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GUIDANCE MANUAL FOR ENVIRONMENTAL SITE CHARACTERIZATION IN SUPPORT OF ENVIRONMENTAL AND HUMAN HEALTH RISK ASSESSMENT

VOLUME 3 SUGGESTED OPERATING PROCEDURES

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PREFACE

This manual is one of a series of volumes dedicated to providing guidance on environmental site characterization in support of environmental and human health risk assessment at contaminated sites. The goal of the environmental site characterization guidance is to provide Canadians with a consistent approach to sampling and analyzing complex environmental matrices, such that the data obtained will be representative and of known quality.

The environmental site characterization guidance consists of four volumes:

Volume 1: Guidance Manual
Volume 2: Checklists
Volume 3: Suggested Operating Procedures [this document]
Volume 4: Analytical Methods

The intent of Volume 3, *Suggested Operating Procedures*, is to provide an additional level of sampling guidance detail than is provided in Volume 1 for selected aspects of the site investigation process. There are 17 SOPs in Volume 3:

SOP #1: Borehole Drilling and Installation of Groundwater Monitoring Wells (in overburden)

SOP #2: Soil Sampling

SOP #3: Low-Flow Groundwater Sampling

SOP #4: Soil Gas PROBE Installation

SOP #5: Soil Gas Sampling

SOP #6: Soil Gas Probe Leak Tests

SOP #7: Collection of In Situ Water Quality Measurements

SOP #8: Near Surface Water Discrete Samples by Direct Dip

SOP #9: Surface Water Discrete Samples with Mechanical Collection Devices

SOP #10: Collection of Surface and Subsurface Sediment Discrete Samples

SOP #11: Collection of Sediment Core Samples

SOP #12: Collection of Pore Water Samples

SOP #13: Plant Sampling

SOP #14: Terrestrial Invertebrate Sampling

SOP #15: Benthic Invertebrate Collection and Processing

SOP #16: Fish Sampling

SOP #17: Small Mammal Sampling

SUGGESTED OPERATING PROCEDURE NUMBER 1: BOREHOLE DRILLING AND INSTALLATION OF GROUNDWATER MONITORING WELLS (IN OVERBURDEN)

SCOPE This suggested operating procedure (SOP) provides guidance on the installation of monitoring wells in overburden soils (i.e., into soil overlying bedrock) using conventional drilling and installation methods. Monitoring wells are intended to provide a semi-permanent access point to an aquifer to allow groundwater sampling and aquifer characterization.

WHEN? Monitoring wells are usually designed based on a site conceptual model. Construction materials are then ordered prior to mobilization to the field site. Once at the site, each desired well location is first checked for potential obstructions such as overhead electrical wires and buried utilities. A borehole is then drilled and carefully logged to the target depth. Information gained during drilling is used to modify, if necessary, the initial monitoring well design. This usually entails adjusting the screen length, completion interval and well depth to target specific hydrogeologic features (e.g., aquifer materials) at depth.

WHY? Properly constructed wells should allow representative groundwater samples to be acquired to determine groundwater quality, and to allow hydrogeologic monitoring and/or testing to be conducted (e.g., water-level monitoring, slug tests). Depending on site-specific objectives, monitoring wells may also be used to monitor the presence and thickness of LNAPLs and/or DNAPLs (though caution should be used with the interpretation of the thickness values obtained as they are likely not representative of the actual thickness in the subsurface).

HOW? Requirements for monitoring well installations are very site-specific, and will depend on the soil, rock and groundwater conditions encountered in the field, the goals of the investigation program, and the availability and limitations of drilling equipment and installation materials.

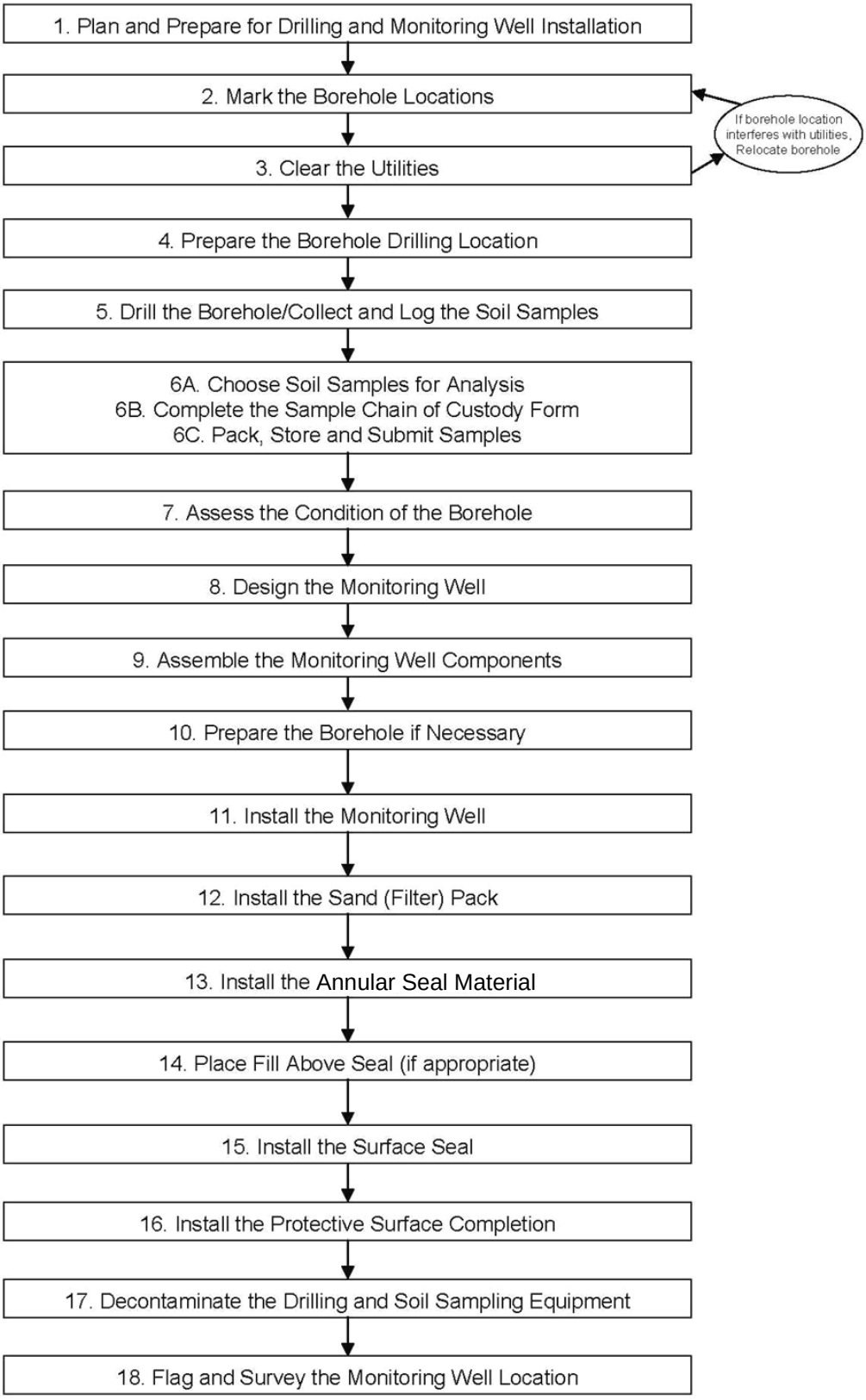
The following are general guidelines for basic installations in boreholes that have been drilled in overburden (soil). Prior to commencing the drilling and well installation program, the conceptual well designs and drilling methods should be identified and reviewed by a qualified person (e.g., geologist, hydrogeologist) to determine whether any deviations from these general guidelines are appropriate.

Monitoring well configurations including well screen length and depth should be determined based on geological and hydrogeological site observations, objectives of the groundwater sampling program and the presence of DNAPL/LNAPL. Well clusters may be installed to monitor several depth intervals within an aquifer. Common monitoring well configurations include (but are not limited to):

- Water table wells, where well screens are completed across the anticipated water table depth. Well screens in such wells should have a maximum saturated screen length of 1.5 metres (m), with unsaturated lengths of up to 1.5 m, to encompass the zone of seasonal water-level fluctuation (especially where floating hydrocarbon product such as gasoline is a concern) and to allow vapour monitoring above the water table; and

- Well screens (up to 1.5 m in length) installed completely below the water table, and across, within or at the base of a water-bearing zone (i.e., just above a low permeability unit).

The steps involved in monitoring well installation are shown below.



Appendix A: Guidelines for Dealing with Bridged Material During Monitoring Well Installation

Essential Information

- ☐ Site plan showing proposed borehole locations and alternates, if necessary
- ☐ Site plan showing existing utilities, if available
- ☐ Project Health and Safety Plan (separate document, beyond scope of this SOP)
- ☐ Waste disposal plan for wastes generated during drilling
- ☐ Geological log of the borehole in which the monitoring well is to be installed
- ☐ Investigation plan detailing chemical parameters of concern, purpose of monitoring well (e.g., sampling, water levels, aquifer testing)

Essential Equipment

- ☐ appropriate health and safety equipment
- ☐ decontamination supplies
- ☐ pre-cleaned sample jars with labels and lids, provided by analytical lab
- ☐ two or three stainless steel trowels
- ☐ wooden stakes, magic marker, flagging tape, and/or spray paint for marking and labelling borehole locations
- ☐ field notebook, pens and pencils
- ☐ camera (and batteries, film or media card)
- ☐ steel measuring tape
- ☐ cooler, packing material and ice packs
- ☐ weighted measuring tape or measuring rods (capable of reaching bottom of borehole). Note: The use of measuring rods is recommended as they can be used to clear bridged material, and are less likely to become entrained in the hole if they are accidentally buried by sloughed material
- ☐ calculator
- ☐ field book, pens, pencils
- ☐ water-level tape (with spare batteries)
- ☐ clean graduated measuring bucket, approximately 22 litres or 5 gallons (for measuring well construction materials and water)
- ☐ decontamination equipment, including:
  biodegradable detergent, rinse water, deionized final rinse water, paper towels, brush, washing container (bucket)
- ☐ monitoring well construction materials (usually provided by the drilling contractor) including casing, screen(s), end caps (top and bottom), sand, sealant material (e.g., bentonite), funnel, cement, potable water supply, and protective surface casings (if applicable), funnel
- ☐ boxes of nitrile gloves
- ☐ lockable j-cap for flush mounts
- ☐ locks

Drilling Contractor Equipment

The drilling contractor should mobilize with:

- ☐ drill rig and soil samplers (including sufficient length of drill stem, casing or auger)
- ☐ appropriate decontamination equipment (e.g., pressure washer or steam cleaner, scrub brushes, and container or tank for wash water)
- ☐ supplies for decontamination station, if appropriate
- ☐ plywood and plastic sheeting (to prevent contamination of ground surface with borehole cuttings)
- ☐ drums with seals and lids for collection of excess borehole cuttings; grease pen or paint for labelling drums
- ☐ asphalt saw or concrete breaker, if applicable
- ☐ sample table (for placing sampler and cores for logging and sub-sampling)

Other Equipment

If soil is likely to contain gasoline, solvents, or other volatile compounds, the following equipment may be required:

- ☐ Portable Photoionization Detector (PID) or Flame ionization Detector (FID) or Combustible Gas Meter
- ☐ Equipment for Soil Vapour Headspace Screening
- ☐ Colourimetric detector tubes

Essential Forms & Plans

- ☐ Borehole Log Form
- ☐ Sample Chain-of-Custody Form
- ☐ Utilities Clearance Report Form
- ☐ Well Installation Log
- ☐ Health & Safety Plan
- ☐ Waste Handling & Disposal Plan

Drill Rig Selection

Boreholes may be drilled using a wide variety of drilling rigs, including sonic, hollow or solid stem auger, air or mud rotary, Becker Hammer or cable tool. Depending on the drill rig used, soil samples may be collected using a split spoon sampler, a continuous corer, or may be carried to the surface using air or drilling mud. Each of these techniques has its distinct advantages and disadvantages and ideal situations for application. Accordingly, the applicability of each drilling technique should be evaluated prior to selecting any one technique to ensure drilling objectives can be met. Much of the following is extracted from SOP# 2048 developed by the US Environmental Protection Agency (March 18, 1996).

