the tube to ensure contents remains in the tube), detach it from the rope/cable, and place it on a stable surface in a broad, shallow pan (stainless steel if testing for organics). Repeat as needed until an acceptable sample is collected.

10.3.5 Processing Benthic Sediment for Inorganic Analyses with Buchner Funnel

For trace elements such as mercury and other metals, benthic sediment samples should be sieved and the fine-grained silt-clay fraction smaller than 60 µm should be saved for analysis. Buchner funnels are used to sieve benthic sediment samples through fine (60-micron) mesh:

1. Prior to use, clean and decontaminate all equipment (i.e., both pieces of the Buchner funnel, the Teflon mesh, and the spoon and/or turkey baster) using approved decontamination procedures (**Section 12.5;** McCarthy, 2014), then rinse all equipment with ambient water at the site.
2. Secure the piece of 60-micron Teflon mesh between the two pieces of a Buchner funnel, ensuring there are no gaps between the funnel and the mesh (**Figure 12**).

NOTE: Inspect the mesh carefully before and during use; replace if torn or damaged.

3. Place the end of the funnel into the sample bottle/jar.
4. At each depositional zone (**Section 10.3.1**) selected for sub-sampling within the sampling frame or grid (**Section 10.3.2**), use a non-metallic spoon or turkey baster to scoop fine sediment onto the sieve in the funnel.
    - Scoop enough sediment so the sieve is completely covered.
    - Do not retain debris on the sediment surface.
    - When sampling stream/river sites, be sure to collect sediment from the bed, not the banks.

- When sampling stream/river sites, collect material at depths less than 0.5 m deep as a safety measure and to minimize loss (wash-out) of surficial fine sediments as the spoon is drawn up through the water column (USFWS, 2010).

5. Use the non-metallic spoon or turkey baster to add minimal amounts of ambient stream water to the sediment in the funnel. Stir the water and sediment in the funnel to form a slurry and allow the slurry to filter through the mesh into the bottle. Gently swirl or tap the funnel to assist water passage, if necessary.
6. After each sub-sample is passed through the funnel, disassemble the funnel, rinse all equipment with ambient water, then reassemble the funnel and mesh.
7. Repeat the sample collection steps above at each depositional zone selected for sampling. **Use the same sample bottle for each sub-sample to produce a single composite sample for the entire sampling frame or grid.**

NOTE: SAPs should specify the minimum volume required for lab analyses. Generally, only a small amount of the fine (silt-clay) sediment material is needed by the lab to conduct analyses, approximately one cubic centimeter of dry sediment material. Because the lab must dry the sample before analysis, field personnel should collect only the volume needed to yield adequate sediment and therefore should use water sparingly during sample collection. For example, a 2L wide-mouth sample bottle should only be filled two centimeters deep with filtered water/sediment. If fine sediment is sparse, it may be necessary to collect additional volume and allow the sample to settle and decant excess water out of the sample bottle within 24-hours of collection before submitting to the lab.

8. Secure the lid on the sample bottle, label the bottle, and store the sample upright in a cooler on ice at the required temperature (generally ≤ 6°C) until delivery to the analytical laboratory.
9. Record the sample on the Site Visit Form.

Figure 12: Buchner funnel
Photo credit: The Science Company

10.3.6 Processing Benthic Sediment for Organic Analyses with 2mm Seive

For organic contaminants (e.g., PCBs), benthic sediment samples should be sieved and the sand and silt-clay fraction smaller than 2.0 mm should be saved for analysis (USGS, 1994). Benthic sediment sampling for organic constituents is conducted using all stainless steel equipment:

1. Prior to use, clean and decontaminate all equipment (i.e., stainless steel bowls or pails, sieve, spoon and funnel) using approved decontamination procedures (**Section 12.5;** McCarthy, 2014), then rinse all equipment with ambient water at the site.
2. At each depositional zone (**Section 10.3.1**) selected for sub-sampling within the sampling frame or grid (**Section 10.3.2**), use a stainless steel spoon to scoop fine sediment into a stainless steel bowl:

- Do not retain debris on the sediment surface.
- When sampling stream/river sites, be sure to collect sediment from the bed, not the banks.
- When sampling stream/river sites, collect material at depths less than 0.5 m deep as a safety measure and to minimize loss (wash-out) of surficial fine sediments as the spoon is drawn up through the water column (USFWS, 2010).

