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# Standard Operating Procedures for Water Quality Sampling

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Version 1.0 (*final draft*)

July 2018

Environment and Climate Change Canada

FWQM&S – Athabasca Arctic Watershed

## Summary of Revisions

Version	Date	Summary of Revisions
1.0	July 2018	<ul style="list-style-type: none"><li>• addressed reviewers comments, editorial changes and formatting.</li></ul>
Draft	November 2017	<ul style="list-style-type: none"><li>• draft for review</li></ul>

This Standard Operating Procedure may be cited as:

Environment and Climate Change Canada (ECCC). 2018. Standard Operating Procedures for Water Quality Sampling. ISBN XXX-X-XXX-XXXX-X. Environment and Climate Change Canada, Water Science and Technology, Freshwater Quality Monitoring and Surveillance, Athabasca Arctic Basin, Saskatoon, SK, 18p.

Cat. No.: xx

ISBN: xx

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# Acknowledgments

We thank all staff of ECCC's Freshwater Quality Monitoring and Surveillance team over many years for contributing to, developing and testing of many previous versions of the protocols within this document. Funding for the production was provided through the Joint Oil Sands Monitoring Program co-led by the Governments of Canada and Alberta.

## Acronyms

CH/DH	Clean hands/dirty hands
ECCE	Environment and Climate Change Canada
EDI	Equal-discharge increment
EWI	Equal-width increment
HDPE	High-density polyethylene
JOSM	Joint (Canada–Alberta) Oil Sands Monitoring
LDPE	Low-density polyethylene
LPFB	Laboratory-Preserved Field Blank
MSDS	Material Safety Data Sheets
OSH	Occupational safety and health
PAH	Polycyclic aromatic hydrocarbon
PETG	Polyethylene terephthalate copolyester
PPE	Personal protective equipment
QA	Quality assurance
QC	Quality control
SOP	Standard operating procedure
USGS	United States Geological Survey
VOC	Volatile organic compound
WHMIS	Workplace Hazardous Materials Information System
WQMS	Water quality monitoring and surveillance

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## 1.0 Introduction

The following standard operating procedures (SOPs) are for the field collection of water quality samples. These procedures are based on various manuals used both nationally and regionally within the Water Quality Branch of Environment and Climate Change Canada (ECCC) (Inland Waters Directorate [IWD] 1973; IWD 1983; Mottle and Crosley 2002; Mottle 1991; USGS variously dated).

The procedures described herein have been applied in routine water quality monitoring of lotic water bodies. Variation in water-body type (e.g., lakes), hydrology, or environmental conditions, as well as changes to program design, may require method adaptation. These procedures do not address study design, occupational safety and health (OSH), analytical requirements, or data management and quality assurance (QA)/quality control (QC) beyond those that may be achieved using these field methods. Consistent use of these procedures will ensure that the data generated are accurate, scientifically robust, and comparable between samplers, sampling events, and sampling sites.

## 2.0 General Considerations

The following considerations should be taken when implementing field activities to assure the integrity of water quality samples and their associated information.

- **Training:** Staff shall be trained in the relevant standard operating procedures (SOPs) for water quality sample collection. The sample collector is the one primarily responsible for the quality and integrity of the sample, up to the time that the sample is delivered to the analyzing laboratory.
- **Collect a representative sample:** Use appropriate methods and quality assurance (QA) measures to ensure that the field sites selected and the samples collected (discrete, depth-integrated, isokinetic, composite, etc.) accurately represent the environment intended for study and can fulfill data-quality objectives (USGS 2006).
- **Pre-trip preparation:**
  - Assign sample numbers and parameter schemas to the sample bottle sets.
  - Ensure an adequate supply of appropriately prepared (cleaned), purchased or laboratory-supplied bottles/containers/preservatives for field trips.
  - Sampling equipment – Understand the physical and chemical limitations of each piece of equipment. Test the equipment. Ensure that the equipment is cleaned according to the appropriate SOP.

- **Awareness of possible sources of contamination:** This will reduce the likelihood of contamination. If contamination is suspected, samplers should be prepared to resample. Spare clean sample bottles should be taken into the field as backup for unforeseen circumstances.
- **Collect a sufficient number of quality control (QC) samples:** This will ensure that data-quality objectives are met. Special logistical considerations may be necessary when dealing with these additional samples.
- **Logistics awareness:** Can the samples be collected, stored, and transported, while adhering to all prescribed holding times and storage temperatures? Will the sample containers retain their integrity every step of the way?
- **Record keeping:** Detailed notes shall be attached to the sample field sheet for future reference and for inclusion in sample initialization, data verification/validation, and data interpretation. These notes may include, but are not necessarily limited to, field observations and measurements, photographs, unusual occurrences, etc.

### 3.0 Equipment

The collection of water quality samples and field measurements can be accomplished using a variety of methods. The methods and equipment that will be used during sample collection will vary based on accessibility of the sampling location (e.g., river thalweg), the type of sample (e.g., discrete versus depth-integrated), the parameter of interest (e.g., a closed sampling system to isolate the water sample from contamination), the season (e.g., under ice versus open water), potential risk to the field crew (e.g., swift current), and the program design and objectives. The equipment types used in the collection of water quality samples are listed in Tables 1 and 2

**Table 1 – Water quality sampling equipment**

Sampling Equipment	Application
Extension Rod/Pole Sampler	May be used from the shore to collect a discrete water quality sample in streams with velocities and depths that prevent wading.
Weighted Sampling Iron (Stainless Steel)	Used to collect quasi-depth-integrated water quality samples from boats, float planes, or under ice.
Peristaltic Pump	An apparatus used to collect a sample at one point in the water column via weighted tubing that submerges it to a specific depth. It is also used for sampling groundwater wells.
Volatile Organic Compound Sampler	Used to collect non-aerated samples into 40 mL glass vials for volatile organic compound analysis. The volatile organic compound sampler can be submerged to a specific water depth.
US DH-95 Isokinetic Sampler, Including:	Used to collect an isokinetic, depth-integrated, discharge-weighted sample (United States Geological



1 L bottle and nozzles with calibrated openings Winch for boat deployment	Survey 4.1.1-A).
Sample Churn Splitter	The churn splitter is used for compositing and subsampling water quality samples that are to be analyzed for "total" inorganic constituents. For example, samples from several verticals in a stream cross-section, differing slightly from each other in chemical quality and sediment concentration, can be placed in the churn and be mixed into a relatively homogenous suspension. Theoretically, any subsample withdrawn from the churn should be equal in chemical quality and sediment concentration to any other subsample from the churn.

**Table 2 – Supporting equipment**

Supporting Equipment	Equipment Description	Application	Equipment Requirements
Multi-parameter Water-Quality Sonde (e.g., YSI® Sonde)	Multi-probe unit allowing for discrete or unattended sampling of a number of water-quality parameters including water temperature, specific conductance, pH, dissolved oxygen, and turbidity.	Measuring water conditions at the time of water-quality sampling.	YSI® ( <a href="http://www.ysi.com/index.php">http://www.ysi.com/index.php</a> ): Units must be calibrated and maintained in accordance with the YSI user's manual. Calibration of the unit should be completed, as a minimum, at the beginning of each field trip and every 5th day of unit use.
Velocity Meter (e.g., Sontek® Flowtracker)	A device that measures stream velocity and may calculate stream discharge using two- or three-dimensional currents. Typically used when wading.	Calculate velocity at a specific water depth at the deployment sites. Measurements may be taken at the time of water quality sampling.	Sontek® Flowtracker ( <a href="http://www.sontek.com/">http://www.sontek.com/</a> ): Follow the operating procedures detailed in the user's manual.
Hand-held weather meter (e.g., Kestrel)	A hand-held (pocket) weather meter allowing users to measure a number of environmental conditions, including wind speed and air temperature.	Measure weather conditions on site during the period of water quality sampling.	Kestrel ( <a href="http://kestrelmeters.com/">http://kestrelmeters.com/</a> ): Sensors are factory calibrated. Consult the instruction manual for maintenance requirements.
Teledyne® RiverRay Acoustic Doppler Current Profiler	Measures water currents with sound, using a sound-wave principle called the Doppler Effect.	Used for stream velocity and discharge measurements in open water and under ice in depths ranging from 0.6 m to 40 m, and flows of up to 5 m/s.	Teledyne® RD Instruments ( <a href="http://www.rdinstruments.com">http://www.rdinstruments.com</a> ): Follow the operating procedures detailed in the user's manual.

## 4.0 Methods

### 4.1 Preparation for Field Trips

Preparation for field trips is dependent on the study design, sampling methods, and parameters of interest. Comprehensive pre-trip planning and preparation are critical to ensure sample integrity and to support the acquisition of robust, quality-assured supporting information. Prior to each trip:

- Assign sample numbers (e.g., YYYYPN##0001).
- Gather and label the necessary bottles (Table 3). The bottle label shall include the sample number and analyses requested.
- Record sample numbers, anticipated station number, and analyses requested in a tracking document.
- In the case of a QA/QC sampling event, QC samples (i.e., field, travel, and bottle blanks; see Appendix F) shall be prepared prior to field departure, as indicated in Section 4.3, “Post-Sample Collection Considerations”.
- Prepare field sheets for the sites to be sampled and include the following information: project name and number, station/site name and number, sample number, collector/submitter, anticipated sampling dates, and any additions or corrections to the bottle type, parameter groups, or preservatives. See Appendix B for examples of project-specific field sheets.
- Prepare laboratory-specific sample submission sheets and include the following information: project name and number, sample number, and analysis requested. See Appendix C for examples of laboratory sample submission sheets.