Hollow-Stem Auger Drilling

Outside diameters of hollow-stem augers generally range from 6 1/4 inches to 22 inches with corresponding inner diameters ranging from 2 1/4 inches to 13 inches. Auger lengths are usually 5 feet which allows for easy handling. However, lengths of 10 of 20 feet may be used for deeper holes drilled with machines capable of handling the extended lengths. Formation samples can be taken in a number of ways, depending on the accuracy required. Cuttings may suffice for shallow depths but become less representative with depth, particularly below the water table. The most accurate samples are obtained with various coring devices, such as split spoons or Shelby tubes, which can be deployed inside the augers. Continuous cores can also be taken with a thin-walled tube which is inserted into the lowest auger and locked in place. The tube is retracted with a wire line and hoist after the hole has been advanced the length of the auger. A bottom plug in the cutting head or bit prevents cuttings from entering the augers until the first core sample is taken and the plug is knocked out.

In unconsolidated material, the augers serve as a temporary casing and sand-packed wells can be constructed inside the augers and then the augers withdrawn. Well development is usually less difficult than with wells drilled by the mud rotary method because a bentonite drilling fluid is not normally used.

Cable Tool Drilling

Cable tool Drilling is a percussion method in which a bit, attached to a drilling string, is lifted and dropped. The drilling string, consists (bottom to top) of the bit, drill stem, drilling jars, socket, and wire cable. A walking beam on the drilling rig provides the lifting and dropping motion to the wire cable and hence to the drilling string. The repeated action breaks or loosens the formation material, which mixes with formation water or water added to the hole by the operator to form a slurry. The slurry facilitates removal of the cuttings, which are periodically removed from the hole with a bailer. In unconsolidated formations, steel casing must be driven or pushed into the ground as the drilling progresses in order to prevent collapse of the borehole. A hardened steel drive shoe on the bottom end of the casing prevents damage during driving. A well may then be constructed inside the steel casing and the casing pulled back. In consolidated formations the casing may be driven through the weathered zone, and seated in solid rock. The hole below the casing may remain open or may be fitted with a smaller diameter inner casing and screen, depending on the sampling requirements. Depending on formation material, extensive well development may often not be necessary.

Rotary Drilling

Mud Rotary Method

For the mud rotary method, the drill bit is rotated rapidly to cut the formation material and advance the borehole. The drill bit is attached to hollow drilling rods which transfer power from the rig to the bit. In conventional rotary drilling, cuttings are removed by pumping drilling fluid (water or water mixed with bentonite or other additives) down through the drill rods, and bit, and up the annulus between the borehole and the drill rods. The drilling fluid flows into a mud pit where the cuttings settle out and then is pumped back down the drill rods. The drilling fluid also cools the bit and prevents the borehole from collapsing in unconsolidated formations.

Sampling may be done from the cuttings but samples are generally mixed and the amount of fine material may not be accurately represented. Coring may be done through the drill rods and bit if a coring bit (with a centre opening big enough to allow passage of the coring tube) is used. When drilling unconsolidated formations, a temporary surface or shallow casing may have to be installed in order to prevent cross-contamination, hole collapse, or wall erosion. The casing (riser pipe), screen, and gravel pack are usually installed in the open hole or through the surface casing. Once the well is constructed, extensive well development may be necessary in order to remove the drilling fluids from the formation.

Air Rotary Method

The air rotary method uses air as the drilling fluid. Air is forced down the drill rods by an air compressor, escapes out to the bit and returns to the surface in the annular space between the hole wall and the drill string. Cuttings are moved out of the hole by the ascending air and collect around the rig. Cuttings are mixed and may not always be representative of the depth currently being drilled. In the conventional air rotary method, the drill string operates in a manner similar to that described for the mud rotary system. In a “hammer” or “down-the-hole” air rotary method, the bit is pneumatically driven rapidly against the rock in short strokes while the drilling string slowly rotates. The use of air rotary methods are generally limited to consolidated and semi-consolidated formations. Casing is often used in semi-consolidated formations and through the weathered portion of consolidated formations to prevent hole collapse. In environmental work, the air supply must be filtered to prevent introduction of contamination into the borehole.

Rotary Sonic Drilling

The rotary sonic drilling method uses an oscillator or head with two eccentric weights or rollers driven by hydraulic motors to generate high forces in a rotating drill pipe. The frequency of vibration of the drill bit or core barrel can be varied between about 50 and 120 cycles per second to optimize drilling rates and penetration of subsurface materials. A dual string assembly allows advancement of an outer casing, with the inner casing or core barrel used to collect samples. During drilling, the inner casing or core barrel is advanced several metres ahead of the outer casing, and then the outer casing is advanced over the inner drill

rods and core barrel, allowing the core to be extracted. The core sample can be taken directly from the core barrel by extruding it into a plastic sleeve, stainless steel tray or other suitable receptor. Monitoring wells can be installed by placing the well assembly down the cased hole, and vibrating and extracting the outer casing while placing sand and bentonite sealant materials. A main advantage of sonic drilling is the ability to obtain large diameter (up to 250 mm) continuous core samples without the use of air or drilling fluids. The drill can penetrate a variety of materials including boulders, wood, concrete and other construction debris without meeting refusal.

1. Planning and Preparing for Drilling and Monitoring Well Installation

☐ Review the drilling and sampling plan, and any relevant information pertaining to subsurface conditions at the planned drilling locations, including soil and groundwater conditions, type, degree and extent of contamination. See Drill Rig Selection.

☐ Determine the appropriate type of drilling rig, soil sampler(s), and well installations for the conditions expected.

☐ Plan the design of each monitoring well installation. The design should be based on a conceptual site model, consistent with the objectives of the investigation, and planned well in advance of commencing the field program.

☐ Obtain and review available information on subsurface and above-grade utilities.

☐ Determine suitable alternative borehole locations in the event that planned locations conflict with on-site uses or utilities.

☐ Prepare a health and safety plan.

☐ Prepare a waste management plan for soil or rock cuttings and other waste materials generated during drilling.

☐ Schedule and book the drilling contractor. Confirm with the drilling contractor the type of soil anticipated, the depths of the planned installations, and the estimated quantities and types of installation materials that should be brought to the site, including (but not necessarily limited to):

- type (material, wall thickness, threaded, *etc*.) and length of casing;
- type (material, wall thickness and slot size) and length of screen;
- end caps or j-plugs for the monitoring well installation;
- sand and sealing material (e.g., bentonite chips);
- cement;

- flush-mount or stick-up protective surface completions;
- measuring rods (preferable) or weighted measuring tape(s) of an appropriate length of steel measuring tape;
- a funnel (with a 2 to 4 centimetre [cm] opening, for controlling the rate at which sand and sealant material is poured into the installation); and
- a supply of potable water (to be provided or arranged for by the drilling contractor)

☐ Confirm that the well casing and screen lengths will be delivered to site wrapped in plastic to prevent cross-contamination.

☐ Ensure that sufficient extra materials (e.g., casing, screen, sand) and supplies are brought to the site by the contractor to accommodate uncertainties in the borehole depths, unforeseen conditions, loss or breakage, *etc*. A general rule-of-thumb is to have on-hand an extra 20% of these materials.

ON-SITE:

☐ Make sure that the sampling equipment is clean (decontaminated).

☐ If possible, and if information regarding the expected degree of contamination is available, plan to drill the initial borehole(s) in the area of least contamination, and proceed to more contaminated zones.

☐ Before drilling commences, check the length of the measuring tape or rods used by the contractor, to confirm whether a segment has been removed (or added).

☐ Review the project health and safety plan.

☐ Throughout the installation process, keep track of the quantities of materials used during the monitoring well construction, the conditions encountered, the duration of the installation process, causes and duration of down-time, *etc*. Record such observations in the field book, so that drilling costs can later be checked.

☐ All measurements of depth should be taken relative to ground surface or a fixed benchmark immediately adjacent to the hole.

☐ The drilling contractor is generally responsible for the installation, measurements, *etc*. Check any measurements that seem questionable.

2. Marking the Borehole Locations

☐ If necessary, use a measuring tape or wheel to scale off the drilling locations relative to fixed benchmarks.

3. Clearing the Utilities

- ☐ The owner, drilling contractor and/or utility locate contractor are typically responsible for clearing utilities (ensuring that drilling will not compromise underground or overhead utilities) with on-site oversight by the investigator to ensure alternative monitoring locations are cleared, if required.
- ☐ Clear the subsurface and above-grade utilities (e.g., overhead wires) prior to commencing drilling.
- ☐ Ensure that there will be sufficient clearance between the drill rig tower and the nearest overhead lines. Check with local power authority for minimum safe work distances.
- ☐ In some cases, additional precautionary measures such as geophysical techniques, hand excavation or vac truck hole excavation to expose utilities may be warranted, prior to commencing drilling.
- ☐ If the planned borehole locations interfere with the utilities, re-locate the borehole(s) to alternative locations as appropriate.

4. Preparing the Drilling Location

- ☐ Inspect the drill stem, auger flights or casing before proceeding and confirm (with the rig operator) whether it has been decontaminated; instruct the operator to decontaminate the equipment if necessary.
- ☐ If necessary, concrete at ground surface can be broken using a concrete breaker, and asphalt can be cut using an asphalt saw or by coring or drilling.
- ☐ If contamination of the ground surface is a concern (for example, in public areas, or where potentially hazardous materials may be encountered), place plastic and/or plywood sheeting adjacent to the planned drilling location, and place the drill cuttings on the sheeting. Alternatively, a hole can be cut in the centre of the plywood and drilling can proceed through the hole.
- ☐ If the drill rig is situated in an area accessible to the public or site workers, restrict access as appropriate using barriers, flagging and/or pylons.
- ☐ Set up a sample table adjacent to the drilling location to facilitate the collection and logging of soil samples.

5. Drilling the Borehole/Collecting and Logging the Soil Samples

- ☐ Commence drilling of the borehole. As the drilling depth increases, samples brought to surface should be examined, logged, and photographed, and representative sub-samples collected for screening and/or laboratory analysis, as specified in the project work plan.
- ☐ Coring drill rigs and sonic-type rigs generate a relatively continuous core from which sub-samples may be collected for analysis.