3. After all sub-samples are collected in the stainless steel bowl, use a stainless steel spoon to homogenize the composited material by stirring the composite sample to a uniform consistency and color (ORSANCO, 2002; USEPA, 2003; Puget Sound Water Quality Action Team, 1997; Washington DOE, 2007, 2014).
4. Use a stainless steel spoon to scoop and pass the entire volume of the homogenized sample through the stainless steel sieve (U.S. standard #10) into a stainless steel bowl or pail (ORSANCO, 2002) to remove particles larger than 2mm. Use the stainless steel spoon to add and stir minimal additions of site native water into the sediments only if necessary to pass sediments through the sieve.
5. Use a stainless steel spoon and stainless steel funnel to transfer sieved sediments into a 1 liter glass jar with a Teflon lid liner (ORSANCO, 2002; Wash. Dept. of Ecology, 2007, 2014).

NOTE: SAPs should specify the minimum volume required for lab analyses.

6. Secure the lid on the sample jar, label the jar, and store the sample upright in a cooler on ice at the required temperature (generally ≤ 6°C) until delivery to the analytical laboratory.
7. Record the sample on the Site Visit Form.

10.4 BIOLOGICAL TISSUE SAMPLE COLLECTION FOR CHEMISTRY ANALYSIS

Biological tissue may be used to determine concentrations of substances (e.g., metals, minerals, chemical compounds) which bioaccumulate in living organisms in an environment such as mercury, PCBs, and selenium. This information may also be used to calculate trophic transfer factors used in ecosystem models to understand the transfer of a substance from suspended particulate to invertebrates and from invertebrates to fish.

10.4.1 Fish Tissue

Collection of tissue samples, including whole fish, fillet, and biopsy plugs, from fish populations in Montana waters by or for DEQ should adhere to the Fish Tissue Sampling Standard Operating Procedure (Mavencamp, 2015).

10.4.2 Macroinvertebrate Tissue

Benthic Invertebrates from Lotic Environments or Shallow Lentic Environments

This macroinvertebrate collection method is modeled after EPA's rapid bioassessment protocol (Barbour *et al.,* 1999), EPA's Environmental Monitoring and Assessment Protocol (EMAP) reach wide sampling technique (Peck, *et al.,* 2006) and DEQ's Sample Collection, Sorting, Taxonomic Identification, and Analysis of Benthic Macroinvertebrate Communities Standard Operating Procedure (DEQ, 2012) which were originally developed for collecting macroinvertebrates for taxonomic identification.

Macroinvertebrate samples are collected from all present habitat types in proportional representation within a sampling frame. The minimum required wet weight of benthic macroinvertebrates (grams) are

collected by performing a total of 20 jabs or kicks using a D-frame dip net, resulting in sampling approximately 3.1 m^2 of habitat (Barbour, *et al.,* 1999). Macroinvertebrates collected within a sampling frame are composited into a single sample, frozen, and submitted to the laboratory for tissue analysis.

Identify the macroinvertebrate sampling frame

1. Identify a sampling frame at the site that:
    - Is relatively homogenous
    - Is at least 100 meters upstream from any road or bridge crossing if possible
    - Has no major tributaries discharging to the stream
    - Encompasses a reach approximately 50 meters up- and downstream from the initial arrival site (~100 m total); a longer sampling frame is acceptable if access permits and is deemed useful to capture additional habitat types.
    - Includes macroinvertebrate habitats on both banks if access allows; if not, collection on one bank is acceptable