**Table 3 – Bottle types and the associated analytical schema labels (subject to change)**

Parameter	Schema/Label (Laboratory Specific)	Bottle Type and Size (Single Use)
Nutrients	None on the bottle	2 L translucent LDPE
Ammonia (NH <sub>3</sub> )	None on the bottle	125 mL translucent HDPE
Major Ions	B Alkalinity Auto; B Conductivity Auto; B pH Auto; B Anions; B Cations; B SAR	500 mL translucent HDPE
Metals	B Metals OS Diss ICP-MS; B Metals OS TR ICP-MS	500 mL translucent HDPE
PAH	PAH2-W, ALK-PAHS-EX	1 L amber glass
Phenol	Phenol	1 L amber glass
Naphthenic Acids	Naphthenic acids	1 L amber glass
CCME-F1	CCME-F1	40 mL amber septum vials (2x)
CCME-F2, F3, F4	CCME-F2, F3, F4	1 L amber glass
Cyanide	None on the bottle	500 mL opaque amber HDPE
Total Hg	None on the bottle	125 mL glass (QC'd and

		supplied by the laboratory)
Methyl Hg	None on the bottle	250 mL PETG (QC'd and supplied by the laboratory)

LDPE = low-density polyethylene; HDPE = high-density polyethylene; PAH = polycyclic aromatic hydrocarbon; QC = quality control; PETG = polyethylene terephthalate copolyester.

- Pack the bottles required for each site in separate containers (e.g., coolers or large Ziploc bags) labelled with the site/station name. This allows easy access to the bottles as needed.
- Glass bottles should be packed very carefully. For example, the use of prefabricated Styrofoam inserts made to fit inside of a standard cooler, along with Styrofoam lining the bottom of the cooler, is very effective for protecting glass bottles during transport. Pack extra inert packing material (plastic bags, bubble pack, ice packs, etc.) separately. These materials will be used when shipping samples to the laboratory.
- Prepare and check any required preservatives. Check the expiry dates on each preservative bottle. If the preservative is expired or is suspected to be contaminated, discard and restock it. Restock fresh preservatives at the beginning of each year at a minimum. Place each preservative bottle in a sealable, overflow container.
- Check that any electronics are in good working order and that the batteries are charged; such equipment can include GPS, field computers, current meters, and cameras.
- Check the cleanliness and operation of each required water-quality-sampling device (Tables 1 and 2).
- Clean water-quality-sampling devices, as per the cleaning SOPs in Appendix D.
- Check, service, and calibrate (pH, conductivity, turbidity, and dissolved oxygen) the water quality multiprobe meter, as outlined in Table 2.
- Pre-plan for shipping to ensure sample integrity following collection (temperature and holding times).

## 4.2 Field Methods

### 4.2.1 Bottle Filling

Regardless of the manner in which the samples are collected, the bottles must be filled and the water must be treated as indicated in Table 4. This bottle-filling procedure maintains a relationship to historical Environment and Climate Change

Canada (ECCC) water quality sampling methods, and it also adheres to laboratory-prescribed procedures. These procedures are subject to change.

**Table 4 – Parameter-specific bottle filling and treatment**

Parameter	Bottle Rinse	Sampling	Preservative	Bottle Type
<b>Nutrients</b>	2x	Direct*	-	2 L translucent LDPE
<b>Ammonia</b>	-	Poured off from the nutrient bottle	1 mL 10% H <sub>2</sub> SO <sub>4</sub>	125 mL translucent HDPE
<b>Major Ions</b>	-	Poured off from the nutrient bottle	-	500 mL translucent HDPE
<b>Metals</b>	-	Poured off from the nutrient bottle	-	500 mL translucent HDPE
<b>PAHs</b>	-	Direct*	-	1 L amber glass
<b>Phenol</b>	-	Direct*	1 mL of 6N H <sub>2</sub> SO <sub>4</sub> (to pH 2)	1 L amber glass
<b>Naphthenic Acids</b>	2x	Direct*	-	1 L amber glass
<b>Cyanide</b>	3x	Poured off from the nutrient bottle	2 pellets NaOH	500 mL opaque amber HDPE
<b>CCME-F1 (BTEX)</b>	-	Direct*	5 drops of 6N H <sub>2</sub> SO <sub>4</sub> (to pH 2)	40 mL (2x) amber septum vials
<b>CCME-F2, F3, F4</b>	-	Direct*	-	1 L amber glass bottle
<b>Total Mercury</b>	2x	Direct*	-	125 mL glass (supplied by the laboratory and double bagged)
<b>Methyl Mercury</b>	2x	Direct*	Freeze as soon as possible (leave room in the bottle for expansion)	250 mL of PETG (supplied by the laboratory and bagged)

\*Direct – denotes sampling directly from water column.

LDPE = low-density polyethylene; HDPE = high-density polyethylene; PAH = polycyclic aromatic hydrocarbon; PETG = polyethylene terephthalate copolyester.

## 4.2.2 Sampling via Hand Dipping

### 4.2.2.1 Wading

If you are unfamiliar with the stream to be sampled, or if the stream bed is subject to change, then explore the stream bed for large obstacles or holes by wading carefully into the stream with the aid of a wading stick, safety line, and lifejacket. Once a safe location has been found, sampling can begin.

Whole-water samples will be collected midstream in a well-mixed zone at mid-water depth, or approximately 30 cm below the water's surface. Ensure that the water upstream has not been disturbed and that arm's length is outside the bow wave and/or stirred-up sediment created by standing in the stream. Use clean, long rubber or nitrile gloves.

Sampling will likely involve carrying bottles to and from the shore several times. If this is the case, move slightly upstream of the prior sampling location to ensure that the water being collected has not been disturbed. Follow the procedure outlined below to collect the samples. Facing upstream:

1. Remove the cap from the bottle, ensuring that only the exterior of the cap is touched.
2. Hold the bottle near its base and plunge its neck downwards below the surface to mid-depth; immediately turn the bottle until the neck points slightly upwards and into the current. Hold the bottle upstream at arm's length while it fills. Avoid sampling the surface film.
3. If required by the laboratory, rinse the bottle and cap (see Table 4) and repeat the filling procedure until all bottles have been filled.
  - a) Metals, major ions, ammonia, and cyanide samples get filled from the bulk 2 L nutrient bottle. Rinse the bulk 2 L bottle twice (Table 4) and fill, as per the filling procedures. Pour off the water from the bulk 2 L into each metal, major ion, ammonia, and cyanide bottle, one at a time, gently swirling the bulk 2 L between each pour to evenly distribute any sediments. Do not touch the mouths of the bottles together while pouring the samples. Dump the remaining water from the bulk 2 L and re-fill.
4. Rinse the inside of each lid with sample water from the sample bottle, cap the bottle immediately, and place the sample in a cooler.
5. If necessary, preserve as soon as possible (Table 4).

#### **4.2.2.2 From a Boat**

In some instances, there are bottles that are not suitable for direct sampling via a sampling iron, and the parameter may be unsuitable for being poured off from a different sampling bottle (e.g., in the collection of major ions, metals, ammonia, and cyanide samples, which are being poured off from the bulk 2 L nutrient bottle). For example, water collected for total and methyl mercury, as well as CCME-F1 (BTEX), must be sampled directly into the sample bottle. In the case of these samples, and in some exceptional circumstances, hand-dipping the bottles from the boat is required. Always sample from the upstream side of the boat to prevent gas and any boat-related contamination of the sample.

1. Put on clean nitrile gloves. If collecting a mercury sample, use the clean-hands (CH)/dirty-hands (DH) procedure outlined in Appendix E.
2. Remove the cap from the bottle, ensuring that only the exterior of the cap is touched. Set the cap upside down on a clean surface.
3. Safely working over the gunwale of the boat, hold the bottle near its base and reach as far as possible away from the side of the boat. Plunge the bottle neck downwards below the surface, to a depth of at least 10 cm; immediately turn the bottle until the neck points slightly upwards and into the current. Hold the bottle upstream while it fills. Avoid sampling the surface film.
4. If required by the laboratory, rinse the bottle and cap (see Table 4) and repeat the filling procedure until all bottles have been filled.
  - a) Metals, major ions, ammonia, and cyanide samples get filled from the bulk 2 L nutrient bottle. Rinse the bulk 2 L bottle twice (Table 4) and fill, as per the filling procedures. Pour off the water from the bulk 2 L into each metal, major ion, ammonia, and cyanide bottle, one at a time, gently swirling the bulk 2 L between each pour to evenly distribute any sediments. Do not touch the mouths of the bottles together while pouring the samples. Dump the remaining water from the bulk 2 L and re-fill.
5. Rinse the inside of each lid with sample water from the sample bottle, immediately cap the bottle, and place the sample in a cooler.
6. If necessary, preserve as soon as possible (Table 4).

#### **4.2.2.3 Under Ice**

In situations where the depth of the water under ice is not sufficient for using a weighted sampling iron or, as in Section 4.2.2.2, "From a Boat", when the parameters and bottles are not conducive to being collected via a sampling iron, a hand-dipped sample under ice can be collected. Water sample collection for

under-ice conditions may require access via a snowmobile to the site. Upon arrival at the sampling location, turn off the snowmobiles and allow the fumes to dissipate before beginning water-sample collection.

Once the sample location has been determined:

1. Shovel and clear any snow and loose ice away from the sampling location. Clear a large working space, so that nearby snow and dirt will not get pushed into the sampling hole. Keep this working area clean.
2. Use an electric ice auger (or hand auger) to drill the hole to prevent contamination, which can result from using a gasoline auger. Clear the hole of all slush and ice prior to collecting any water samples. Keep the drill hole clear as sampling proceeds.
3. Determine the depth of the water and the ice thickness.
4. If the bottom sediment was disturbed while auguring, do not proceed to collect water samples in this location. If the program requirements allow for minor location movement, try clearing a new hole a few metres upstream of the existing one (repeat steps 1 and 2). Auger slowly and be mindful of the shallow water depth in the area; try to avoid disturbing the bottom sediment with the auger. If the water depth below the ice is not sufficient to lower and raise a sampling iron, proceed as follows, while hand-dipping the bottle for collection.
5. Put on a long nitrile glove to protect the whole arm. If collecting a mercury sample, use the CH/DH procedure outlined in Appendix E.
6. Remove the cap from the bottle, ensuring that only the exterior of the cap is touched. Set the cap upside down on a clean surface.
7. Lay flat on the surface of the ice. Hold the bottle near its base and plunge its neck downwards as far as possible below the surface of the ice; once the bottle is situated below the ice and in flowing water, turn the bottle until the neck points slightly upwards and into the current. Hold it in place until the bottle has finished filling.
8. If required by the laboratory, rinse the bottle and cap (see Table 4) and repeat the filling procedure until all bottles have been filled.
  - a) Metals, major ions, ammonia, and cyanide samples get filled from the bulk 2 L nutrient bottle. Rinse the bulk 2 L bottle twice (Table 4) and fill, as per the filling procedures. Pour off the water from the bulk 2 L into each metal, major ion, ammonia, and cyanide bottle, one at a time, gently swirling the bulk 2 L between each pour to evenly distribute any sediments. Do not touch the mouths of the bottles together while

pouring the samples. Dump the remaining water from the bulk 2 L and re-fill.