- ☐ Where split-spoon samplers (or other samplers driven into the soil ahead of the drill casing or auger) are employed, as a general guideline, soil samples should be collected at least every 1.5 metres. Smaller intervals should be used if the soil conditions are relatively variable. Appropriate sub-samples can then be collected from the split-spoon sample for chemical analysis.

- ☐ Sub-samples should be collected from the intervals, depths and/or types of soil specified in the soil sampling program. The number and location of samples to be collected will vary depending on the purpose and objectives of the sampling program.

- ☐ Significant quantities of soil or rock cuttings are brought to the surface when using solid- or hollow-stem auger, air or wet rotary, and Becker Hammer methods.

- ☐ Soil cuttings should be placed in separate cuttings piles according to the presence or absence of contamination. By doing so, the effort and cost of managing, treating and/or disposing of the soil can be minimized. Eventually the excess cuttings may be sampled and/or drummed for disposal in accordance with the waste management plan, which should be prepared prior to undertaking the drilling program.

- ☐ Fill in the borehole log (see the example in SOP #1) as completely as possible. All depths should be measured as depth below ground surface. Information should include, but not necessarily be limited to:

    - the file identification, site identification and borehole number;

    - the type of drill rig used (e.g., hollow stem auger, air rotary), the casing or auger diameter, bit type (if applicable), and rate of advance;

    - the type of sample collected (e.g., split spoon, grab sample - if carried up by air or taken from a solid-stem auger);

    - the depth from which the samples were collected (top and bottom of depth range);

    - the top and bottom depth and a description of any observed fill zone or stratigraphic unit; distinct boundaries should be indicated as a solid line at the appropriate depth;

    - a description of each sample;

    - the percentage of sample recovered (e.g., 50% if only 9 inches of sample was present in a split spoon sampler driven 18 inches); the drilling and sampling method employed may significantly affect the sample recovery;

    - the sample number;

    - if applicable, the moisture condition of the sampler immediately on recovery (e.g., water on the split spoon);

- any significant groundwater observation;

☐ **Collection of samples for VOLATILE ANALYSES**

- Most soil sampling methods, and the collection of sub-samples, result in the loss of volatile chemical compounds.

- Samples collected from cuttings brought up to the ground surface using air or wet rotary or Becker Hammer methods are particularly unsuitable for analysis of volatile chemical parameters.

- If **a sonic** drill rig is used, sample cores are generally bagged in plastic as they are recovered. Note that the drill stem may heat up as it advances into the ground, potentially volatilizing contaminants present in the soil. Screening of the core may be conducted by puncturing holes in the plastic to take organic vapour readings (e.g., using and photo ionization detector (PID)) at several locations along the length of the core. Sub-samples may be desirable at locations having elevated PID readings, and may be collected by scraping smeared soil from the outside of the core, and quickly pushing a coring device into the core to collect a relatively unexposed sub-sample. The core, approx. 5 g, is then extruded into a laboratory provided vial containing methanol or aqueous sodium bisulphate preservative, and the vial is resealed. Complete the sample label and place the sample in a cooler with cold packs.

- If a **split spoon** sampler is used, a sample for volatile analyses may be collected by placing a clean brass tube into the split spoon prior to driving the sampler. Upon recovery of the sampler, quickly remove the filled tube, wrap it at both ends using aluminum foil and tape the foil in place. Label the sample tube and place in a cooler with cold packs.

☐ Sub-samples for volatile organic compounds (VOCs) may be collected from the split spoon samples using special hermetic sub-sampling devices such as the En Core™ sampler, which is designed to minimize VOC losses. Note that the hold time for hermetic sampling devices is 48 hr.

☐ Sub-samples collected from split spoon samples may also be submitted for VOC analysis but the results will be biased low, due to the significant loss of volatiles.

☐ **Collection of soil sub-samples for NON-VOLATILE or SEMI-VOLATILE analyses:**

- Collect sub-samples from a split spoon sample, auger sample or sonic core using a clean trowel, after first removing any soil that may have smeared on the outside of the sample. Place the sub-sample in the appropriate jar, fasten the lid firmly and the complete the label.

- If Becker Hammer, air or wet rotary methods are used, a sub-sample may be collected by using a sieve or screen and catching some of the material brought to surface. The fine fraction may be limited or absent, and, with Becker Hammer, coarser material may be

pulverized, so the sub-samples not be representative. Place the sub-sample in the appropriate jar, fasten the lid firmly and complete the label. Samples from Becker Hammer, air or wet rotary methods will be highly disturbed and are not considered suitable for environmental characterization purposes.

☐ Place all samples in a cooler with ice packs as appropriate and maintain at $\leq 10^{o}$C in transit (but not frozen) and prior to analysis.

☐ If used, decontaminate the sampler between sampling depths.

6A. Choosing Samples for Analysis

☐ Soil samples should be selected to meet the objectives of the investigation.

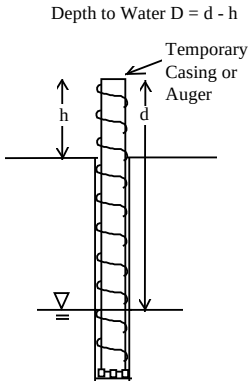
6B. Completing the Sample Chain-of-Custody Form

☐ Complete the sample chain-of-custody form with the required information; retain a copy and submit the completed form with the samples to the analytical laboratory.

6C. Packing, Storing and Submitting the Samples

☐ Ensure that the samples are delivered to the laboratory, and the laboratory has sufficient time to start the analysis, within the recommended holding time. Sometimes the delivery of the samples at the end of the day or work week can result in delays in commencing the analysis, so keep this in mind when shipping or transporting the samples.

7A. Assessing the Condition of the Borehole



☐ Once the borehole drilling is complete, measure the depth of the hole with the rods or weighted tape, either through the open drill stem, augers or temporary casing, or in the open hole (if solid stem auger flights are removed). Record the measurement in the field notes.

NOTE: If using a weighted tape when measuring the depth of the hole (and especially when adding sand pack or seal material to the well), the drilling contractor should jig the tape up and down, to assist in identifying the bottom of the borehole, and to free the tape if material settles onto and traps the tape.

☐ Measure the depth to water in the borehole and record the measurement in the field book. Include the time elapsed since drilling concluded, and the time the measurement was taken.

☐ The water level in the borehole may be significantly lower than actual water table if:

- the measurement is taken soon after drilling finished;

- the formation contains clay or silt (low permeability); and/or
- air rotary drilling was employed (can temporarily force water away from the borehole).

☐ The water level in the borehole may be higher than the actual water table due to the introduction of water in the installation casing or borehole to overcome upward soil pressure which sometimes causes soil formation material to heave up into the borehole.

7B. Borehole Abandonment

☐ Boreholes that will not be completed with a monitoring well should be properly abandoned after the condition of the borehole has been recorded. The borehole should be abandoned by sealing the entire length of the borehole with a suitable grout (e.g., bentonite or a cement-bentonite mixture). A tremie pipe may be necessary to place the grout to ensure that the full length of the borehole is sealed.

8. Designing the Monitoring Well

☐ Consult with a qualified person (e.g. geologist/hydrogeologist) to determine the final design for the monitoring well (including the length of the screen section, the interval for placement of the screen and the sand pack) based on the geological conditions encountered, the water-level and the intent of the investigation.

☐ Sketch the casing design in notes and list the number and types of components to be used.

9. Assembling the Monitoring Well Components

☐ Plywood or plastic sheeting should be placed in a suitable location next to the borehole, and the monitoring well components placed on the sheeting to prevent cross-contamination from the ground surface.

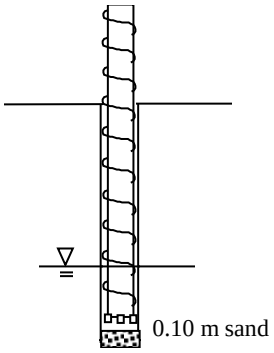
☐ Ensure that drilling contractor has clean gloves or puts on nitrile gloves during assembly of the monitoring well.

☐ The casing, screen and bottom cap components should be assembled. The length of the assembly and total components should then be measured and the measurements recorded.

IMPORTANT: Check that all o-ring seals or threaded sections between the well components are in good condition, to ensure proper hydraulic seals.

☐ Compare the length of the assembly to the required design length depth. Make corrections to the assembly as warranted.

10. Preparing the Borehole (if necessary)



Volume
$V = L \times \pi (0.5 \times D)^2$
where
D = outside diameter of augers, temporary casing

L = length of borehole to be filled

- ☐ If the well assembly is designed to rest on the bottom of the borehole, a minimum of 0.10 m of sand (filter pack material) should be added to the bottom of the borehole before installing the well assembly.

- ☐ If the bottom of the hole is more than 0.5 m below the bottom of the screen design depth (and the design does not call for an open hole to be left below this), the borehole section should be filled to the required depth in the following manner:

  - Calculate the expected volume of material required to fill the borehole section to the correct depth. Use the outside diameter of the temporary casing or auger flights for the calculation (as the temporary casing or drill stem will be pulled to above this section during filling).

  - The required volume of bentonite and clean fill should be mixed using a mix ratio of 1:3 bentonite chips to clean fill.

  - Leave the weighted tape or rods in the hole, but jig the tape/rods up and down while adding the mixed material.

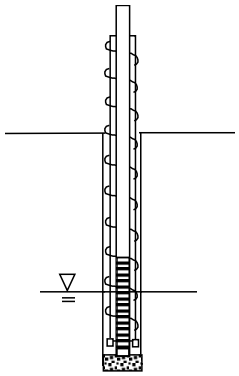
IMPORTANT: Movement of the tape or rod can prevent bridging by agitating any material that begins to adhere to the walls of the borehole, temporary casing and/or well assembly (causing it to loosen and fall).

If bridging occurs, see Appendix A at the end of this procedure.

- The mixed fill should be slowly poured into the open hole or temporary casing.

- Compare the volume actually required to fill the section of hole with expected volume. If more material was required than estimated, the borehole is oversized (e.g., the walls may have collapsed or sloughed during drilling).

11. Installing the Monitoring Well Assembly

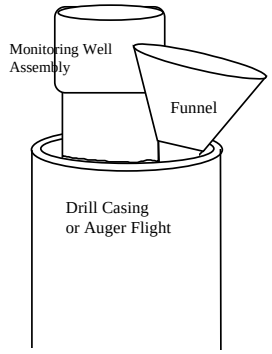


- ☐ The well assembly (casing, screen and bottom cap) should be lowered into the borehole in the correct order (bottom cap in first) until it reaches the bottom of the borehole and sits on the 0.10 m of filter pack material added (or is at design depth if it is to be suspended in well).
- ☐ The well assembly should be clamped in place if it is to be suspended in the hole.
- ☐ Measure the height of the well casing above the ground (or other fixed datum such as rig deck, *etc.*) to the nearest cm and record it in the installation log.

MPORTANT: Make sure the top cap is placed firmly on the well assembly to prevent material from entering during installation of the sand pack and seal.