Identify habitat types and associated collection techniques

2. Determine the habitat types that exist within the sampling frame and the most appropriate methods for collecting macroinvertebrate samples in each (Barbour, *et al*., 1999):
    - Cobble (hard substrate): Prevalent in riffles (and runs) and often dominant in high-gradient, mountain streams. Sample shallow areas with coarse (mixed gravel, cobble or larger) substrates by holding the bottom of the dip net against the substrate and dislodging organisms by kicking the substrate for 0.5 m upstream of the net.
    - Snags and other woody debris: Snags and other woody debris that have been submerged for a relatively long period (not recent deadfall) provide excellent colonization habitat. Sample submerged woody debris by jabbing in medium-sized snag material (sticks and branches). The snag habitat may be kicked first to help dislodge organisms, but only after placing the net downstream of the snag. Accumulated woody material in pool areas are considered snag habitat. Large logs should be avoided because they are generally difficult to sample adequately.
    - Vegetated banks: When lower banks are submerged and have roots and emergent plants associated with them, they are sampled in a fashion similar to snags. Submerged areas of undercut banks are good habitats to sample. Sample banks with protruding roots and plants by jabbing into the habitat. Bank habitat can be kicked first to help dislodge organisms, but only after placing the net downstream.
    - Submerged macrophytes: Submerged macrophytes are seasonal in their occurrence and may not be a common feature of many streams, particularly those that are high gradient. Sample aquatic plants that are rooted on the bottom of the stream in deep water by drawing the net through the vegetation from the bottom to the surface of the water (maximum of 0.5 m each jab). In shallow water, sample by numbing or jabbing the net along the bottom in the rooted area, avoiding sediments where possible.
    - Sand (and other fine sediment): Usually the least productive macroinvertebrate habitat in streams, this habitat may be the most prevalent in some streams. Sample banks of unvegetated or soft soil by bumping the net along the surface of the substrate rather than dragging the net through soft substrates; this reduces the amount of debris in the sample.

Collect the macroinvertebrate tissue sample

3. Sample habitat types in proportion to their representation of surface area of the total macroinvertebrate habitat in the reach. For example, if cobble riffles comprise 50% of the habitat in the sampling frame, and snags comprise 20%, then 10 jabs should be taken in riffles, 4 jabs are taken in snags, and the remaining 6 jabs are taken in any remaining habitat types. Habitat types contributing less than 5% of the stable habitat in the sampling frame need not be sampled.
4. Begin sampling at the downstream end of the reach and proceed upstream. Collect 20 jabs or kick samples over the length of the reach:
    - A single jab consists of forcefully thrusting the net into a productive habitat for a linear distance of 0.5 meters.
    - A kick is a stationary sampling accomplished by positioning the net and disturbing the substrate within a 1 ft^2 area upstream of the net. First, position a 500 µm D-frame kick net within the representative habitat type and hold the net vertically upright with the base of the frame in contact with the substrate and the open portion of the net facing into the flow. Second, pick up substrate within a 1ft^2 area in front of the net that is golf ball size or larger and clean them off into the net. Third, disturb the substrate over the same 1 ft^2 area immediately in front of the net for 30 seconds; carefully avoid sweeping the substrate and organisms out of the path of flow in front of the net opening. If there is no flow, use your hands or feet to push material into the net. If there is no flow, and the sampling location is full of aquatic vegetation, sweep the net over the 1 ft^2 area while disturbing the substrate.

Transfer the macroinvertebrate tissue sample into sample container

5. Periodically (i.e., after every 3 jabs or kicks or more often) rinse and transfer the sample into a container for macroinvertebrate collection (Barbour, *et al*., 1999; Bautts, *et al*., 2005; Arcadis, 2012). Rinse and consolidate the collected material in the net by running clean stream water through the net two to three times. Empty contents of the net into a shallow-bottomed tray or 500 µm sieve. Remove large debris.
6. Use forceps to collect macroinvertebrates from the tray and place them into a 4oz. wide-mouth glass sample jar with a Teflon lid. Composite the jabs/kicks collected from multiple habitats within the sampling frame to obtain a single sample.
    - Rinse the hand-picked organisms with clean stream water to remove attached sediments.
    - Determine target organisms used for the tissue analysis in the field based on size and availability (i.e., larger and/or more common organisms are preferred, and multiple species may be combined in a sample to meet mass requirements as needed).
7. Weigh the sample with a battery-powered field balance until the minimum required weight is achieved.