9. Rinse the inside of each lid with sample water from the sample bottle; cap the bottle immediately and place the sample in a cooler.
10. If necessary, preserve as soon as possible (Table 4).
11. If the temperatures are well below freezing, precautions must be taken to prevent the samples from freezing and/or breaking during transport.

#### **4.2.3 Sampling with an Extension Rod**

Occasionally, a site that is typically sampled via wading is not wadable for a variety of reasons. Water levels and/or water velocities may be dangerously high or there may be dangerous ice conditions at the water's edge. Under such conditions, it may be necessary to collect samples using an extension rod to sample the main flow of the stream. When using a sampling rod:

1. Rinse the end of the rod in the sample water to remove potential contaminants.
2. If required by the laboratory, rinse the bottle and cap (Table 4). It is best to do this downstream of the filling location. Complete the required rinses and return the cap to the bottle.
3. Collect the samples one at a time, securing the bottle in the sampling extension. Un-cap the bottle directly prior to sampling and place the cap aside on a clean surface.
4. Collect the water samples facing upstream into the current to ensure that any contaminants introduced from the extension rod flow away from the sample bottle. Extend the sampling rod out to the sampling location and submerge the bottle below the stream's surface, sampling into the current. If the sampling area has been contaminated (i.e., stirred up streambed) move slightly upstream to collect the sample.
5. When the bottle is full, lift the extension rod out of the water and bring it back onto the shore.
6. If required by the laboratory, rinse the bottle and cap (see Table 4) and repeat the filling procedure until all bottles have been filled.
  - a) Metals, major ions, ammonia, and cyanide samples get filled from the bulk 2 L nutrient bottle. Rinse the bulk 2 L bottle twice (Table 4) and fill, as per the filling procedures. Pour off the water from the bulk 2 L into each metal, major ion, ammonia, and cyanide bottle, one at a time, gently swirling the bulk 2 L between each pour to evenly distribute any



sediments. Do not touch the mouths of the bottles together while pouring the samples. Dump the remaining water from the bulk 2 L and re-fill.

7. Rinse the inside of each lid with sample water from the sample bottle; cap the bottle immediately and place the sample in a cooler.
8. If necessary, preserve as soon as possible (Table 4).

#### **4.2.4 Sampling with a Weighted-Sampling Iron**

##### **4.2.4.1 From a Boat**

Upon arrival at the sampling location, and if it is safe to do so, anchor the boat and shut off the boat motor. Allow the fumes to dissipate before beginning water-sample collection. Keep aware of other boat traffic and natural hazards. Always sample from the upstream side of the boat to prevent boat-related contamination of the sample.

Determine the depth of the station using an appropriate depth-sounding device (wading rod, sounding line with weight, or acoustic sounding instrument). From the upstream side of the boat:

1. Place and secure a sample bottle in the sampling iron. Remove the cap from the bottle and set it aside on a clean surface.
2. Lower the sampling iron to the surface of the water. Ensure that the bottle does not come in contact with the boat, as this may cause dirt or other contaminants to fall into the bottle.
3. Lower the sampling iron into the river at a uniform rate until the sampling iron sinks as close to the bottom as possible. Raise the sampling iron at the same uniform rate. The objective of this method is to completely fill the bottle as it returns to the water's surface. It may take some practice to fine tune the transit rate for different bottle sizes and stream depths. By using this method, a quasi-depth-integrated sample may be obtained.
4. Do not allow the sampler to come in contact with the stream bed, as this may stir up bottom sediments, thus contaminating the water sample. Predetermine the water depth and measure out the length of rope required to sample the whole water column; this will help prevent contacting the stream bed when sampling. Measure an extra length of the sampler rope and tie it to the boat, so that the sampling iron cannot be inadvertently lost.
5. If required by the laboratory, rinse the bottle and cap (see Table 4) and repeat the filling procedure until all bottles have been filled.

- a) Metals, major ions, ammonia, and cyanide samples get filled from the bulk 2 L nutrient bottle. Rinse the bulk 2 L bottle twice (Table 4) and fill, as per the filling procedures. Pour off the water from the bulk 2 L into each metal, major ion, ammonia, and cyanide bottle, one at a time, gently swirling the bulk 2 L between each pour. Do not touch the mouths of the bottles together while pouring the samples. Dump the remaining water from the bulk 2 L and re-fill.
6. Rinse the inside of the lid with sample water from the bottle, if required (see Table 4); cap each bottle immediately after filling and place in a cooler.
7. If necessary, preserve as soon as possible (Table 4).

#### **4.2.4.2 Under Ice**

Water-sample collection for under-ice conditions may require access via a snowmobile to the site. Upon arrival at the sampling location, turn off the snowmobiles and allow the fumes to dissipate before beginning water-sample collection.

Once the sample location has been determined:

1. Shovel and clear any snow and loose ice away from the sampling location. Clear a large working space, so that nearby snow and dirt will not get pushed into the sampling hole. Keep this working area clean.
2. To prevent any oil, gas, or exhaust contamination, avoid using a gas-powered ice auger. Use an electric ice auger (or hand auger) to drill a hole through the ice. Using an ice-hole de-slusher, clear the hole of all slush and ice prior to collecting any water samples. Keep the drill-hole clear as sampling proceeds.
3. Determine the water depth and ice thickness.
  - a) If the bottom sediment was disturbed while augering, do not proceed with collecting water samples in this location. If the program requirements allow for minor relocation, try clearing a new hole a few metres upstream of the existing one (repeat steps 1 and 2). Auger slowly and be mindful of the shallow water depth in the area; try to avoid disturbing the bottom sediment with the auger. If the depth of the water below the ice is not sufficient for lowering and raising a sampling iron, proceed as follows by hand dipping the bottle for collection.
4. Place a sample bottle in the sampling iron.
5. Lower a clean, opened bottle in the sampling iron to the surface of the water. Quickly lowering the sampling iron through the ice column and into

the flowing water below it will minimize the amount of ice-melt water collected.

6. Lower the sampling iron at a uniform rate until the sampling iron sinks as close to the bottom as possible. Raise the sampling iron at the same uniform rate. The objective of this method is to completely fill the bottle as it returns to the water's surface. It may take some practice to fine tune the transit rate for different bottle sizes and stream depths. By using this method, a quasi-depth-integrated sample may be obtained.
7. Do not allow the sampler to come in contact with the stream bed, as this may stir up bottom sediments, thus contaminating the water sample. Predetermining the water depth and measuring out the length of rope required to sample the whole water column will help to prevent contacting the stream bed when sampling.
8. If required by the laboratory, rinse the bottle and cap (see Table 4) and repeat the filling procedure until all bottles have been filled.
  - a) Metals, major ions, ammonia, and cyanide samples get filled from the bulk 2 L nutrient bottle. Rinse the bulk 2 L bottle twice (Table 4) and fill, as per filling procedures. Pour off the water from the bulk 2 L into each metal, major ion, ammonia, and cyanide bottle, one at a time, gently swirling the bulk 2 L between each pour. Do not touch the mouths of the bottles together while pouring the samples. Dump the remaining water from the bulk 2 L and re-fill.
  - b) Rinse the inside of the lid with sample water from the bottle; cap each bottle immediately after filling and place in a cooler.
  - c) If necessary, preserve as soon as possible (Table 4).
  - d) If the temperatures are well below freezing, precautions must be taken to prevent the samples from freezing and/or breaking during transport. The use of prefabricated Styrofoam inserts made to fit inside of a standard cooler, along with Styrofoam lining the bottom of the cooler, is very effective for protecting glass bottles during transport. If the temperatures dictate, hot-water bottles may be useful for keeping samples from freezing inside their coolers.

#### **4.2.5 Sampling with a Peristaltic Pump (from a Boat)**

For this purpose, the portable peristaltic pump is used to collect a point sample at 60% depth in the system being sampled. The peristaltic pump can be used for depths of up to 8 m. To sample with the peristaltic pump, specifically the Geotech Geopump® 2 Peristaltic Pump:

1. Set up the pump. Remove the pump from its case. Plug the power cord from the pump into the outlet; plug the other end into the power source (i.e., 12 V). Set the lever to the left to open the pump head.
2. Put on clean gloves to set up the tubing. Remove the clean tubing (Appendix E) from its bag. Insert one end of the tubing into the pump head, leaving about 20 cm of tubing extending out of the pump head. Ensure the overhanging tubing is expelled over the side of the boat and is freely suspended, so it is not resting on any surfaces. Set the lever to the right to close the pump head and secure the tubing. Attach the other end of the tubing onto a weighted object that can be suspended from the boat (i.e., a sampling iron). Place the sampling iron overboard (avoid hitting the wall of the boat) and lower it to 60% depth. Tie the sampling iron off on a boat cleat to proceed with sampling.
3. Determine the direction of flow that is required to expel water from the pump end of the tubing (this depends on the way in which the tubing is placed into the pump head) and set the toggle switch accordingly for flow direction (forward or reverse).
4. Turn the On/Off toggle switch to “ON”. Change gloves.
5. At each sampling location, flush the tubing for approximately 10 minutes. It takes 1 minute to completely flush tubing that is 19 m in length; therefore, flushing the tubing about 10 times with the hose volume of water is ideal. The flushing time can be varied to achieve 10 rinses based on the length of the tubing being used.
6. Prepare the sampling container to be collected. Proceed with using the CH/DH sample-collection technique (Appendix E). Remove the lid from the sample bottle and hold it under the tubing; do not insert the tubing into or touch the tubing to the bottle. Rinse the bottle if required (Table 4) and then fill.
7. Rinse the inside of the lid with sample water from the bottle; cap each bottle immediately after filling and place in a cooler.
8. If necessary, preserve as soon as possible (Table 4).
9. Turn the pump “OFF”.
10. Set the lever to the left to open the pump head and remove the tubing. If the tubing is going to be used again at the same site, it can be removed from the pump and carefully coiled by the CH person. Keep the tubing clean as it is brought into the boat. The CH person can then place the tubing back inside the bag it originally came out of.