12. Installing the Sand Pack (or Filter Pack)

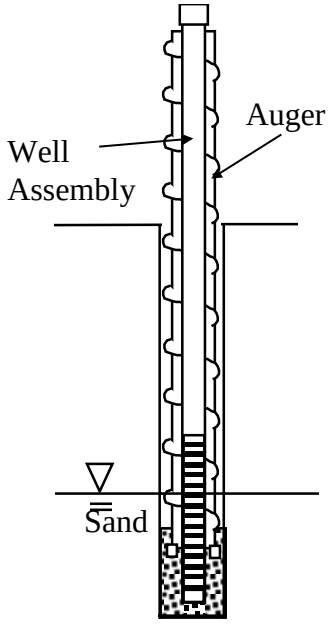
☐ Calculate the volume of sand material needed to fill the filter pack section.



- ☐ Ensure that the well assembly is centred in the borehole (PVC centralizers may be used to ensure that the well remains centred within the borehole during installation.)
- ☐ Measure the depth to the bottom of the hole again and record the measurement.
- ☐ The measuring tape or rods should be left in the hole while adding the filter sand, but kept opposite the pouring side.
- ☐ Using a funnel to control the rate and location of pouring, the filter sand should be poured slowly into annular space while jigging the tape or rods up and down.

IMPORTANT: Do not fill the annular space with more than 0.5 m of sand pack material at a time, without partially extracting the temporary casing/drill stem, as this may cause bridging.

If bridging occurs, see Appendix A at end of this procedure.



A. For Open Hole Installation

- ☐ When the sand has reached the desired level (as a general rule, the top of the sand pack should be about 0.3 m higher than top of screen), measure the depth to the sand and record it on the installation log.

B. For Temporarily Cased Installation

I) Stop adding the sand when it is about 0.50 m above the bottom of the temporary casing and drill stem.

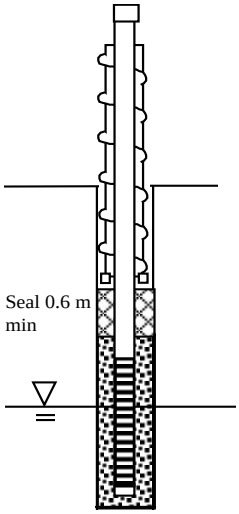
II) Measure and record the depth to the sand.

III) The temporary casing and drill stem should be pulled up (about 0.5 metres) to expose a new section of open borehole. The sand should fall slightly as the open hole becomes exposed (as indicated by the increased depth to the sand).

IV) Measure and record the new depth to the sand. Compare it to the above measurement to ensure that the sand pack is not being pulled up with the casing/drill stem.

V) Sand should be added and the temporary casing /drill stem pulled up in stages until the design depth for the sand pack has been reached.

13. Installing the Annular Seal Material



- ☐ The seal material should be placed in same manner as the sand pack (i.e., added in stages as the casing is pulled up), but should be **added more slowly to prevent bridging.**
- ☐ Calculate the volume of seal material required to fill design length.
- ☐ **The seal length should be a minimum of 0.6 m**. However, the seal length may be varied depending on the formation characteristics and the available space in the borehole. For example, if the top of the sand pack is close to the surface, or if a multiple completion is to be installed in the borehole (i.e., monitoring well and gas probe, or nested piezometers), then there may not be sufficient room to install a 0.6 m seal).

IMPORTANT: Seal material typically consists of bentonite chips or pellets. Bentonite (a clay mineral) becomes sticky when wet; therefore, bridging is a very common problem when placing seal materials. In addition, once the material has bridged, it may begin to hydrate and swell, making the problem worse.

☐ Measure and record final depth to the top of the seal material when finished.

14. Placing Fill Above the Seal (If Appropriate)

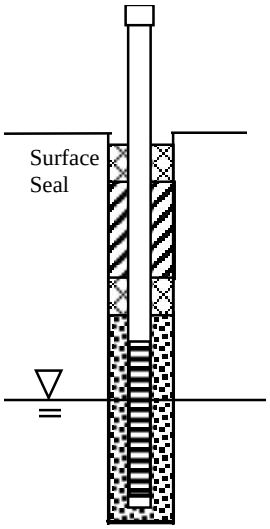
☐ If the top of the seal does not correspond to the ground surface, the remainder of the annular space should be sealed to within 0.6 m of ground surface.

☐ The strong preference of this SOP for Backfill is bentonite grout slurry such as Volclay™ to prevent the borehole from becoming a vertical conduit for potential contaminant migration. Alternative and less desirable seals may be appropriate, depending on local availability and site conditions. Such seals may include backfilling with bentonite pellets or chips that are hydrated with water following placement, or mixtures of bentonite powders with non-contaminated granular fills (e.g., sand) in proportions of at least one part bentonite to five parts fill.

☐ Place fill material in the same manner as the sand pack.

IMPORTANT: If installing a monitoring well in a low permeability formation (e.g., clay, glacial till, unfractured rock, *etc.*), following installation of the sand pack and seal, the entire borehole annulus should be grouted to surface using a bentonite slurry to prevent the well from becoming a vertical conduit for surface water.

15. Installing the Surface Seal



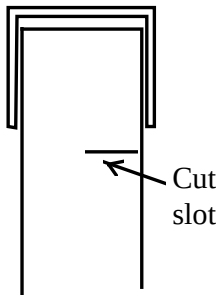
☐ The upper 0.6 m of the borehole should be sealed to prevent surface water from entering the borehole. See Step 16 below if a protective surface completion is to be installed.

16. Installing the Protective Surface Completion

☐ An appropriate surface completion should be selected based on the site conditions. The surface completion may consist of:

- a lockable stickup completion, which is easiest to locate (e.g., in vegetated areas) and is most suitable at sites where there is a low probability of damage due to vehicle traffic; or
- a flush-mount surface completion, which is installed flush with the ground surface and usually has a bolted-down cover.

A. Lockable Stickup Completion



☐ The length of the monitoring well casing should be measured and cut (if required) to allow it to fit inside the stickup assembly. Leave room for the monitoring well cap.

☐ The casing should be cut so that top cap is no more than 2 to 5 cm below the cover to allow access for a hand to remove the cap.

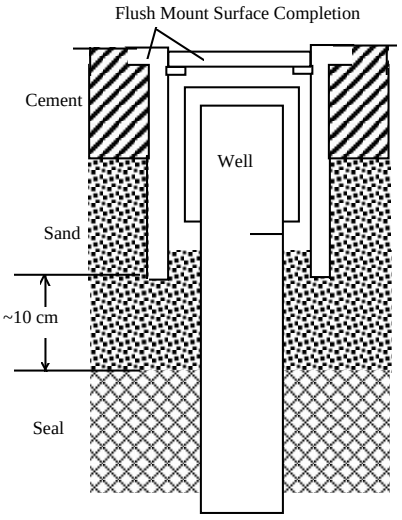
☐ The top cap or J-plug should be placed firmly on the casing, but not forced as it will be very difficult to remove. If the fit is too tight, the outside of the well casing should be sanded until a snug fit is achieved.

☐ The stickup assembly should secured using cement.

☐ A ventilation hole or slot should be drilled or cut into the top of the well casing (below the level of the top cap) to allow equilibration of the well casing air volume with changing atmospheric (barometric) conditions when the top cap is in place.

☐ The stickup completion should be labelled (using a metal tag or paint stick) with the monitoring well identification, date of completion, depth of well casing, and screened interval.

B. Flush Mount Surface Completion



- ☐ The monitoring well casing should be measured and cut (if required) to fit inside the flush mount assembly.
- ☐ The well casing should be cut so that the top cap is approximately 2 to 5 cm below cover to allow room for the top cap.
- ☐ The top cap or J-plug should be placed but not forced onto the well casing. If the fit is too tight, the outside of the well casing should be sanded until snug fit is achieved.
- ☐ The flush mount completion should be placed in borehole; the seal material should be 0.1 m below the bottom lip - remove seal material if necessary.
- ☐ Filter sand should be added so that it fills part of the flush mount interior, but is below the top of the well casing.

IMPORTANT: The purpose of the filter sand is to provide drainage for any surface water that enters the flush mount assembly. The water will be able to drain out the bottom of the assembly via the filter sand and into the surrounding soil. If the assembly fills with water, it may flow into the well through the ventilation hole and alter the water chemistry (and possibly introduce contaminants) in the monitoring well.

- ☐ The outside annulus of the flush mount completion assembly should be filled with cement to secure it in place.
- ☐ The surface completion should be labelled (using metal tag or paint stick) with monitoring well identification, date of completion, depth of well casing, and screened interval. Felt marker fades rapidly and hence should not be used.

17. Flag and Survey the Monitoring Well Location

- ☐ Mark the location of the monitoring well on the site plan and measure off the distance from the well to two or three fixed reference points or monuments. Completions should be marked, using stakes, flagging, *etc*. so that they can be located easily.
- ☐ Once all of the boreholes have been drilled and monitoring wells installed, survey in the elevations of the ground surface and the top of well casing at each installation.

APPENDIX A
Guidelines for Dealing with Bridged Material During Monitoring Well Installation

1. Attempt to jig tape or rods up and down.

IMPORTANT: If the tape or rods can still be moved up and down, but cannot be pulled up past the bridge, a void exists in the annulus. If the tape or rods cannot be moved, the material added to the annulus may simply be higher than expected (due to incorrect calculations, or volume measurements) and the end of the tape or rods may have been covered. In this case, gently apply an increasing pull on the tape or rods, but do not pull hard enough to break the tape or rods.

2. Attempt to remove the tape or rods from the hole.

3. If the tape or rods can be removed, measure and record the depth to the bridge. If they cannot be removed, take the measurement with a second measuring device (if available).

4. Estimate the thickness of the bridged material; it is often possible to detect the bottom of the bridge when removing a weighted tape.

5. If the bridge is near the bottom of the open well, check the length of the temporary casing in the hole to make sure the filter pack or seal material is not present inside the casing (i.e., material may not be bridged, simply too much has been added and sand pack is now up too high).

6. Use the tape or rods to tap or push on the bridge to dislodge the bridged material (measure depth).

7. Move the well assembly back and forth in an attempt to dislodge the bridged material (measure depth).

8. Check with the Site Investigation Coordinator (Project Manager) before proceeding with this step:

- If the material still cannot be dislodged, as a last resort, pour 5 to 10 litres of clean (potable) water down the annulus.
- Record the volume of water used and ensure that a sample of the water is collected for analysis of the same chemical parameters as the groundwater to be sampled at that location.
- If water has been added to the installation, during well development an attempt should be made to purge a volume at least three times the volume of water added.

Guidelines for Dealing with Bridged Material During Installation (continued)

9. Measure the depth again to determine whether the bridge has moved.

10. If necessary, repeat steps 5 to 7 and measure the depth to the bridge.

11. If the bridge has still not moved, measure and record the distance from the top of the well assembly to ground surface, to nearest cm.

12. Pull the temporary casing/drill stem up 0.20 m.

13. Measure the height of the top of well assembly to determine whether it was pulled up with the temporary casing/drill stem. If this is the case, try to push the well assembly back down to the original depth.