NOTE: If the minimum wet weight of macroinvertebrate tissue is not achieved from the initial 20 jabs/kicks, perform additional jabs/kicks as needed (again in relative proportion to the habitat types available) until the minimum sample size has been achieved.

8. If necessary, sort the organisms according to QAPP or SAP instructions (e.g., identify to order or class, or terrestrial versus aquatic).
9. Once sorted, place organisms in a labeled jar, vial or whirl-pac bag inside another bag in a cooler on ice.

10. Freeze until delivery to the analytical laboratory.

Benthic Invertebrates from Deep Lentic Environments

Sampling is performed using a benthic dredge equipped with two auxiliary weights and ventilation holes in the top to minimize shock waves and a sampling area of one square foot, or Ponar or Ekman grab sampler (**Section 10.3.3**).

1. Attach the dredge to a hydraulic winch spooled with 1/4-inch aircraft cable and lower it to the bottom.
2. Record the number and location of dredges on the Site Visit Form.
3. Collect two dredges per location and empty the contents into a sample collection container. Use stainless steel if sampling for organic such as PCBs; otherwise a plastic tub is sufficient.
4. Take the tub to shore and wet-sieve the contents using a 500 µm sieve.
5. Transfer the material retained on the 500 µm sieve from each dredge into a 1-L wide mouth Nalgene bottle and place it on ice in a cooler for transport to a controlled laboratory environment.
6. If necessary, according to Project QAPP or SAP instructions, dump the contents onto a clean white tray and use forceps to pick all visible macroinvertebrates from the sample with the aid of a light microscope when necessary. If necessary, sort the organisms according to QAPP or SAP instructions (e.g., identify to order or class, or terrestrial versus aquatic).
7. Place organisms in a labeled jar, vial or whirl-pac bag inside another bag in a cooler on ice.
8. Freeze until delivery to the analytical laboratory.

Surface Invertebrates from Lentic Environments

Macroinvertebrates on the surface can be sampled using a neuston net (**Figure 13**) or similar device. A neuston net is a tapered net with a rectangular mouth (1.0 meter wide by 0.3 meter high) and a removable collection container at the tapered end of the net. Mesh must be at least 500 µm for macroinvertebrates unless specified otherwise in the project SAP.

Figure 13: Neuston net

Photo credit: Austin Baldwin, USGS

1. Tow the net so that it samples a 1 meter wide section of the lake surface for 600 meters starting at the lat long indicated by the net location. Conduct nearshore tows in a zig-zag pattern along shore and offshore tows in a straight line with the assistance of a global positioning system (GPS) unit. Conduct additional tows as needed to obtain the minimum wet weight of macroinvertebrates tissues necessary for laboratory analysis.

2. After each tow, remove all macroinvertebrates from the bucket and net and place them in 125 ml sample bottles on ice in a cooler for transport to a controlled laboratory environment.
3. Dump the contents onto a clean white tray and use forceps to pick all visible macroinvertebrates from the sample with the aid of a light microscope when necessary. If necessary, sort the organisms according to QAPP or SAP instructions (e.g., identify to order or class, or terrestrial versus aquatic).
4. Place organisms in a labeled jar, vial or whirl-pac bag inside another bag in a cooler on ice.
5. Freeze until delivery to the analytical laboratory.

Sample Preparation & Analysis

Project planning documents must specify the minimum required wet weight of macroinvertebrates needed to satisfy analytical requirements. This minimum required weight will depend on the laboratory performing the analysis, the required reporting limits, the style of reporting (wet weight versus dry weight), and the number and type of analyses requested. All samples must be placed in sterile sample containers (e.g., amber glass jars, vials, whirl-pacs). Sample jars must be labeled with Site Visit Code, Waterbody Name, Collection Date and Collector's Name. Lids should be secured tightly and stored upright in a dark place. During collection and short-term transport, samples must be maintained on ice at 0 to 4 ^{o}C, and as soon as possible must be stored frozen at ≤ -20 °C until they are received by the analytical laboratory.