**Note:** If using the peristaltic pump and tubing to collect mercury samples, use the CH/DH sample-collection technique to set up the pump and tubing, as well as to collect the sample bottle. The CH/DH technique – as outlined in the Mercury Protocol from Flett Laboratories, and which is used to collect mercury samples – can be reviewed in Appendix E.

#### 4.2.6 Sampling with a VOC Sampler (from a Boat)

The volatile organic compounds (VOC) sampler is designed for the collection of non-aerated samples in 40 mL glass vials to determine volatile organic compounds (USGS variously dated. The VOC sampler can be used in the collection of CCME-F1 samples in the open-water season. Prior to use, the VOC sampler should be cleaned properly, as per the details in Appendix D. When ready to use the sampler in the field:

1. Put on new nitrile gloves and remove the VOC sampler from the clean poly bag.
2. Undo the latches on the sampler lid and place the lid upside down (so it rests propped on the air exhaust tube, not on the copper inlet tubes).
3. Remove the caps on the 40 mL glass amber septum vials and set the caps aside on a clean surface where they will not be contaminated, knocked off, or blown away. Place the vials in the cups inside the sampler.
4. Replace the lid back on the sampler, carefully placing the copper inlet tubes into the vials. Ensuring that the latches line up with the notches on the lid, secure the lid on the sampler.
5. Suspend the sampler over the side of the boat from a rope and lower it to 60% depth. Secure the rope to a cleat or side rail, as the sampler needs to remain suspended at one depth for 7 minutes. Over the next 7 minutes, the vials will flush seven times and the final sample will be retained in the final 15 to 20 seconds of filling.
6. Prepare the materials required to preserve the sample while waiting for the VOC sampler to fill.
7. Once the sampler has been suspended under water for 7 minutes, bring it to the surface. With clean, gloved hands, remove the lid.
  - a) If the sampler is not completely full, it has not rinsed the vials the required seven times. Dump the collected water out of the sampler and vials and begin again. This time, leave the sampler in the water longer to ensure it is full before retrieving it.
  - b) Carefully remove the vials from the VOC sampler by gripping the vial around the neck with a pair of clean (methanol-rinsed), stainless-steel

tongs. There should not be any headspace in the vial. Preserve the samples and cap immediately. Invert the vial several times to mix in the preservative.

- c) Place the vials in the cooler with ice packs; however, during the winter sampling months, extra precautions will have to be taken to ensure that the samples do not freeze.

**Note:** Sampling with a VOC sampler in winter, or below 0°C conditions, is very difficult. The small-diameter inlet tubes are prone to freezing. The use of the VOC sampler is often limited to open-water seasons when the sampler is not subject to freezing.

#### 4.2.7 Collecting a Flow-Weighted (Isokinetic) Depth-Integrated Sample

The collection of isokinetic, depth-integrated samples involves using either an equal-width-increment (EWI) or equal-discharge-increment (EDI) sampling method. The EWI or EDI methods usually result in a composite sample that represents the discharge-weighted concentrations of the stream cross-section being sampled. The EWI and EDI methods are used to divide a selected cross-section of a stream into increments with a specified width. The term “vertical” refers to that location within the increment at which the sampler is lowered and raised through the water column.

1. EWI verticals are located at the midpoint of each width increment.
2. EDI verticals are located at the centroid, a point within each increment at which the stream discharge is equal on either side of the vertical.

If properly implemented, the EDI and EWI methods should yield identical results. The uses and advantages of each method are summarized in the “USGS National Field Manual for the Collection of Water quality Data Chapter A4” (USGS 2006). Isokinetic samplers, such as the DH-95, are used to obtain a discharge-weighted sample along the stream cross-section.

The EWI, used in the Joint (Canada–Alberta) Oil Sands Monitoring (JOSM) program, does not require pre-existing data on the depth and velocity across the cross-section. Rather, the river is divided into equal-width increments, or “panels”. Samples are collected by lowering and raising the isokinetic sampler through the water column at the center of each panel at a rate (transit rate) determined by the current velocity and depth of the water measured in the deepest part of the river (USGS 2006). The combination of a constant transit rate used to sample at each panel, regardless of the depth and isokinetic property of the sampler, results in a discharge-weighted sample that is proportional to total streamflow. EWI isokinetic samples were typically obtained from 10 panels across the Athabasca River, composited in a clean sampling churn, and the composite was analysed as one sample.

**Notes:**

- For the JOSM, isokinetic composite samples were collected only for major ions, nutrients, and metals. This method was not used to collect samples for organic compounds and mercury due to concerns over contamination.
- All equipment that touches the sample, such as the isokinetic bottles, isokinetic nozzles, and sample churns, are cleaned using the equipment-cleaning methods for non-organic water samples described in Appendix D.

**Field methodology:**

1. Determine the wetted width of the river transect using a measuring tape, rangefinder, or GPS.
2. Determine the width of each panel. Typically, 10 panels were used for the EWI method of isokinetic sampling on the Athabasca River for JOSM; therefore, the wetted width of the river was divided by 10 to obtain the width of the panels.
3. The water depth and river velocities need to be measured at the midpoint of each panel in order to determine the transit rate for the isokinetic sampler. When using the EWI method to collect an isokinetic sample across the river, the transit rate is the same for each panel that is sampled. Refer to the “USGS National Field Manual for the Collection of Water quality Data Chapter A4” (USGS 2006) for guidelines on how to determine transit rates and bottle nozzle sizes.
4. Once the correct transit rate and nozzle size for the sampling bottle have been determined, a test sample should be taken with the sampling winch. Some winches are manually operated via a hand crank and some may be motorized. Ensure that the winch meter has been zeroed at the water’s surface, so that the sampler is submersed to the correct depth.
5. Collect an isokinetic, depth-integrated sample at each panel across the river. Each isokinetic sample taken is poured into a pre-cleaned 8 L sample churn, so that it contains a composite sample made up of all individual panel samples.
6. Once all of the panels at a transect have been sampled, use the sample churn to dispense the cross-sectional composite sample into the individual sample bottles for each unique analyte. The churn paddle should be gently agitated up and down to homogenize the sample and keep the sediments suspended while pouring off the sample.

### 4.3 Post-Sample Collection Considerations

Tasks to be performed following sample collection are program specific, but several considerations should be taken after each water-sampling event to aid in the QA of the sample and corresponding data.

- The field sheets should be complete and accurate. Metadata (sample number ID, site, date/time of collection, parameters collected, etc.) and field notes serve as the original documented source. Anything that is noteworthy from the sampling event should be documented. This may include possible sources of contamination, alternate methods used to collect the water sample, and anything that strays from the SOPs pertaining to that sample. File the original field sheet document and create and save an electronic copy.
- Laboratory submission forms and chain-of-custody forms must be accurately prepared. Sample numbers, sample dates and times, site names, and analyses requested should all be accurate. Create a copy of the original to keep on file.
- Ensure timely and appropriate shipping; know the holding times for the collected samples and have shipping plans in place to get the samples to the laboratories on time. Take appropriate measures to keep the samples between 0°C and 4°C and prevent breakage of the sample bottles (i.e., more ice packs are required in the coolers during the summer months than in the winter months, there should be adequate use of packing inserts and bubble wrap, the lids should be secured, etc.).
- Update the sample number tracking logs with the samples collected and corresponding metadata (site, date, time, etc.).

### 4.4 Quality Assurance, Quality Control (QA/QC)

For the purposes of the 2012–2017 JOSM program, QA/QC was defined as a sample set collected for every 10%–15% of environmental samples taken. A QA/QC sample set consists of obtaining a series of triplicate samples and a series of three blank samples, for all parameters sampled, as well as a fourth blank for any samples that require preservation in the field. The samples collected as part of the QA/QC set include the following types:

- **Field blanks:** The field blank supply bottles are filled with type 1 water (following identical bottle filling and rinsing procedures, as when collecting the environmental samples), and they are capped and shipped to the site with all the other sample bottles. Field blank supply bottles are opened at the site, and the type 1 water is transferred to a clean, empty field blank bottle (following identical bottle filling and rinsing procedures, as when collecting environmental samples) and treated and handled as an



environmental sample would be (i.e., preserved if required). The true field blank bottle is sealed, placed in the cooler and shipped to the labs with the environmental samples.

- **Field blanks with laboratory preservative:** The supply bottles are filled with type 1 water (following identical bottle filling and rinsing procedures, as when collecting environmental samples); they are then preserved, capped, and shipped to the site with all the other sample bottles. Field blanks with laboratory preservative are only for those samples requiring preservation in the field (i.e., ammonia, phenol, BTEX, and cyanide). In the field, the type 1 water is transferred to a clean, true field blank with a laboratory preservative bottle, sealed, placed in the cooler, and shipped to the laboratories with the environmental samples.
- **Travel blanks:** These are filled with type 1 water (following identical bottle-filling and rinsing procedures, as when collecting the environmental samples), preserved, capped and shipped to the site with all of the other sample bottles. The travel blanks travel to the site with the other samples; they are left sealed in the cooler for the duration and then shipped for analysis with the other samples.
- **Bottle blanks:** These are filled with type 1 water and stored at 4°C at the location of filling (they do not travel to the sample site); they are shipped to the laboratory for analysis. Shipping should be timed, so that the bottle blanks arrive at the laboratory for analysis on the same day as the other QA/QC samples and environmental samples shipped from the field.
- **Triplicate samples:** This involves the physical collection of three environmental samples at the same location in the water body, in quick succession, one after another. These replicates are collected sequentially, using identical sampling procedures for the collection of each bottle.