14. If necessary, repeat steps 5 to 8.

15. If the bridge is still in place, attempt to pull (recover) the well assembly from the hole.

16. If the well assembly can be removed from the hole, disassemble (if required due to length) and decontaminate it, and place the well components to the side on the clean plywood or plastic sheeting. If the casing cannot be removed properly abandon the hole.

17. Measure the depth of the hole to determine whether material is still bridged.

18. If material is still stuck in the drill casing/stem, have the contractor clean it out using drill rods.

19. Re-drill the hole to the design depth and measure.

20. Re-start the installation.

REFERENCES

USEPA, 1996. Standard Operating Procedures 2048 - Monitor Well Installation.

FIELD BOREHOLE LOG

Boring Number BH07-1 | Depth 0 to 21 ft. | Sheet 1 of 1
Project XYZ DEVELOPMENT | Job No. 07-1234-5618 | Date MARCH 14, 2007
Location CANADA | Elevation 123.4 m | Datum GEODETIC
Casing HOLLOW STEM AUGER | Casing Hammer, wt N/A drop N/A
Weather OVERCAST / RAIN / 5°C | Sampler Hammer, wt 140 lb. drop 30 inch
Drill Rig MARL 10 AUGER | Driller JOHN DOE | Engineer TOM SMITH

DEPTH ELEV. (m / ft.)	SOIL STRATIGRAPHY	BLOWS PER FOOT	DEPTH SCALE (ft.)	COMPLETION	SAMPLES Cond.	Type	No.	Recov	PID (ppm)	SAMPLE DESCRIPTION & BORING NOTES
(ft.)	GROUND LEVEL		(ft.)							COMMENCED WORK AT 8:00AM. MOVED RIG AND SET UP TO 8:30AM. COMMENCED DRILLING AT 8:45AM
0.15 (0.5)	TOPSOIL	4				2" DO	1	12/18	0.0	Sa1 (SCN 87654-01) 0'-1½' – brown, moist, m-c sand trace to some gravel and debris. Debris as fragments of brick, concrete <1" Ø and organics. Upper 6" is black organic topsoil with roots. PID = 0.0 ppm, no stain, no odour
	Compact, moist, brown, medium to coarse SAND, trace to some gravel, trace debris (brick and concrete fragments <1" dia.) [FILL]	6	1							
		8								
		7	2							
		7	3			2" DO	2	18/18	0.0	
		6	4							Sa2 (SCN 87654-02) 2½'-4' – brown, moist, m-c sand trace to some gravel, trace debris (brick, concrete) no stain, no odour PID = 0.0 ppm
		5	5							
	– Weak hydrocarbon like odour at 5 feet bgs.	8				2" DO	3	6/18	38	
		8	6							Drilled to 5' – light to moderate auger resistance
	– Sheen on soil at 7.5 ft. bgs.		7							
		5	8							Sa3 (SCN 87654-03) 5'-6½' – brown, moist, m-c sand trace to some gravel, trace debris (concrete, brick, 4" Ø angular) PID = 38 ppm, weak hydrocarbon-like odour noted
		5				2" DO		0/18		
		6	9							
		6								
3.05 (10)		5	10			2" DO	4 5	18/18	82	7½'-9' – sample lost
	Very dense, damp, grey-brown, clayey SILT, trace fine gravel [NATIVE]	20								Sa4 (SCN 87654-04) 9-10' (upper 12" of DO sample) brown, moist, m-c sand, trace to some gravel, trace debris (concrete, black). weak HC-like odour, PID = 82 ppm
			11							Sa5 (SCN 87654-05) 10'-10½' (lower 6" of DO sample) dense, grey-brown, clayey silt, trace fine gravel
			12							Spoon noted to be wet at tip.
		25	13			2" DO	6	18/18	3	
	– Water table estimated at 10 ft. based on water inflow	25								Drilled to 12½' considerable auger resistance, Inner rod wet at base
		30	14							
			15							
4.73 (15.5)	Very dense, dry, dark grey, clayey SILT, trace fine gravel [NATIVE]		16							
		45 60 for 6"	17			2" DO	7	6/12	0.0	Sa6 (SCN 87654-06) 12½'-14': damp, grey-brown, clayey silt, trace fine gravel. no stain no odour. PID = 3 ppm
			18							
			19							Strong auger resistance. Driller inferred that hit a stratigraphic contact at 15.5 ft +/-
		36	20			2" DO	8 9	18/18	0.0	Sa7 (SCN 87654-07) 16'-17': dry dark grey clayey silt, trace fine gravel. no stain no odour, no debris, PID = 0.0 ppm
7.25 (20.5)		40								
7 (21)	Slightly weathered, thinly bedded, grey, fine SILT, faintly porous, medium strong SILTSTONE	40	21							Strong auger resistance. Water in hole
			22							Sa8 (SCN 87654-08) 19½-20½: dry, dark grey clayey silt, trace fine gravel. no stain, odour, debris; PID = 0. (upper 12" of spoon)
			23							Sa9 (SCN 87654-09) 20½-21: Sl. weathered, thin bed, grey, sl. porous, M. strong, SILTSTONE. N. stain, no odour
	End of Hole		24							End of Hole at 21', Install monitoring well.
			25							Note: Used dry headspace to measure TOV concentration PID had 10.6 eV lamp.
										Completion – Screen (2" PVC) 12½' → 7½'; Bentonite 21' → 13' Sand (10/20) 13' → 6½'; Bentonite 6½' – 2'; Concrete 2'-0' steel flush mount cover at surface
					Notes	Encountered rock at 20.5 ft. Impractical to advance further with augers				End at 12:30 pm

SAMPLE CONDITION
DISTURBED
FAIR
GOOD
LOST

SAMPLE TYPES
A.S. - Auger sample
C.S. - Chunk sample
D.O. - Drive open
R.C. - Rock core
S.T. - Slotted tube
T.O. - Thin walled, open
T.P. - Thin walled, piston
W.S. - Wash sample

ABBREVIATIONS
Wh - Weight, hammer
Ph - Pressure, hydraulic
Pm - Pressure, manual
V - In-situ vane shear test

SPECIAL NOTES: (water conditions etc.)
Time: 8:45 – 12:30 Depth of Hole: 21 ft
Hrs. Productive: 3:45 Depth of Casing: N/A
Hrs. Delayed: 0 Depth to Water: 10 ft

SUGGESTED OPERATING PROCEDURE NUMBER 2: SOIL SAMPLING

SCOPE This suggested operating procedure (SOP) provides general direction and guidance for the collection of representative soil samples for chemical characterization purposes. There are numerous methods to collect soil samples, and specific procedures may apply to each method in the acquisition of the soil samples. However, detailed descriptions of the specific procedures are beyond the scope of this technical procedure. Additional information on soil sampling is provided in Volume 1 of the *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as "*Guidance Manual"*).

APPLICATION Collection of soil samples for the purposes of chemical testing.

WHEN? *In-situ* soil samples are typically collected at three steps in the assessment and remediation of contaminated sites:

1. At the initial testing program stage to assess potential impacts to soils;
2. As part of the detailed testing program to delineate contamination; and,
3. At the confirmatory sampling step to assess the effectiveness of remedial measures in reducing chemical concentrations in the soils.

Ex-situ soil samples are typically collected for assessment of the bulk chemical concentration in a given volume of soil (e.g., stockpile sampling).

WHY? Soil sampling may be conducted to identify and define the limits of contamination *in-situ*, to assessing the need for further delineation, and to determine remediation requirements. Soil sampling may also be conducted to assess the bulk chemical quality of soils excavated from a site for the purposes of defining treatment requirements and/or verification of concentrations to facilitate appropriate decisions for off-site disposal.

HOW? Soil samples may be collected using a variety of sampling methods. The choice of sampling method depends primarily on: 1) the type of soil being sampled (i.e., geology, soil properties); 2) the analyses to be performed, 3) the sampling depth (e.g., shallow versus deep), and 4) the type and/or purpose of the sample (i.e., discrete versus composite, investigatory versus confirmatory).

It may also be required to screen the soil samples for the purposes of assessing the presence of volatile organic chemicals, or specific chemical constituents (i.e., using field test kits). Soil vapour headspace tests may be conducted using wet or dry headspace techniques and organic vapour monitoring field instruments (i.e., photoionization detector (PID), flame ionization detector (FID) or combustible

gas detector). If field screening is to be conducted, a larger sample volume may be required.

TYPES ***In-Situ* Discrete Samples:** The definition of an *in-situ* discrete sample is:

A sample obtained from a single sampling point or location, and is considered normally as being taken from a single use of the sampling tool used to obtain the sample.

Composite Samples: A composite sample is:

A sample obtained by combining a number of discrete samples into one homogenized sample in order to represent the average concentrations of the area and volume of material over which the combined discrete samples were taken.

Composite sampling should never be conducted where volatile or semi-volatile analyses are required, as the compositing process can result in losses of volatile constituents. Composite sampling has the potential for diluting contaminant concentrations; see Volume 1 section 5.3.4 for more information on requirements for use of composite sampling.

COLLECTION An important consideration in soil sampling is how the soil samples are to be collected and handled. A good rule-of-thumb to consider is that each handling step between the acquisition of the *in-situ* soil to placing and sealing the sample in an appropriate container will likely add some degree of error or bias to the analytical result. Consequently, minimizing the handling and disturbance of the soil is critical at the sampling stage.

The use of clean and appropriate sampling equipment is also essential to minimize potential sampling bias. Wherever possible, clean equipment (either appropriately decontaminated equipment, or new, disposable equipment) should be utilized. Certain chemicals may also react with certain types of material, and therefore the composition of the sampling equipment needs to be considered prior to sampling (e.g., felt pens that are not water soluble contain chemicals that could impact samples analyzed for volatile organic compounds (VOCs). Clean disposable nitrile gloves are recommended with replacement between samples. Good quality stainless steel equipment is recommended for sampling, as it is sturdy, minimally reactive and can be decontaminated through conventional, field means (i.e., scrubbing to remove gross contamination, washing with laboratory-grade detergent (Alconox™ or Liquinox™, for example) or solvents (as necessary), and rinsing with distilled water. However, nickel has been found to leach from stainless steel. If this is a PCOC at a site, sample contact with stainless steel should be minimized.

Since laboratories can detect very low concentrations of VOCs, it is important for smokers and perfume wearers to be as smoke free and perfume free as possible. This may mean no smoking in the vehicle that stores the samples, and washing hands thoroughly before sampling. Do not expose samples, or sampling equipment to vehicle exhaust or other point sources of contamination.

STORAGE Soil samples intended for chemical analysis should be stored in appropriate sampling containers, and placed, immediately, in refrigeration (i.e., a cooler with ice) prior to and during transport to the analytical laboratory. The soil samples should be maintained at $\leq 10°C$ in transit (but not frozen), to reduce the potential for biological alteration/activity that could affect the analytical result, but also kept from freezing (the sample container may be ruptured by expansion caused by the freezing).

Use care when storing materials onsite to avoid contamination. Do not expose sampling materials to vehicle exhaust or other point sources of contamination.

Due to variation in holding times for different chemical compounds, soil samples should be submitted to the analytical laboratory as soon as practical, and ideally on the same day as the samples were collected. To avoid potential exceedances of holding times, the sampler should review the sample holding times with the designated analytical laboratory and develop a strategy for sample collection and submission.