Sample analysis procedures are outside the scope of this SOP. Analytical requirements must be specified in project QAPPs and SAPs.

Documentation

Record the sample on the Site Visit Form. On the Summary Form, describe pertinent information such as the habitat types encountered, the dominant species present in the sample, or the conditions of sampling (e.g., high flows, treacherous rocks, difficult access).

Decontamination of Field Equipment

After sampling is complete at a given site, rinse all equipment thoroughly including nets, dredges, trays and sieves using ambient stream water. Visually examine all equipment and clear of any remaining organisms or debris before sampling at another site.

11.0 DATA AND RECORDS MANAGEMENT

All hardcopy documentation of the data, such as completed Site Visit Forms and laboratory EDDs, are stored and archived by the Water Quality Planning Bureau. Data is reviewed, validated, and stored according to DEQ's Water Quality Planning Bureau quality assurance and data management systems for environmental data operations.

11.1 SITE DOCUMENTATION

All sites where samples are collected must be geo-located and documented on a Site Visit Form (**Appendix A**) with the following information:

- Site Visit Code
- Waterbody name

- Latitude and longitude (Datum NAD83)
- Waterbody description (if already established in MT-eWQX database)
- Assessment unit identification (AUID)

11.2 SAMPLE LABELING

Each sample container must be labeled with the following information:

- Site Visit Code
- Waterbody name
- Date collected
- Personnel who collected the sample
- Indication if sample was filtered or not filtered
- Preservation method(s)

Fill out labels using pencil or fine-point permanent marker.

Affix labels to the sample bottle and cover completely with clear plastic tape to protect the label from water damage.

11.3 FIELD FORMS AND CHAIN-OF-CUSTODY

All samples collected must be recorded on a Site Visit Form with the following information:

- Site Visit Code
- Analytes
- Indication of media (water, sediment, etc.)
- Indication of blank or duplicate
- Preservation method(s)

All samples submitted to an analytical laboratory must be entered onto a Site Visit Form/Chain-of-Custody Form. Whenever samples are relinquished from one person to another, chain-of-custody signatures must be completed to maintain a record of who possesses and is responsible for the samples at all times, from the time of collection by field personnel to the time samples are received at the analytical laboratory.

Chain-of-custody signatures must be accompanied by the date and time when samples were relinquished and received, and the sample delivery method used (e.g., delivery by hand, United States Postal Service, FedEx, UPS).

11.4 DATABASE COMPATIBILITY

All data collected by or for DEQ must be stored in the Montana EQuIS Water Quality Exchange (MT-eWQX) database. Data submitted to MT-eWQX is sent to the national Water Quality Portal (NWQMC, EPA and USGS, 2019), which fulfills the requirement of environmental monitoring projects funded by federal monies, such as 319 grants or 106 funds. Procedural and formatting requirements (DEQ, 2018) must be followed to submit Electronic Data Deliverables (EDDs) to MT-eWQX.

12.0 QUALITY ASSURANCE AND QUALITY CONTROL

The data quality assurance (QA) program for field sampling consists of three parts: (1) adherence to the SOP procedures for sample/data collection and periodic evaluation of sampling personnel, (2) consistent instrument calibration methods and schedules, and (3) the collection of a field quality control (QC) samples during each sampling event. Further data quality control and quality assurance procedures will be addressed thoroughly in each project's Quality Assurance Project Plan.

12.1 FIELD DUPLICATE SAMPLES

Field duplicate samples are multiple samples collected by the same person, at the same time and place, following the same method, and using the same equipment as was used to collect routine samples. Field duplicates help to test precision of samples and check that samplers are following approved protocols.