The filling of the blank samples should be done as close to the date of the QA/QC event as possible to minimize storage times. The date of filling should be recorded on the bottles and on the field sheet. A schematic of the blank bottle filling and sampling procedure can be viewed in Appendix F. The blank samples are stored in a refrigerator at 4°C until they are required to be transported in coolers with ice packs. In the field, at the QA/QC site, blanks are collected before any of the environmental samples are taken.

## 5.0 References

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# Appendices

## Appendix A. Examples of Further Equipment Lists (Project Specific)

### 319 Summer Sampling Packing List

Activity	Equipment
Wetted Widths	GPS 720S (on FWQMS boat)
	Markers for wetted widths
	Hammer to install signs
	Range Finder with tripod
	Spare GPS with site coordinates
IsoKinetic	DH-95 isokinetic sampler
	Motorized plate with remote and power cable
	bottles with increments on side
	nozzles
	Churns (3x)
	stopwatch
Jiffy Crane	Crane arm
	sampling iron
VOC sampler	VOC sampler
	tongs
	VOC field and trip blank vials
	rope to deploy of side of boat
Peristaltic Pump	black case with pump and power cords
	tubing
	gloves
	twist wire for attaching tubing to isokinetic cable
Sampling	All bottles as per field trip
	QC bottles as needed
	preservatives
YSI case	calibrated YSI
	5 meter underwater communication cable
	guard with extra weight attached to bottom
	tap water to place probe into for storage
Field Observations	field sheets
	field notebook
	camera
	kestrel weather station
Boat Needs	9.9 hp kicker motor with fuel line
	first aid kits
	extra gas
	life vests (1 for each person in boat + spare)
	cell phone charger (12v)
	Waders (3)
	rubber boots
	connector cleaner
	silicone grease for 2" isokinetic pipe
	spare bulbs for trailer
Extras	flow tracker & rods
	spare bottles
	spare batteries (AA & AAA)
	Electronics box
	bug spray
	paper towel
	extra water

### 319 Winter Sampling Packing List

Activity	Equipment
Wetted Widths	GPS with site coordinates range finder Markers for wetted widths Hammer to install signs Drill with spare batteries Ice thickness measurement kit Depth sounder Marked rod for depth checking (back-up)
Hole drilling	Jiffy electric auger (spare to be kept in trailer) Two deep cycle batteries large snow shovels small sled to pull the auger and battery by hand Ice thickness measurement tool
Velocity Meter	Flowtracker with 5m cable in hot case Water Survey winter rods (4 rods) Flowtracker to water survey rod adapter * lots of extra hot packs
Sampling	Sampling iron rope light hammer to knock ice off sampling iron churns, jugs in cooler or specialized box
Peristaltic Pump	black case with pump and power cords Clean tubing gloves (mercury and nitrile) twist wire for attaching tubing to sample iron Hot box to keep tubing thawed
Sampling	All bottles as per field trip QC bottles as needed preservatives
YSI case	calibrated YSI 5 meter underwater communication cable guard with extra weight attached to bottom hot box for YSI
Field Observations	field sheets field notebook camera kestrel weather station
Snowmobiles Shelter toboggan Long toboggan	two snowmobiles in trailers covered shelter toboggan 1 toboggan with cover 1 large cargo net for shelter toboggan spare tie ropes spare small shovels Ice safety kit tow ropes
Extras	spare bottles spare batteries (AA & AAA) Electronics box paper towel extra tap water personal protective gear ceramic heaters extra 12v batteries

# Appendix B. Field Sheet Examples

PROJECT #319 OIL SANDS LONG-TERM WQ MONITORING														page 1 of 2						
Date/Time	Station	Sampled by:	GPS left (West) bank (dd)	GPS right (East) bank (dd)	W	W	Air Temp (C)	Precip	Cloud cover (%)	Wind (km/hr)	Wetted Width	① PA	② PA	③ PA						
Panel No.	Ideal Distance from West shore (m)	Actual Distance from West shore (m)	Depth (m)	Velocity (m/sec)	GPS Readings (latitude/longitude) (dd)	Sample Number	Sample Time (MDT)	Temp (°C)	SpCond (µS/cm)	pH	D.O. (mg/L)	Turbidity (NTU)	Saturation	Burifunction	Estimation	Flatt	PYLET	Peru	Comments	
1						20XXPN88							√	√	√	√	√	√	√	panel vertical grab for M, panel 1
2																				
3																				
4						20XXPN88							√	√	√	√	√	√	√	panel vertical grab for M, panel 4
5																				
6																				
7																				
8																				
9																				
10						20XXPN88							√	√	√	√	√	√	√	panel vertical grab for M, panel 10
Reserved for QC Blanks																				
Field Blank																				
Field Blank/P																				
Travel Blank																				
Bottle Blank																				
Reserved for QC Blanks																				
Field Blank																				
Field Blank/P																				
Travel Blank																				
Bottle Blank																				

Date/time					
Station:					
Sampled by:					
page 2 of 2					
<b>NOTES:</b>					
<b>Field Personals:</b>					
<b>Notes:</b>					
<b>QA/QC:</b>					
Date and Time CCME - F1 Trip and Field blanks filled:		and	Milli-Q system (room # and resistivity reading) -	and	Filled by:
<b>Preservatives:</b>					
NH3 (10% H2SO4) from NLET:	[LAB]	Date Prepared:	Lot #:	Date Prepared:	Date 1st opened:
	[FIELD]	Date Prepared:	Lot #:	Date Prepared:	Date 1st opened:
Cyanide (NaOH) from PNELT: Date received:					
Phenol and CCME-F1 (6N H2SO4) from Fisher: cat #:	SX1247-1	Date Purchased:	Lot #:		
	[LAB]	Date Poured into working bottle:			
	[FIELD]	Date Poured into working bottle:			

**Field Sampling Sheet**

**Environment Canada  
Prairie and Northern Region Project 339**

Project Description: \_\_\_\_\_ Date: \_\_\_\_\_

Station: \_\_\_\_\_ Sampled By: \_\_\_\_\_ Time Zone: \_\_\_\_\_

Narrative: \_\_\_\_\_

Air Temp (°C) \_\_\_\_\_ Ice Thickness (m) \_\_\_\_\_ Cloud Cov. % \_\_\_\_\_ % Ice Cover \_\_\_\_\_ Wind \_\_\_\_\_ Precip. \_\_\_\_\_

Sample Frequency \_\_\_\_\_ Sample Method \_\_\_\_\_

Comments: _____	Sample #									
_____	Location									
_____	Time									
_____	D.O. (M/L)									
_____	Water Temp (°C)									
_____	Spec Cond (25°C)									
_____	Turbidity (NTU)									
_____	PH									
_____	Depth (m)									

**NLET SASKATOON**

Container	Parameters	Preservation
2L PE	Physicals, NFR/NFRF Nutrients	Cool to 4°C
125ml PE	N Amonia	1ml 10% H <sub>2</sub> SO <sub>4</sub> Cool to 4°C

**NLET BURLINGTON**

Container	Parameters	Preservation
500ml PE	Major Ions	Cool to 4°C
500ml PE	Buck Metals	Cool to 4°C
1L PE	Amber	Cool to 4°C

## Appendix C. Laboratory Submission Sheet Examples

EOALRSD MONITORING and RESEARCH SAMPLE SUBMISSION FORM V2.0 FORMULAIRE DE DEMANDE D'ANALYSES DU ULAOSR											
Client Information		Project Information		Collection Information		Analysis Information		Sample Information		Remarks	
Client Name	Client Address	Project Name	Project Location	Collector	Date	Time	Zone	Sample ID	Sample Type	Remarks	Temp. on Arrival
Client Name: <b>max@chuv.com</b> Client Address: <b>max@chuv.com</b>		Project Name: <b>max@chuv.com</b> Project Location: <b>max@chuv.com</b>		Collector: <b>max@chuv.com</b> Date: <b>max@chuv.com</b>		Time: <b>max@chuv.com</b> Zone: <b>max@chuv.com</b>		Sample ID: <b>max@chuv.com</b> Sample Type: <b>max@chuv.com</b>		Remarks: <b>max@chuv.com</b> Temp. on Arrival: <b>max@chuv.com</b>	
Additional analysis requests (check boxes):											
<input type="checkbox"/> Ammonia <input type="checkbox"/> NO3&NO2 (DL=0.010 mg/L) <input type="checkbox"/> TDN <input type="checkbox"/> TP <input type="checkbox"/> TP_Diorthoaid <input type="checkbox"/> TSS/SS <input type="checkbox"/> TURBIDITY <input type="checkbox"/> COLOUR TRUE <input type="checkbox"/> Chlorophyll (OC) <input type="checkbox"/> PON-POC											
Additional sample information (check boxes):											
<input type="checkbox"/> sample water <input type="checkbox"/> sample water <input type="checkbox"/> sample water											









## Appendix D. Cleaning Water Quality Sampling Field Equipment

### Standard Operating Procedure – Preparing Acid Baths

**Objective:** Acids baths are commonly used in the preparation of field sampling equipment that will be used for the collection of water quality samples for metals analyses (including Hg). The acid bath is changed every 7 months, or when there is an accumulation of sediment in the tub. Pre-cleaning the equipment is important for ensuring that the amount of sediment introduced to the acid bath is minimized.