In addition, the analysis to be performed will influence the type and composition of the sampling container used. It is important to identify to the analytical laboratory what type of analysis is to be conducted on the soil samples and so that the laboratory can supply the appropriate sample containers. In general, for organic analyses, clean, glass sample containers are preferred. For metals analyses only, plastic sample containers are preferred.

SAMPLING CHECKLIST

Planning and Preparation:

- ☐ Conduct a detailed review of site features and sampling objectives.

- ☐ Identify the chemicals of potential concern (COPCs) and potential depth of occurrence. Select sampling and analytical methods (and equipment) based on site-specific conditions and project objectives. Review health and safety requirements based on site features and COPCs, develop separate project Health and Safety Plan (this is beyond scope of this SOP) and implement all appropriate checks (e.g., service clearance, subsurface structure review, *etc*.) and personal health and safety precautions (e.g.,

chemically resistant clothing and gloves, respiratory protection, ear and eye protection, *etc*.).

- ☐ Obtain, calibrate and prepare, as necessary, required field equipment, including field monitoring equipment, field test kits, sampling utensils, laboratory supplied containers, *etc*.

- ☐ Based on project objectives, determine the sample volume requirements. Check that the selected sampling method can produce the required volumes. If not, reconsider the sampling method.

- ☐ Correspond with the analytical laboratory concerning sampling requirements, holding times and preservation. Develop a sampling plan to avoid exceeding established analytical holding times. Note holding times for individual chemicals and identify detection limits required to meet applicable criteria.

- ☐ Determine and plan the field quality control samples that will be obtained for the project including field duplicate samples, field blanks, equipment blanks, and trip blanks (see Chapter 3 of *Guidance Manual* for further details). Laboratory supplied containers are recommended. Where feasible, conduct testing of quality control samples early on in the field program so that adjustments can be made, when warranted. Detection limits should be determined in accordance with applicable criteria.

- ☐ Assemble the necessary equipment, containers, materials and documents prior to mobilization to the site.

Sample Collection:

1) Layout sampling equipment at an appropriate, accessible and clean location. Use plastic sheeting to protect equipment from dirt or other contaminants. Check the function of field monitoring equipment. Prepare, as necessary, decontamination materials and equipment.

2) Prepare field notebook or sampling sheets to document the sample collection, observations and field screening results. Keep the notebook or sampling sheets in a clean and secure, but readily accessible location.

3) Collect the soil sample using new disposable gloves and the type of equipment utilized for the project (i.e., trowel, hand auger, drilling rig, backhoe, *etc*.). Try to collect sufficient soil to, as a minimum, fill the sample container, and if possible, complete field screening or testing work. [Note: All equipment that comes in contact with a sample should be decontaminated between each sample collection]

4) When collecting the soil sample from a piece of equipment (i.e., a shovel, split spoon sampler, backhoe bucket, *etc*.), collect the sample from a zone that preferably has not come into contact with the piece of equipment, to avoid potential cross contamination. Where feasible, scrape off any smeared soil at the contact between soil and the sampling equipment before collecting the soil sample.

5) If collecting a soil sample *in-situ* (from an excavation wall or base, for example), typically the soil sample is collected over a discrete interval from between approximately 0.05 metres (m) below surface (i.e., to avoid the smear/mixing zone at surface) to up to 0.25 m depth. For *ex-situ* (stockpile) sampling it is recommended to obtain samples from within the core of the stockpile; collection of soil samples from the surface of the stockpile is not acceptable [Note: Before collecting samples from an excavation, consult the site health and safety plan. Do not collect samples from steep or potentially unstable walls, unless clearance is given from a qualified professional. Never enter a test pit or confined space without appropriate safety procedures in place, and never enter an unsupported test pit greater than 1.2 m in depth.]

6) Place the soil sample directly into the appropriate sample container. Fill the sample container such that as little as possible headspace remains in the sample container. Where field preservation may be warranted (i.e., samples for volatile organic compound analyses [see section 5.6 of Volume 1]), extrude the soil sample core into the container containing the preservative as per published methods and/or in consultation with the analytical laboratory. Seal the top of the sample container once it is filled. Label, document (including Chain-of-Custody entries) and store the sample appropriately (i.e., in a cooler, or like container). To avoid breakage of glass containers, add a suitable insert packing material in the cooler.

7) Document the location of the soil sample (area, depth), the composition of the soil sample (soil type, colour, moisture, density, *etc*), and any unusual features (staining, odours, waste materials, construction debris or other non-native constituents).

8) Conduct field screening on the remaining soil sample, as required. Do not submit the sample subjected to field screening (i.e., headspace vapour test sample) to the analytical laboratory for chemical analysis since there is potential for losses and bias through volatilization and cross contamination.

9) In the case of composite soil samples, collect discrete samples, as noted above, from the zone or material to be assessed. Note that the number of discrete samples included in a composite generally varies as a function of the volume of material and with the level of contamination anticipated. Samples for VOC analysis should not be composited.

For each discrete sample, collect an equal volume portion and place this portion in a clean container (typically a stainless steel mixing bowl). Once the portions of the discrete samples have been placed in the bowl, homogenize the discrete samples to form a composite sample. Once homogenized, collect a sub-sample of soil and place this soil sample in an appropriate sample container. Fill the sample container such that minimal to no headspace remains in the sample container. Seal the top of the sample container once it is filled. Label, document (including Chain-of-Custody entries) and store the sample appropriately. Preparation of composite samples can result in the loss of volatile organic contaminants and semi-volatile contaminants, as such, this approach is discouraged for sites with these PCOCs.

Document the location of the composite soil sample (area, depth, volume), the composition of the soil sample (soil type), any unusual features (staining, odours, waste materials, debris, *etc*.), and the compositing process used (number of discrete samples, locations, *etc*.). [In the case of composite soil samples, it is recommended that the discrete samples that make up the composite sample be retained for potential future analysis, should unusual or unexpected results be found for the composite sample. In the case of stockpile sampling, it is recommended that selected discrete samples be analysed along with the composite sample, to assess potential variability within the stockpile.]

10) In the case of the collection of field duplicate soil samples, at the time of sample collection, split the soil sample into two, equal volume segments (i.e., for a core sample, split the core down the middle), and place each split sample in a separate sample container. For a composite sample, this may be accomplished by splitting the homogenized sample and filling separate sample containers.

11) Following the collection of each soil sample, complete the field notes (including the photographing of the sample and sample location, if warranted), decontaminate the sampling equipment used, discard disposable equipment (i.e., disposable gloves), and prepare for the next sample. All washwater, cutting and other discarded materials should be managed appropriately to prevent the spread of contamination.

12) Soil sampling equipment (e.g., split-spoons, bowls, utensils) should be decontaminated prior to collecting each sample. The decontamination procedure should consist of cleaning with soapy water (e.g., Alconox) followed by a thorough distilled-deionized water rinse. For certain organic contaminants, an initial cleaning step involving solvents (e.g., hexane or acetone) will be required. For heavy equipment (drilling rods, excavator buckets), a high temperature pressure wash is recommended to remove soil and contamination. The frequency of decontamination of such equipment may

depend on site-specific factors and the type of contaminant expected or encountered.

13) At the end of each day, verify that the samples collected are consistent with those logged on the Chain-of-Custody form(s) and review and augment descriptions, as necessary. Submit the samples to the analytical laboratory as soon as possible. [Note: Once the samples are sealed in the containers, the containers should not be opened for additional inspection prior to submission to the analytical laboratory. Where possible, retain the "extra" sample used for field screening purposes to review soil descriptions.]

Documentation:

1) Document all sampling activities and sampling conditions, and retain documents in a safe and secure place.

2) Strive for completeness in field notes. Include sample collection procedure, sample equipment decontamination procedures, the sample location, sample depth, sample volume, any compositing used to form the sample, description of soil particle sizes, staining, odours, waste materials, construction debris, other non-native objects, native organic matter, woodwaste, non-aqueous phase liquid (NAPL) and field screening data. A checklist for soil description includes the following (refer to Chapter 5 of the *Guidance Manual* for further details):

   - Moisture content
   - Colour
   - Mottling
   - Soil composition
   - Particle shape
   - Structure or fabric
   - Debris
   - Odour
   - Staining
   - Presence of non-aqueous phase liquids (fuels, solvents)
   - Compactness or consistency
   - Thickness
   - Mottling and colour variations along fracture surfaces (secondary porosity features)

3) Supplement written observations with photographs, unless not permitted by the site owner or responsible person. Use a scale or item of known dimension in the photographs for size comparison purposes.

4) Record observations, weather conditions, and sampling notes in durable field notebooks and/or on standard forms. Record relevant sampling information on the sample containers (labels) and record sample information on Chain-of-Custody forms. Always record the complete information needed for identification of the sample and project either immediately prior to filling the sample container or immediately after. Do not wait to fill out the sample label at a later time.

5) If samples are collected for legal purposes, implement appropriate, additional security measures including, but not limited to, adhesive lid seals and security locks for sample storage.

6) Identify the sampling locations on a site map, if available and if feasible.

7) Mark the sampling locations with appropriate equipment (stakes, spray paint, flagging, *etc*.) if locations are to be surveyed at a future date, or if the sampling locations are to be re-visited. The use of GPS to determine location may also be an option.

ADDITIONAL INFORMATION

Refer to Chapter 5 of the *Guidance Manual* and other relevant chapters, for additional information pertaining to soil sampling.