- Refer to the SAP for specific guidance regarding total number of duplicate samples required for a project (typically 10% of the total number of samples collected throughout a sampling season).
- Refer to the SAP or project manager for guidance regarding where and when field duplicate samples should be collected. Generally, duplicates are collected at randomly-selected sites.
- Collect one set of field duplicate samples per parameter being analyzed by the lab.
- Follow sample documentation and labeling protocols used during routine sampling (**Section 11**), though use a separate Site Visit Form and a unique Site Visit Code for duplicate samples to differentiate duplicate samples from routine, blank, or other samples collected during the sampling event at a site. On the Site Visit Form, indicate "field duplicate" by checking the box and recording the Site Visit Code of the routine samples they accompany in the space provided.
- Select a sampling location that allows for the collection of two sets of samples side-by-side upstream from any previous disturbances.
- Follow exactly the same procedures used in rinsing, collecting, preserving, handling, and storing routine samples for the duplicate samples so two identical samples have been collected at the same site.
- If filtering samples, use the same syringe used to filter routine samples to collect duplicates, but use a new set of filters.
- Submit duplicate samples to the laboratory at the same time routine samples are submitted.

12.2 FIELD BLANK SAMPLES

Field blanks are samples of analyte free water poured into a container in the field, preserved and delivered to the laboratory with field samples (EPA, 2009). The primary purpose of field blanks is to measure the magnitude of contaminant concentration that might have been introduced into the samples a result of sampling-related activities (USGS, 2006).

- Field blanks must be prepared using laboratory-grade distilled or deionized water.
- Field blank samples must be prepared while in the field.
- Prepare a set of field blank samples during each sampling event and for each parameter being analyzed by the lab.
- Field blank samples are often collected at the end of a sampling trip following completion of activities at the final sampling site.

- Use new syringes and filters when preparing filtered field blanks.
- Follow sample documentation and labeling protocols used during routine sampling (**Section 11**), though use a separate Site Visit Form and a unique Site Visit Code for field blank samples to differentiate them from routine, duplicate, or other samples collected during the sampling event at a site. On the Site Visit Form, indicate "field blank" by checking the box provided.
- Follow exactly the same procedures used in rinsing, collecting, preserving, handling, and storing routine samples for the field blank samples except use laboratory-grade distilled or deionized water rather than ambient stream water.
- Submit field blank samples to the laboratory at the same time as routine samples.

12.3 TRIP BLANK SAMPLES

Trip blank samples are clean samples of a matrix that are taken from the laboratory to the sampling site and transported back to the laboratory without having been exposed to sampling procedures (EPA, 2009). Trip blanks are typically only analyzed for volatile compounds, and results include shipping and laboratory sources of contamination (EPA, 2009).

- If needed, the laboratory will provide trip blanks samples in the actual sample containers.
- Do not open trip blanks at any time.
- If pre-bagged by the laboratory, leave in bag and affix label to the outside of the bag.
- Keep prepared trip blanks with the investigative samples throughout the sampling event (DNREC, date unknown)
- Generally, trip blanks are labelled and placed in the cooler with the other completed samples at the end of a sampling trip following the completion of activities at the final sampling site (i.e., at the same time as field blanks).
- Follow sample documentation and labeling protocols used during routine sampling (**Section 11**), though use a separate Site Visit Form and a unique Site Visit Code for trip blank samples to differentiate them from routine, duplicate, or other samples collected during the sampling event at a site. Trip blanks samples can go on the same Site Visit Form as field blank samples if both are being prepared at the same time. On the Site Visit Form, indicate "trip blank" by checking the box provided.
- Follow the same storage procedures for trip blanks as routine samples (e.g., place upright in a cooler surrounded by ice at < 6^{o}C).
- Submit trip samples to the laboratory at the same time as other routine, duplicate, and field blank samples.

12.4 RINSATE/EQUIPMENT BLANK SAMPLES

Rinsate/equipment blank samples are samples of analyte free water poured over or through decontaminated field sampling equipment prior to the collection of environmental samples. Their purpose is to assess the adequacy of the decontamination process (EPA, 2009).

- Flush laboratory-grade distilled or deionized water through/over decontaminated sampling equipment and fill sample containers (e.g., bottles, VOA vials) with the rinsate.