### **WARNING**

It is important to note that one should **NEVER ADD WATER TO ACID; ALWAYS ADD ACID TO WATER.**

### **Equipment and supplies:**

- Personal protective equipment (PPE) – Laboratory coat, face shield, apron, and long-sleeved Neoprene gloves.
- One (1) 2.5 L jug of 37.5% HCl (Fisher Scientific FLA144S-212).
- Sodium bicarbonate (Tronox).

### **Acid bath setup:**

1. Put on PPE.
2. Fill the acid bath with 30 L of Milli-Q water; there should be a line on the tub indicating this amount or it can be calculated using the volume formula ( $L \times W \times H$ ). Remember  $1 \text{ cm}^3$  equals 1 mL.
3. Add 2.5 L of HCl to the bath slowly and carefully.
4. Place the lid back on the bath and let the mixture equilibrate for 1 to 2 hours.

### **Acid bath disposal:**

1. Put on PPE.
2. Slowly add the sodium bicarbonate, observing the chemical reaction taking place.
3. Once you have enough sodium bicarbonate to ensure that the reaction has completed, add another scoop to ensure completion.
4. Dispose of neutralized acid in the sink; rinse the sink with copious amounts of water post-disposal.

## **Standard Operating Procedure – Cleaning of Peristaltic Pump Tubing**

**Objective:** The purpose of this SOP is to outline the cleaning practices for platinum-cured silicone tubing used to collect water quality samples for mercury analysis. The following protocol was adapted from the FLETT instruction on mercury sampling using tubing (Appendix E). It is also important to note before proceeding that all Material Safety Data Sheets (MSDS) should be available and reviewed, and that the acids should be disposed of in accordance with disposal guidelines.

### **Equipment and supplies:**

- PPE.
- One (1) 2 L container of 1% HCl.
- One (1) polypropylene autoclave tray (large enough for tubing).
- Two (2) clean plastic bags per tube.
- One (1) 2 L container of deionized water.
- Nitrile gloves.
- One (1) waste container for HCl.

### **Before/after sampling events:**

1. Put on the nitrile gloves, laboratory coat or apron, and safety glasses. Make sure to work in a fume hood or well-ventilated place free of dust or other contaminants.
2. Set up the working station with a peristaltic pump, deionized water tub, HCl tub, and tubing.
3. Rinse the tubing in a sink with warm tap water to remove any sediment or debris.
4. Remove excess water and place the tubing into a peristaltic pump. Put one end of the tubing into clean, deionized water and run 2 L of deionized water through the tubing. Run the pump until the water is exhausted and the pump runs dry. Dispose of the waste deionized water.
5. Take both ends of the tubing and place in the 1% HCl. The rest of the tubing should go into a clean, acid-rinsed, non-metal container to be kept clean.
6. Turn on the peristaltic pump and pump the HCl through the tubing for 2 hours; ensure all interior surfaces of the tubing are rinsed.
7. Drain the tubing until no acid remains. Place one end of the tubing into the clean, deionized water tub and the other end into a waste container. Flush the tubing

with 4 L of water and remove any excess water by holding the tubing in a vertical position.

8. Carefully seal the tubing in two (2) plastic bags and mark it with a “Clean tubing” label with the date of cleaning and the name of the person who did the cleaning.

## Standard Operating Procedure – Cleaning of the VOC Sampler

**Objective:** The purpose of this SOP is to outline the procedures for cleaning the VOC sampler before sampling for volatile organic parameters such as CCME-F1 and/or BTEX. These procedures are adapted from the USGS operating procedures for the cleaning of equipment used for organic-compound sampling (2004). Generally, all equipment should be cleaned in an area protected from sources of contamination. The following sampling procedures are best performed in a laboratory setting, but they can be modified for field situations.

### Equipment and supplies:

- PPE
- Nitrile (or other disposable, solvent-resistant) gloves
- Free-rinsing, non-perfumed laboratory detergent (e.g., Fisher brand Sparkleen I)
- Firm sponge or soft brush
- Wash basin and drying surface (preferably stainless steel)
- Aluminum foil
- Methanol (ACS pesticide grade)
- Methanol fluorocarbon-polymer wash bottle
- Clean polymer bags
- Clean, designated cooler (of appropriate size)

**Working with methanol:** When working with methanol, it is important to closely follow all MSDS and Workplace Hazardous Materials Information System (WHMIS) protocols. Ensure you have reviewed all MSDS information before handling methanol. It is important to remember to work in a well-ventilated space and to use as instructed.

### Procedure:

1. Put on nitrile gloves, laboratory coat or apron, and safety glasses.
2. Prepare the cleaning area. Wash down the stainless-steel sink and counter with the detergent solution, and rinse with hot water. If no stainless-steel working surface is available, cover the area to be used for drying the equipment components with aluminum foil.
  - a) At this time, it is worthwhile to clean the designated storage container (i.e., cooler) that is used to house and transport the sampler. Scrub the interior and

exterior of the cooler with the detergent solution. Rinse thoroughly and dry out the inside.

3. Change gloves. Prepare a basin of the detergent solution. Remove the lid from the VOC sampler and place all components in the detergent solution. Using a sponge or soft brush, scrub the exterior and interior of the sampler as best as possible. Make sure any adhering materials, such as oil, sediment, algae, etc., are removed. Pay particular attention to cleaning under the inlet cover of the VOC sampler, as it is a hard-to-reach area.
4. Drain the detergent solution from the basin and rinse the sink and equipment thoroughly with tap water to remove detergent residue. Again, pay particular attention to rinsing under the VOC sampler inlet cover, as well as the copper inlet tubes. Change gloves.
5. Rinse the exterior and interior of the sampler with a minimum amount of methanol using a methanol fluorocarbon-polymer wash bottle. Make sure to methanol rinse under the inlet cover, as well as down the copper inlet tubes, as best as possible.
6. Place the methanol-rinsed components onto the clean stainless-steel (or aluminum-foil-covered) surface.
7. Allow the sampler components to completely air dry in an area free of airborne contaminants. If required, aluminum foil can be draped loosely over the equipment as it dries to protect from potential airborne contaminants.
8. Once all VOC sampler components are completely dry, secure the lid back on the sampler base, place it in a clean poly bag, and seal the bag. Place the VOC sampler inside the designated (and cleaned) cooler.
9. Label the cooler as VOC sampler cleaned, note the date, and initial.

**Note:** If multiple sites are being visited on the same day, pre-clean a second VOC sampler to avoid unnecessary field cleaning between sites.



## Standard Operating Procedure – Churns and Isokinetic Sampler

**Objective:** Equipment re-used for composite sampling, such as churn splitters and isokinetic bottles and nozzles, were cleaned using a series of steps for nonmetal equipment. Steps include a detergent wash, an acid bath, and a deionized water rinse, before being dried and bagged for transport. For a detailed schematic on the flow of the operations, review Figure 3-2 of the [USGS Equipment Cleaning Protocol](#). Details on the procedure can be viewed in Section 3.32 titled “Churn Splitters” under the [USGS Equipment Cleaning Protocol](#) (Wilde 2004).

### Equipment and supplies:

- PPE
- Liquinox
- 3% acid bath
- Milli-Q water

### General considerations:

- Personnel completing the protocol should first be trained by a senior technician before proceeding with cleaning.
- Carefully go over the acid-bath protocol and ensure all PPE is worn.

### Procedure:

Pre-wash all equipment with Liquinox soap in a laboratory sink, ensuring removal of any visible debris or sediment. Remove the spigot and clean inside.

1. Rinse thoroughly with deionized water.
2. Allow the equipment to air dry, on a clean surface.
3. Add all equipment to the acid bath; completely submerge all equipment.
4. Let sit overnight.
5. Carefully remove the equipment from the acid bath and put onto a clean autoclave tray; ensure all remaining acid has drained from the equipment surface into the bath.
6. Completely rinse all equipment seven times with Milli-Q (deionized) water.
7. Let air dry on a clean surface.
8. Bag and mark “Cleaned” with date and initials.

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## **METHYL & TOTAL MERCURY WATER SAMPLE COLLECTION AND HANDLING (NO PRESERVATION)**

Samples should be collected only into rigorously cleaned Teflon or glass bottles with Teflon-lined caps. Under no circumstances should ordinary plastic (i.e., polyethylene, polypropylene, or polyvinyl chloride [PVC]) containers be used, as they are very permeable to Hg gas from the air. It is critical that the bottles have tightly sealing caps to avoid diffusion of atmospheric Hg through the threads (Gill and Fitzgerald 1985). As an added precaution, the cleaned bottles are dried, capped, filled with high-purity 0.4% HCl, and double bagged into new Ziploc bags in the clean room. The sample bottles are then stored in wooden or plastic boxes until required.

### **Dip Sampling of Surface Water – Use Only Double Bagged Bottles**

Bottles are clean on both the interior and exterior and are enclosed in two (2) plastic Ziploc bags. During non-freezing weather, the bottle will usually be shipped containing clean 0.4% HCl. During freezing weather, when the contents may be accidentally frozen, the bottles may be shipped empty.

The samples are collected using rigorous ultra-clean protocols (Gill and Fitzgerald 1985), which are summarized as follows:

1. At least two people, wearing fresh, unpowdered, clean-room gloves at all times, are required on a sampling crew.
2. One person (“dirty hands”) pulls a bagged bottle from the box and opens the outer dirty bag, avoiding touching inside that bag.
3. The other person (“clean hands”) reaches in, opens the inner bag, and pulls out the sample bottle.
4. The bottle is opened and the acidified water is discarded downstream (away) of the sampling site.

- "Clean hands" rinses the bottle at least twice with sample water, discarding the rinse water downstream (away from the site), and then fills the bottle, holding it near the bottom during filling with the mouth about 10 cm below the water's surface facing into the current flow. When sampling from a boat (or aircraft), the boat should be slowly moving and the sample taken from the side of the boat closest to the direction of movement. Fill the bottle according to the following table.