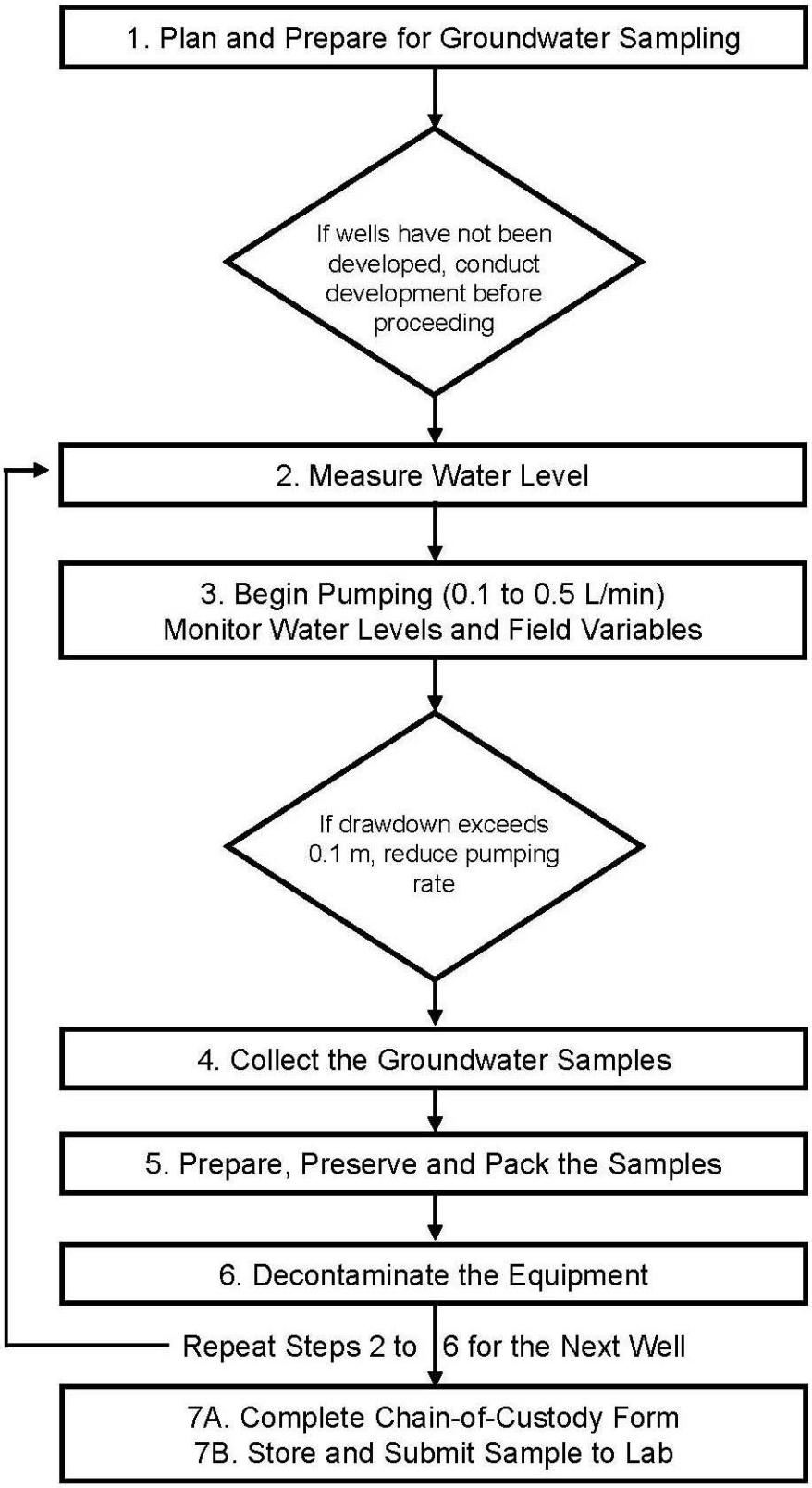
SUGGESTED OPERATING PROCEDURE NUMBER 3: LOW-FLOW GROUNDWATER SAMPLING

SCOPE This suggested operating procedure (SOP) provides guidance on the sampling of monitoring wells using low-flow methods. It is applicable to groundwater sampling, and **is not appropriate where non-aqueous phase liquids (NAPLs) are present in the well**. There are different ways of groundwater sampling that can provide acceptable results; however, the most appropriate method will depend on site-specific conditions. Low-flow sampling is best suited to monitoring wells completed in permeable formations (hydraulic conductivity greater than 10^{-5} m/s). Where appropriate, low-flow sampling can reduce the volume of purge water, and minimize disturbance and sample turbidity. Additional information on low-flow sampling is provided in Barcelona *et al*., 2005; USEPA 1996; and Barcelona *et al.*, 1994.

WHEN? Low-flow sampling is used to obtain a representative groundwater sample from a monitoring well. Samples should be acquired after a rest period of at least one week following well installation and development. Samples may be acquired on different days and at different times through the year to provide information on data precision and temporal changes in groundwater chemistry.

WHY? Sampling techniques using conventional approaches can sometimes disturb conditions at the well screen, yielding non-representative samples for chemical analysis. For example, turbid samples are commonly generated by using bailers or inertial lift samplers that are inappropriately utilized. This can sometimes yield non-representative, elevated results for some metals and highly sorbing organics such as polyaromatic hydrocarbons (PAH) and polychlorinated biphenyls (PCBs). Some conventional approaches also may cause degassing of volatiles in the well during sampling, resulting in low concentrations of volatiles that are not representative of actual groundwater conditions.

HOW? The main steps in low-flow groundwater sampling are shown in the following diagram and described in the procedure below. The sampling procedures should be completed in the order presented, at each monitoring well location. All reusable equipment must be decontaminated in accordance with applicable procedures, prior to the commencement of sampling.


1. Plan and Prepare for Groundwater Sampling
If wells have not been developed, conduct development before proceeding
2. Measure Water Level
3. Begin Pumping (0.1 to 0.5 L/min)
Monitor Water Levels and Field Variables
If drawdown exceeds 0.1 m, reduce pumping rate
4. Collect the Groundwater Samples
5. Prepare, Preserve and Pack the Samples
6. Decontaminate the Equipment
Repeat Steps 2 to 6 for the Next Well
7A. Complete Chain-of-Custody Form
7B. Store and Submit Sample to Lab

Essential Information

- ☐ Monitoring well log or drawing of installation (showing depth of casing, diameter of well screen, casing and filter pack, screen length, pump depth, ground surface elevation, and measuring point elevation).
- ☐ Record of the most recent water-level measurements (if applicable).
- ☐ Site plan showing the location of the well.
- ☐ Project Health and Safety Plan (separate document, beyond scope of this SOP).

Equipment Checklist

- ☐ calculator (for calculating purge volumes)
- ☐ appropriate health and safety equipment (develop separate project Health and Safety Plan)
- ☐ field book, site plan, pens, pencils, indelible marker
- ☐ one or more of the following to access the well:
  - ☐ key (if needed) for wellhead and/or j-plug
  - ☐ socket wrench for opening bolt-down well covers (usually 9/16 inch)
  - ☐ pry bar or screw driver (if needed) to open surface covers
- ☐ electronic water-level probe
- ☐ interface probe
- ☐ de-ionized distilled water
- ☐ graduated container (e.g., 4 litre bucket) for measuring volume of water removed
- ☐ container(s) for collecting purged water (i.e., drum or tank) if water cannot be disposed of on-site
- ☐ pail (approximately 20 litre) for the temporary collection of purged water
- ☐ knife (for cutting tubing)
- ☐ fish hook (to retrieve lost items downhole)
- ☐ camera (and film)
- ☐ field meters (pH, conductivity, temperature, dissolved oxygen, redox potential) and calibration solutions
- ☐ 250 mL plastic beaker or a flow-through cell (preferred) for measurement of field indicator parameters and flow rate confirmation
- ☐ disposable bailers and bailer cord (as back-up)
- ☐ garbage bags
- ☐ plastic sheeting or plywood
- ☐ filtering apparatus (for dissolved metals samples only)
- ☐ sample preservatives
- ☐ borehole logs
- ☐ decontamination equipment:
  - ☐ biodegradable detergent, rinse water, deionized water, paper towels, brush, bucket (for washing equipment)
- ☐ pre-cleaned sample bottles with labels and lids, including extra sample bottles and labels, provided by analytical lab
- ☐ cooler with appropriate packing material
- ☐ ice packs (or heat packs, if appropriate)

One of more of the following sampling devices:

- ☐ **Bladder pump** and pump tubing, controller and air compressor/power source or nitrogen gas
  - or **Centrifugal pump** (downhole submersible, e.g., Grundfos), pump tubing and power source (electrical or gas-powered generator with fuel)
  - or **Peristaltic pump (where water table is less than 3 m)**, silicon or Tygon™ tubing*, sample collection tubing**, battery, power cables.

* silicon or Tygon™ tubing is used in the drive head of the peristaltic pump; the pump should be configured to collect the water sample under vacuum before contacting the Tygon™ tubing; however, if not possible, the length of this piece should be minimized due to potential adsorption problems.

** the sample collection tubing should be made of high density polyethylene (HDPE), polytetrafluoroethylene (PTFE), PTFE-lined polyethylene or similar material. This will reduce the potential for adsorption of chemical parameters.

Other Equipment

If groundwater is likely to contain organic contaminants such as PCBs, organic solvents may be required.

Essential Forms

- ☐ Groundwater Development, Purging and Sampling form (or log book).
- ☐ Sample Chain-of-Custody form.

1. Planning and Preparing for Groundwater Sampling

- ☐ Schedule sampling to ensure that the samples are delivered to the laboratory, and that the laboratory has sufficient time to start the analysis, within the recommended holding time. Sometimes delivery of samples at the end of the day or work week can result in delays in commencing the analysis, so keep this in mind when shipping or transporting samples.
- ☐ If several wells are to be sampled, plan to collect groundwater samples from the wells expected to be least contaminated first, and then proceed to more contaminated areas.
- ☐ Organize the groundwater sampling equipment and forms.
- ☐ Determine the type and size of sample bottle and any preservative required for the chemical parameters to be analyzed.
- ☐ All sample bottles and preservatives should be provided by the **selected analytical laboratory**. If volatile organic compounds (VOCs) are to be analyzed, trip blanks for QA/QC purposes should be prepared by the analytical laboratory.
- ☐ Review all steps in this procedure, particularly Step 5 - *Preparing, Preserving and Packing the Samples*. If samples will be shipped or transported a significant distance, proper packing of the sample bottles (to ensure that no breakage occurs) is particularly important.
- ☐ Ensure that the well was properly developed **at least one week** prior to sampling and that water in the well has reached static level. Where wells have not been sampled for periods of several months or years, well re-development may be necessary prior to sampling to remove sediment accumulations from the bottom of the well.
- ☐ Review relevant information regarding well installation, recent water levels and water quality during previous sampling round.
- ☐ Determine how purged water will be handled and/or disposed of (i.e., does it require collection and temporary containment or can it be disposed of on-site) and make appropriate arrangements. Before going to the site, estimate the volume to be purged (if possible), and ensure that sufficient containers (e.g., drums) will be available for the water collection.
- ☐ Ensure that all sampling equipment, including pumps, bailers and filters, has been properly decontaminated.

On-Site Preparation:

- ☐ Lay out plastic sheeting or clean plywood (on which to place sampling equipment) adjacent to the monitoring well, to prevent contamination of the equipment by the ground surface.
- ☐ Put on appropriate personal protective equipment. This will include, at a minimum, disposable latex or nitrile gloves and safety glasses. Rubber boots, and rain gear or tyvek suit are also recommended. See project Health and Safety Plan for guidance.
- ☐ Prepare and lay out the sample bottles and filtering equipment on the plywood or plastic, as appropriate (see Step 5 of this procedure).

- ☐ Prepare temporary on-site waste storage area for temporary waste storage (e.g. filters, tubing, *etc*.).

2. Measuring the Water Level

1. Ensure that the water-level tape and interface probe have been decontaminated.
2. Monitor the well for the presence of non-aqueous phase liquid (NAPL). If product is present, consult project manager to determine whether a product sample and/or water sample should be collected.
3. Measure the depth to water below the top-of-casing.
4. Measure the height of the top of casing above or below nominal ground surface.
5. Record measurements on a Groundwater Development, Purging and Sampling form or log book.
6. Measure the depth to the base of the well. Calculate the thickness of accumulated silt on the base of the well, and therefore the depth interval of open screen. If the available screen height is insufficient for sampling (i.e., less than 0.3 m), then re-development may be required.
7. Decontaminate the water-level tape after taking the reading. This may be accomplished as the tape is extracted from the well, by spraying with de-ionized water and wiping dry with paper towel.