- Follow sample documentation and labeling protocols used during routine sampling (**Section 11**), though use a separate Site Visit Form and a unique Site Visit Code for field equipment blank samples to differentiate them from routine, duplicate, or other samples collected during the sampling event at a site. Equipment blank samples can go on the same Site Visit Form as field blank and/or trip blank samples if both are being prepared during the same site visit. On the Site Visit Form, indicate "field equipment blank" by checking the box provided.
- Follow the same storage procedures for trip blanks as routine samples (e.g., place upright in a cooler surrounded by ice at < 6°C).
- Submit trip samples to the laboratory at the same time as all other routine, duplicate, and field blank samples.

12.5 DECONTAMINATION OF FIELD EQUIPMENT

To avoid cross-contamination between samples, all collection equipment and supplies that come into contact with a sample prior to use will be decontaminated between uses following approved procedures (McCarthy, 2014). Site- and project-specific procedures will be cited or described in project SAPs.

The following is a brief overview of decontamination procedures:

a. Wearing disposable gloves, thoroughly rinse the equipment with deionized or distilled water over a wash basin.

b. If collecting inorganic constituents only, rinse nonmetallic components over the same wash basin using a stream of dilute acid solution. After the acid rinse is complete, triple-rinse the equipment using deionized water over the same wash basin.

c. If collecting organic constituents only, wash with phosphate-free detergent solution (1%; Alconox or Liquinox soap), then rinse thoroughly with tap water or deionized water until agitated rinse water produces no more suds (Wilde, 2004). Perform a tertiary rinse using certified ACS HPLC grade organic solvent. Perform a final rinse with analyte-free deionized or distilled water.

d. If decontaminated equipment is not to be used immediately, allow to air dry and wrap in aluminum foil or seal in a recloseable plastic bag until use.

Key considerations pertaining to decontamination include:

- The target analytes are an important factor in selecting the appropriate decontamination method and whether the samples are for inorganic (metals) or organic constituents will determine the appropriate method and acid or solvent used for decontamination. For metals, dilute (5%) nitric (HNO_3) or hydrochloric acid (HCL) is used; nitric acid may be used only if there are no nitrogen samples being collected, while hydrochloric acid should be used if nitrogen samples will be collected. For organics, organic solvents such as hexane, isopropyl alcohol, acetone, or methanol are used to decontaminate the equipment (McCarthy, 2014).
- Decontamination with solvents should always be performed in well-ventilated areas, preferably on an open deck of a vessel or outdoors if on land; organic solvents are flammable and possibly explosive.
- All washing and rinsing solutions produced during decontamination procedures should be captured in a container for proper disposal; some organic rinsates volatilize quickly and are difficult to capture, but field personnel should employ best efforts to capture as much as

possible (McCarthy, 2014). Discard rinse water when the solution is greater than 6.0 pH (Wilde, 2004).

- Components made of fluorocarbonpolymer plastic generally can withstand a solvent rinse; verify with the manufacturer before using an organic solvent on components constructed of any other plastic.
- Do not use organic solvents on equipment used to collect organic carbon samples (Wilde, 2004).
- When collecting sub-samples to produce a composite sample, a project may consider using a tiered approach to decontamination in which a less-thorough cleaning procedure is conducted before moving between sub-sampling locations within a site whereas a more thorough decontamination procedure is conducted before moving between different sampling sites. This detail should be specified in a project SAP.

13.0 REFERENCES

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APPENDIX A – SITE VISIT FORM

The following is an example Site Visit Form; DEQ modifies this form to meet project-specific needs.

Place Site Visit Label Here

Site Visit Form

Project ID:____________________

Date: ______________ **Time:** ____________ **Personnel:** ______________________________

Waterbody: ______________________________ Location: ______________________________

Station ID: ______________ HUC: ____________ County: ______________ AUID: ____________

Latitude: . Longitude: . Elevation: __________ ft m

Field Duplicate to __________ ☐ Field Blank ☐ Trip Blank ☐ Field Equipment Blank ☐