Bottle Type	Glass			Teflon		
	Total Mercury		Methyl Mercury	Total Mercury		Methyl Mercury
Season	Summer	Winter	All Year	Summer	Winter	All Year
Fill to	~95%	~50%	~50%	~95%	~85%	~80%

- The cap is replaced, firmly tightened, and the bottle is re-bagged in the opposite order from which it was removed.
- Clean-room gloves are changed between sample locations and whenever something not known to be clean is touched. If obvious contamination of the "clean hands" gloves has not occurred, they may be retained in a clean plastic bag for use by the "dirty hands" person at the next sampling location.

### Pump Sampling Using Bagged Bottles

Bottles are clean on both the interior and exterior and are enclosed in two (2) plastic Ziploc bags. During non-freezing weather, the bottle will usually be shipped containing clean 0.4% HCl. During freezing weather, when the contents may be accidentally frozen, the bottles may be shipped empty. These bottles can be labelled with a waterproof marker or adhesive labels prior to use, as they will not be dipped into the sample medium.

Samples are collected using rigorous ultra-clean protocols (Gill and Fitzgerald 1985), which are summarized as follows:

- At least two persons, wearing fresh, unpowdered, clean-room gloves at all times are required on a sampling crew.

2. One person (“dirty hands”) pulls a bagged bottle from the box and opens the outer dirty bag, avoiding touching inside that bag.
3. The other person (“clean hands”) reaches in, opens the inner bag, and pulls out the sample bottle.
4. The bottle is opened and the acidified water is discarded downstream of (away from) the sampling site.
5. It is recommended that at least 10 pump/tubing volumes of sample water be passed before rinsing the bottles. It is best to have two (2) sampling personnel, one who handles the pump and another who holds the bottle. Try to wash the exterior of the pump-delivery tube by pointing the tube vertically and allowing the exiting water stream to fall back onto the tube for several seconds.
6. “Clean hands”, wearing clean-room gloves, uncaps the bottle and places the bottle mouth in front of the sample water stream, being careful NOT to insert the tube into the bottle. This is to avoid contaminating the bottle with Hg, which may be on the exterior walls of the tube. After filling to about 30% capacity, the bottle is swirled and the rinse water is discarded. A second rinse is similarly performed. Finally, the bottle is filled with the sample and tightly capped, with filling carried out according to the following table:

Bottle Type	Glass			Teflon		
	Total Mercury		Methyl Mercury	Total Mercury		Methyl Mercury
Sample Type						
Season	Summer	Winter	All Year	Summer	Winter	All Year
Fill to	~95%	~50%	~50%	~95%	~85%	~80%

7. The cap is replaced, firmly tightened, and the bottle re-bagged in the opposite order from which it was removed. [Re-bagging is not critical; just ensure that the bottle is well enclosed in bubble wrap.]
8. Clean-room gloves are changed between sample locations and whenever something not known to be clean is touched. If obvious contamination of the “clean hand” gloves has not occurred, they may be retained in a clean plastic bag for use by the “dirty hands” person at the next sampling location.

9. Fill out the sample sheet, listing the sample names, sampling dates, and the analysis required. **If glass** – The bottles should be carefully packed to avoid breakage during transport, in a fashion similar to the way in which they were packed when sent to you. Make certain that there is no empty space in the cooler before shipping – voids can be filled with bubble wrap.

\*\*\* The above is extracted from the protocols of Nicholas Bloom, John Rudd, and Bob Flett – September 1993 \*\*\*

## **Sample of the Cleaning Procedure for Peristaltic Pump Tubing**

With a length (e.g., 10 m) of platinum-cured silicone tubing, the following can be (and has been) used to prepare for trace-level Hg water sampling.

In a clean container, the coiled tubing can soak in 1% HCl; acid must first be aspirated through the tubing, such that it is likely to be free of air bubbles. The container can be sealed up, with the tubing left to soak overnight at room temperature.

The next day, the tubing can be drained and the HCl removed or discarded. Tap water can now be used to continue to leach any mercury from the tubing. Using a sink, the tubing can remain in the container, while one end is connected to the faucet. Using a low flow rate (200–300 mL/minute), the water first flushes the interior of the tubing and goes on to fill the container to overflow, flushing the exterior of the tubing in the process. This can also be left to carry on overnight.

The following day, after draining the tubing of tap water, 1 to 2 L of deionized water can be aspirated through as a final rinse. The tubing is aspirated further until there is no more liquid in the tubing, aside from the small droplets that are likely to be present on the interior walls.

The tubing is then carefully sealed up into two or more clean plastic bags, in preparation for transport to the field.

## **Model 7570 Peristaltic Pump Operation**

To start the pump, turn the rotary switch to 'INTERNAL BATTERY OPERATION' and flip the power switch to 'ON'. The pump should be good for several hours of continuous operation before the batteries run down. At each sampling location, before filling a bottle, run the pump for 10 minutes with the hose inlet at the desired depth. It takes about 1 minute to completely flush the hose (~19 m); after 10 minutes, the tube will have been flushed with 10 hose volumes of water. This should be ample for cleaning the hose of the previous sample or contamination. Adjust the pumping time proportionately upward if you use a longer hose. Sample shallow depths before deeper depths at the same site. Try to keep the hose clean; it should not contact the boat.

When deploying, the hose should be pulled (with gloved hands) directly from the plastic bag; then it can be entered into the water. When recovering the hose, use gloved hands; try to get the hose into the plastic bag without touching anything.

The pump has an internal charger that should recharge the batteries in about 12 hours. It is necessary to plug the power cord into a 115V outlet; set the rotary switch to the 'Recharge on 115 AC' position AND turn the 'POWER' switch to the 'ON' position. [If the internal charger malfunctions, you can recharge the batteries with a regular 12V trickle charger. This will require removal of the dark=brown cover by undoing four (4) small Robertson screws on the case bottom and then sliding the cover off sideways. Very carefully, install the charger alligator clips on the batteries while they are in the case – do not remove the batteries from the pump. Do not let the clips touch any metal parts of the pump other than the battery terminals, as a short circuit may occur. The correct terminals are diagonally opposite from each other on the outside edges of the dual battery pack. The red mark indicates where the positive lead of the charger should connect. A diagram is included to show where to hook up the battery clips.] The pump can also run directly on 115 VAC by turning the rotary switch to the 'RUN ON 115 VAC' position.

Using the pump and tubing to obtain “deep”-water samples will likely require you to “weight” the tubing. There are already two (2) brass weights on the tubing, which should be sufficient. These weights have been added to the tubing at approximately 30 cm (1 foot) above the end that goes into the water (to reduce risk of contaminating the water entering the tubing). If it is a windy day, the boat should be “driven” in such a way as to compensate for the wind, so that the boat is relatively stationary. It should look like the tubing is going straight down from where it enters the water. It should not look as though the tubing is being “dragged” by the wind, current or boat movement.

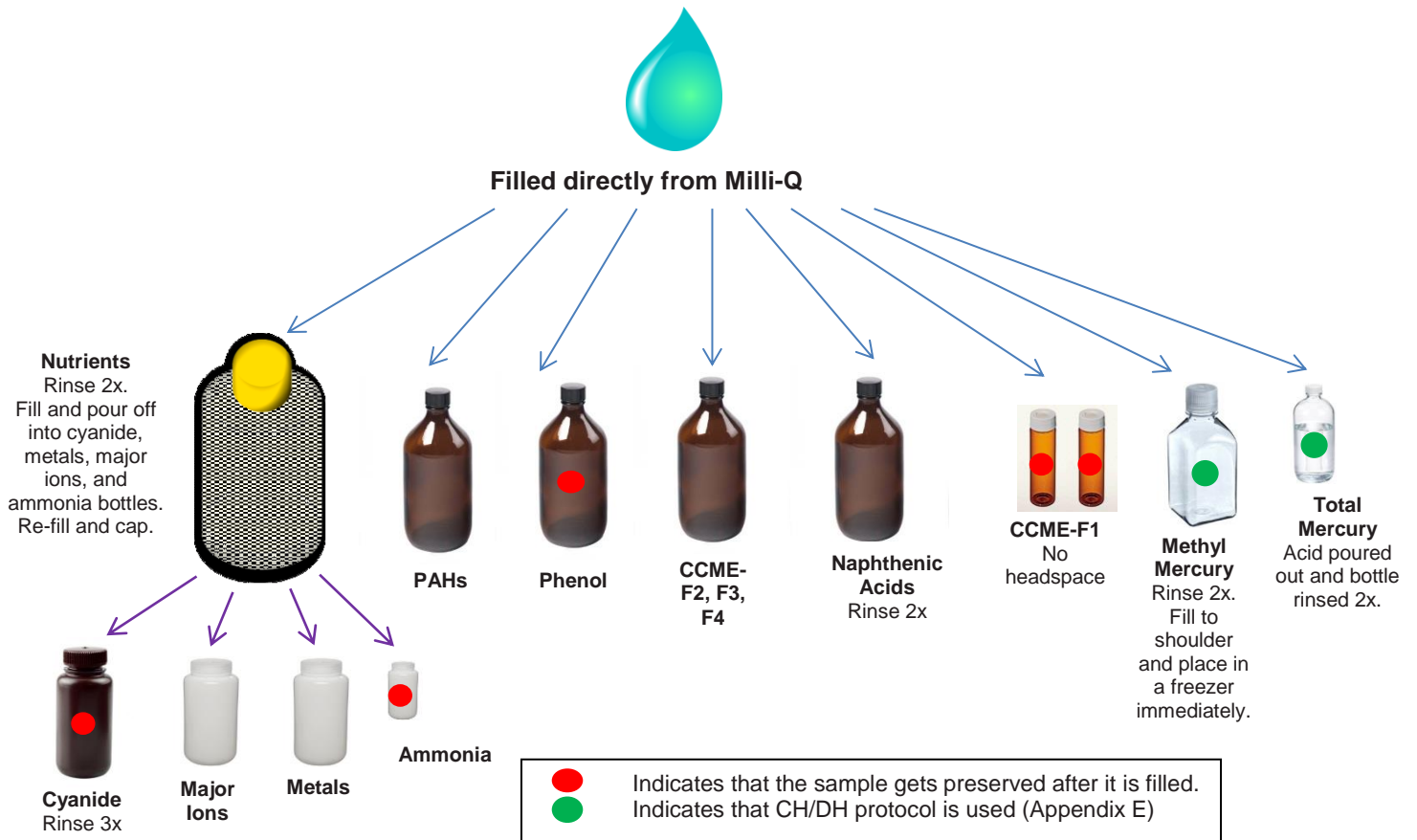
If you are sampling where standing trees may be present underwater, it is strongly recommended that a sonar unit (fish finder) is used, so you can avoid getting the hose tangled in the trees. You could potentially lose the hose and/or get particulates in your sample.