Samples Collected		**Sample ID**	**Sample Collection Information/Preservation**
Water	☐		GRAB EWI BACT
Analysis:			0.45µ Filtered | HNO_3 H_2SO_4 H_3PO_4 HCL | Ice Frozen
Analysis:			0.45µ Filtered | HNO_3 H_2SO_4 H_3PO_4 HCL | Ice Frozen
Analysis:			0.45µ Filtered | HNO_3 H_2SO_4 H_3PO_4 HCL | Ice Frozen
Analysis:			0.45µ Filtered | HNO_3 H_2SO_4 H_3PO_4 HCL | Ice Frozen
Analysis:			0.45µ Filtered | HNO_3 H_2SO_4 H_3PO_4 HCL | Ice Frozen
Analysis:			0.45µ Filtered | HNO_3 H_2SO_4 H_3PO_4 HCL | Ice Frozen
Analysis:			0.45µ Filtered | HNO_3 H_2SO_4 H_3PO_4 HCL | Ice Frozen
Analysis:			0.45µ Filtered | HNO_3 H_2SO_4 H_3PO_4 HCL | Ice Frozen
Sediment	☐		SED-1
Analysis:			Preserved: None Other:
Benthic Chl-a	☐		Sample Method: C=Core H=Hoop T=Template N=None
Composite at Lab ☐ AFDW ☐ Visual Est. <50 mg/m2 ☐			Sample Location: R=Right C=Center L=Left
Transect: A__-__ B__-__ C__-__ D__-__ E__-__			F__-__ G__-__ H__-__ I__-__ J__-__ K__-__
Phytoplankton Chl-a	☐		D1 Filtered:_________mL D2 Filtered:_________mL
Phytoplankton CNP	☐		CN Filtered:_________mL P Filtered:_________mL
Algae	☐		PERI-1-MOD PERI-1 OTHER:
Macroinvertebrates	☐		MAC-R-500 OTHER: # of Jars:

Field Measurements		Time: am pm	**Field Assessments**
Water Temp: °C °F		Air Temp: °C °F	Photos ☐ Aquatic Plant Visual Assessment ☐ SAM ☐
Bar. Pressure: mm/Hg		SC: uS/cm	Aquatic Plant Tracking ☐ Rosgen ☐ NRCS ☐
pH:	DO: mg/L	Turbidity: NTU	EMAP ☐ Total Discharge ☐ Channel X-Section ☐
Turbidity: Clear ☐ Slight ☐ Turbid ☐ Opaque ☐			Wetland ☐ Bacteria ☐ Other:
Flow: ft3/sec (Dry Bed ☐ Stranded Pools ☐)			Only Transect F ☐ Total Site Length__________m
Meter ☐ Meter-Auto ☐ Float ☐ Gage ☐ Visual Est. ☐			Transect Length______m Average Wetted Width______m

Data Loggers	
	Temperature ☐ YSI ☐ MiniDOT ☐ EC ☐ TruTrack ☐ AquaRod ☐ Weather Station ☐
	Deployed ☐ Cleaned/Checked ☐ Retrieved ☐

Chemistry Lab Information		
Lab Samples Submitted to:	Account #:	Term Contract Number:
Invoice Contact:		
Contact Name & Phone:		EDD ☒ Format: MT-eWQX Compatible
1) Relinquished By & Date/Time:	1) Shipped By: Hand ☐ FedEx/UPS ☐ USPS ☐	1) Received By & Date/Time:
2) Relinquished By & Date/Time:	2) Shipped By: Hand ☐ FedEx/UPS ☐ USPS ☐	2) Received By & Date/Time:

Lab Use Only - Delivery Temperature: Wet Ice_______°C Dry Ice_______°C

Rev. 3/9/2017

Place Site Visit Label Here

Site Visit Form Continued

Field Meter Calibration			
pH Meter:	Manufacturer & Model:		Date of Last Calibration:
	Comments:		
Multiparameter Meter:	Manufacturer & Model:		
	Date of SC Calibration:	DO calibrated at site visit ☐	
	Comments:		

Site Visit Comments
Data Logger Notes:
Time data logger removed:
Time data logger returned:
☐ Data logger cleaned
☐ Data logger downloaded
☐ Data logger re-launched
Photos:
#
#
#
#
#
#