**The “deep”-water sample should be collected after all surface samples have been obtained, but before you deploy an anchor or use any other equipment that requires a line to be in deep water where you wish to sample. This is to avoid contamination of the mercury sample.** At this point, the “dirty hands” person should get the pump ready so that water can flow through the tubing from the depth to be sampled for ~10 minutes. “Dirty hands” should unzip the exterior bag on a clean sample bottle, so as to make it accessible to “clean hands”. At the end of the 10-minute tubing purge, the “dirty hands” person then holds up the exit end of the tubing vertically like a fountain, such that the water will fall back over the exterior of the tubing; rinse it for ~30 seconds. “Clean hands” should remove the bottle from the interior bag and open it. The “rinsed” end of the hose is then passed over to the “clean hands” person, who will rinse the bottle a few times with this water (the hose is **not** to be inserted into – nor should it touch – the bottle); that person will then fill the bottle with the sample (being sure to leave ~5 mL of room at the top of the bottle).

## Appendix F. QA/QC Blank-Filling and Sampling Procedure

**Note:** All blanks are filled as close to the QA/QC event date as possible.

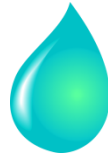
### Bottle Blanks and Travel Blanks



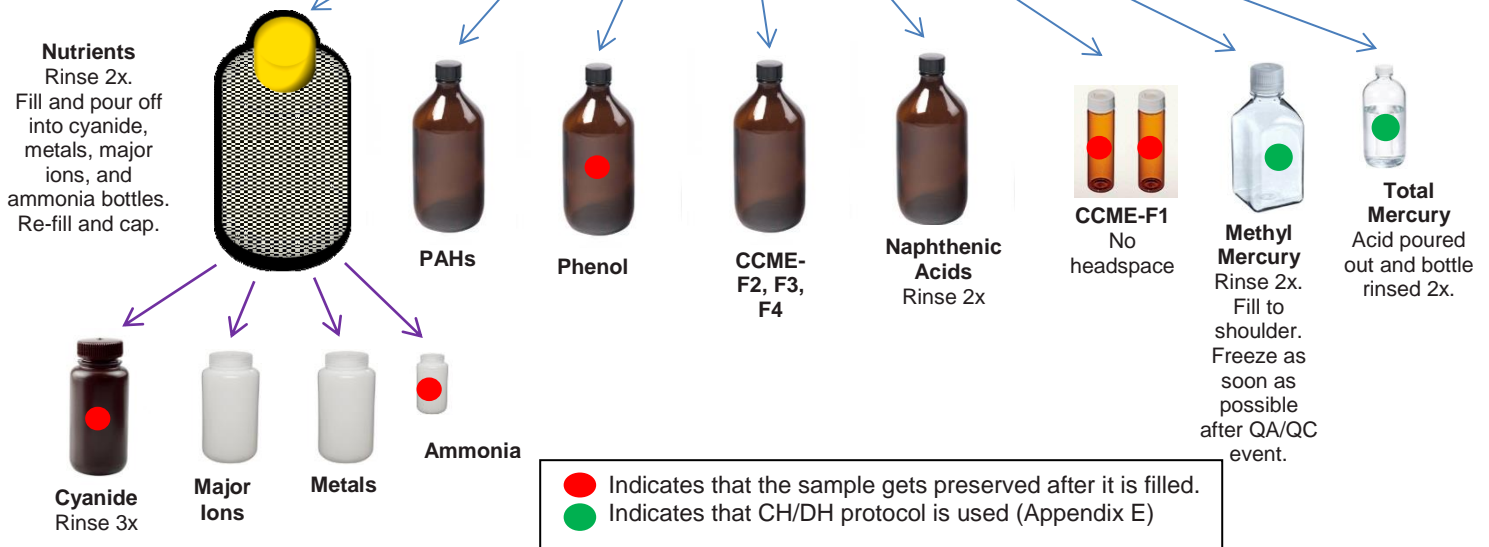
#### Bottle Blanks:

- are stored in the laboratory (or the location where they were prepared) at 4°C after filling (except for methyl mercury, as it is stored frozen); and
- are shipped to the laboratories for analysis on the day of the QC event occurring in the field, so that the corresponding samples arrive at the laboratory around the same time.

## Travel Blanks



Filled directly from Milli-Q



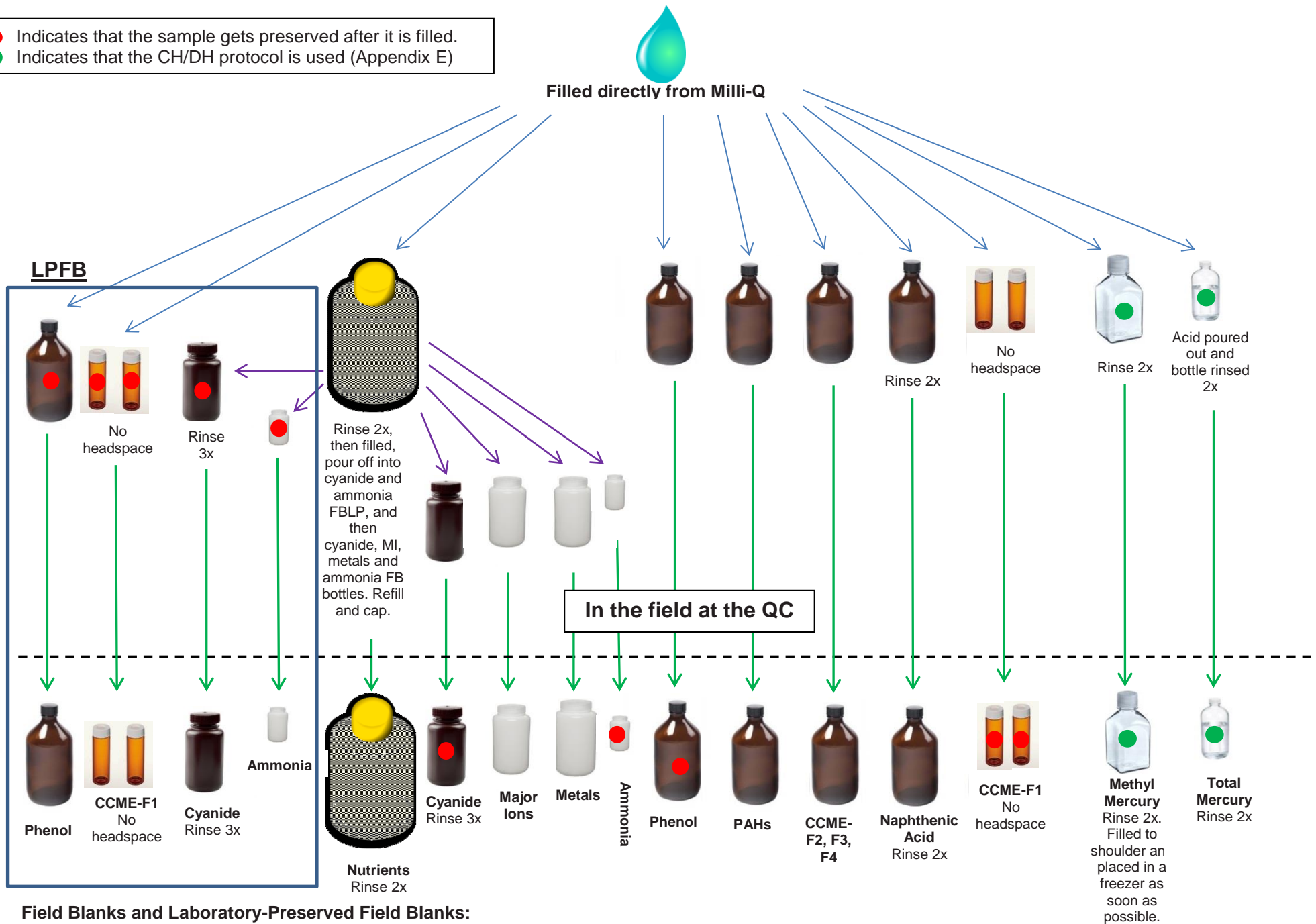
### Travel Blanks:

- are ALL stored at 4°C and are shipped or driven to the site for the QA/QC event; and
- travel to the sampling site on the QA/QC day, remain unopened, travel back, and are then shipped to the laboratory with the field samples.



## Field Blanks and Laboratory-Preserved Field Blanks (LPFB)

- Indicates that the sample gets preserved after it is filled.
- Indicates that the CH/DH protocol is used (Appendix E)



### Field Blanks and Laboratory-Preserved Field Blanks:

- are ALL stored at 4°C and are shipped or driven to the site for the QA/QC event; and
- travel to the sampling site on the QA/QC day, are opened and transferred into the true blank bottle in the field; they then travel back and are shipped to the laboratory with the field samples.

Additional information can be obtained at:

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Public Inquiries Centre

7th Floor, Fontaine Building

200 Sacré-Coeur Boulevard

Gatineau QC K1A 0H3

Telephone: 1-800-668-6767 (in Canada only) or 819-997-2800

Email: [ec.enviroinfo.ec@canada.ca](mailto:ec.enviroinfo.ec@canada.ca)