3. Begin Pumping (0.1 to 0.5 L/min). Monitor Water Levels and Field Variables

1. Set up pump apparatus at well. If a fuel-powered generator is used, set up generator downwind of well.
2. The depth of the pump intake should be placed within the screened interval of the well, preferably at least 0.3 metre above the base of the well, to minimize contact of the pump with any accumulated sediment in the well. The depth below the top of casing should be recorded.
3. Wear nitrile gloves during the pumping process.
4. Set the pump carefully in the well and secure at the required depth. The depth should be consistent between sampling events for each well.
5. Minimize movement of equipment in the well as far as practicable so as to minimize disturbance of the water column, and thereby minimize turbidity, volatilization losses and oxygenation of the groundwater.
6. Attach pump discharge hose to the flow-through cell and set up calibrated field probes to meters (i.e., pH, temperature, electrical conductivity, dissolved oxygen, redox potential).
7. After set-up, re-measure water level in well after pump placement.
8. Start pump from a zero-flow control position, and then gently increase the flow to a rate between about 0.1 and 0.5 L/min. Measure the flow rate using a graduated cylinder or bucket.

9. If required, collect discharged water in an appropriate container for subsequent disposal or treatment.

10. Purge at least one volume of water estimated to be contained within the pump tubing (for a standard 12 mm inside diameter hose, this equates to 0.1 L/metre of hose).

11. Once the volume of water from tubing has been purged, commence measuring and recording water quality variables every three to five minutes until three consecutive readings have stabilized, as defined below:

pH:	+/- 0.1
Conductance	+/- 3% of reading
Dissolved Oxygen	+/- 10% of reading or 0.2 mg/L (whichever is greater)
Temperature	Must be measured, but no stabilization is required

Other variables that may also be used to assess stability include oxidation-reduction potential.

ORP:	+/-10 mV
Turbidity	+/-10%

However, turbidity is often the last variable to stabilize and may result in excessive purge times if stability criteria are overly stringent.

12. Note that some parameters will stabilize more quickly than others, and some may never stabilize. Visually assess sample turbidity, adjust flow rate, if necessary, to minimize turbidity, and record visual observations on Groundwater Development, Purging and Sampling form or log book.

13. Measure and record the water level at the same time as water quality variables. If water level drops below about 0.1 m of static level, then reduce pumping rate.

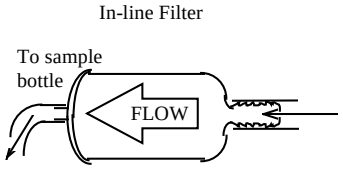
14. If water level continues to decline below 0.1 m of static, and pumping rate is at or less than 0.1 L/min, then the sample may be obtained, recognizing that data obtained may be compromised (i.e., subject to significant sampling bias).

15. A final set of field measurements should be taken immediately before collecting the sample. Record total volume purged, final purge rate, field measurements, and water level at the end of purging.

4. Collect Groundwater Samples

1. Collect the sample by pumping water directly into the sample bottle(s) or upper portion of the filter apparatus, if applicable (see Steps 5 and 6 for further detail).

2. The sample may be filtered, if necessary, by attaching a disposable in-line filter to the end of the tubing (i.e., using silicon tubing to join the two together). ***Pump at least 0.5 litre of sample water through the filter*** prior to filling the sample bottle. Pump this excess into the purge water container(s). The in-line filter should be discarded after the sample is filtered.



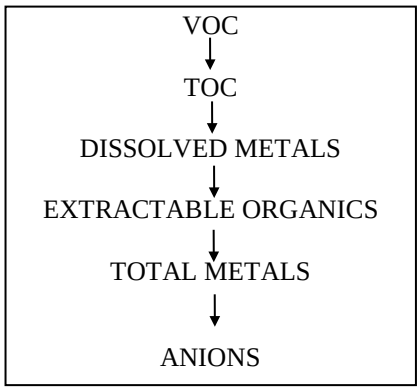
3. Record the observations on the Groundwater Development Purging, and Sampling form including colour, turbidity, odour (if present) and sheen (if present) of sample.

5. Prepare, Preserve and Pack the Samples

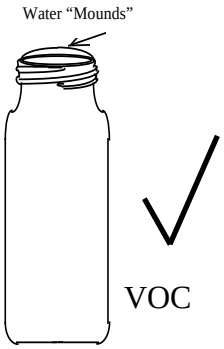
IMPORTANT: The results of any laboratory analysis are dependent both on the ability to collect a representative sample and to maintain that sample until it can be analyzed. Sample preparation, preservation and storage procedures are designed to ensure that the sample is properly maintained.

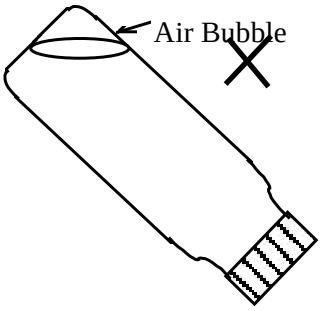
1. As a general guideline, fill the bottles for those samples that require special handling first (e.g., filtering, preservative) followed by those that do not.

2. Sample for volatile organic compounds (VOC) first, followed by Total Organic Carbon (TOC) and those constituents that require field filtration (i.e., dissolved metals). Collect samples for analysis of extractable organic constituents, total metals and anions last.



3. Fill VOC sample bottles completely until water "mounds" on top of rim. Place the cap on snugly and then ensure that no air bubbles are trapped in the bottle. Check this by tipping the bottle upside down and tapping the bottle to dislodge any bubbles. If bubbles are present, discard the sample and resample.





4. Samples to be analyzed for dissolved metals must be field-filtered and acidified.

5. If samples require a preservative:

   - **If the preservative is already present** in the sample bottle provided by the analytical laboratory, do not rinse the bottle, but fill to the rim.

   - **If the preservative is not already present** in the sample bottle provided by the laboratory, rinse the bottle and cap with the sample water three times, then fill the sample bottle to the base of the neck, taking care to leave sufficient room for the preservative. Add preservative immediately.

6. If samples do not require a preservative, rinse sample bottle and bottle cap three times, and then fill bottle to the rim.

Sample Label:

Site ID_____
Sample ID_____
Matrix Type_____
Date/Time Sampled_____
Parameters_____
Preservatives_____
Filtered Y/N_____
Sampler Initials_____

7. Seal all sample bottles tightly.

8. Label all samples clearly with an indelible marker. Include sample number/ID, sample location, date and time collected, sampler, project descriptor, parameter to be analyzed and preservative (if added).

9. Collect a sufficient number of duplicate samples to meet data quality objectives and QA/QC requirements. Collect duplicate samples if access to the sampling location is difficult or if the sampling procedure is particularly time consuming. This will help ensure that a sample is available if breakage of some of the bottles occurs during transport or at the laboratory.

10. Pack samples in a cooler and maintain at ≤ 10°C in transit (but not frozen) using ice packs. If the samples are collected and/or transported in very cold weather, use heat packs, if required, to prevent freezing.

11. Pack the samples in a transport container such that they will not break due to impact or vibration. Do not let sample bottles touch each other or they may break. Place generous amounts of packing material between all glass bottles.

6. Decontaminate the Equipment

1. Thoroughly decontaminate the water-level probe, bailer or pump, filtering apparatus (discard used in-line filters) and all other relevant sampling equipment before sampling the next well.

2. Repeat Steps 2 to 6 for the next well.

7A. Completing the Sample Chain-of-Custody Form

1. Complete the sample chain of custody form once all wells have been sampled.

7B. Storing and Submitting the Samples

1. Ensure that the samples are delivered to the laboratory, and that the laboratory has enough time to start the analysis, within the recommended holding time. Sometimes delivery of samples at the end of the day or work week can result in delays in commencing the analysis, so keep this in mind when shipping or transporting samples.

REFERENCES

Barcelona, M.J., H. A. Wehrmann, and M.D. Varljen. 1994. *Reproducible well sampling procedures and VOC stabilization criteria for ground water sampling.* Ground Water 32, No. 1. pp. 12-22.

Barcelona, M.J, M.D. Varljen, R.W. Puls and D. Kamininski. 2005. *Ground Water Purging and Sampling Methods: History vs Hysteria.* Ground Water Monitoring and Remediation, 25, No. 1, Winter.

United States Environmental Protection Agency (USEPA), 1996. *Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures.* EPA/540/S-95/504.

SUGGESTED OPERATING PROCEDURE NUMBER 4: SOIL GAS PROBE INSTALLATION

SCOPE This suggested operating procedure (SOP) provides guidance on the installation of soil gas probes. Because there are different ways of constructing and installing probes that provide for acceptable results, a range of options are provided. Additional information on soil gas probe installation is provided in Volume 1 of the *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as *"Guidance Manual"*).

WHEN? Soil gas probes are typically installed for one of three purposes: (i) to collect data in support of a risk assessment, (ii) for initial screening of volatile contamination to optimize subsequent intrusive investigations and locations of boreholes and monitoring wells, and (iii) to monitor biodegradation processes typically through testing of oxygen, carbon dioxide and methane.

HOW? Soil gas probes may be installed external to a building (refer to as "external" probes) or below a building foundation slab. When close to the underside of the slab, such probes are referred to as "subslab" probes. Probes can be constructed of a variety of materials and installed using several techniques. Critical aspects to probe construction include: i) the use of materials that are inert and non-sorptive, ii) the design of seals that minimize the potential for short-circuiting of atmospheric air to the soil gas collection point, and iii) surface completion including a valve to allow the probe to be sealed between sampling events. The selection of probe type is dependent on the conceptual site model (CSM) and on project specific objectives. Considerations for CSM development are provided in Chapter 4 of the *Guidance Manual* and in Checklist No. 4 (Volume 2).

Though various options exist, the fundamental concepts for probe construction are similar. The following checklist provides a general list of considerations for soil gas survey design and probe installation and construction.

PROCEDURE

Soil Gas Sampling Design:

An overview of selected considerations for soil gas sampling design is provided below. Refer to Volume 1 of the *Guidance Manual* for additional details.

- Identify the objectives of the soil gas sampling program.
- Integrate the CSM into the soil gas sampling design.

- Identify areas, and/or buildings, of potential concern.

- Identify targeted areas for investigation. Often it is appropriate to start with soil gas characterization near the contamination source zone. Consider using grid patterns (or transects) and vertical profiles between the contamination source and the building of potential concern to evaluate the vapour transport pathway. Consider whether subslab soil gas probes are warranted.

- Where possible, external probes should be installed at a minimum depth of 1 metre (m) to reduce the likelihood of ambient air being drawn through surficial soils (referred to as "short-circuiting"). External shallow probes less than 1 m deep may be warranted where there is a shallow water table and/or contamination. Good practice is to place a plastic sheet around a shallow probe to minimize atmospheric air intrusion. Recommended minimum dimensions of the plastic sheet are 1.5 m by 1.5 m. The plastic should be weighted down at the edges with sand or sand bags.

- External probes that are used for risk assessment purposes should be installed to a minimum depth half-way between the source of soil vapour contamination and the lowest point of the building of concern. This is because shallow data may be non-representative of conditions below the building.

- Subslab probes are typically installed directly below the slab. However, if exfiltration of building air could occur, consider also installing deeper probes beyond the advective soil gas flow zone (e.g., approximately 1 m below slab may be reasonable).

- When external soil vapour concentrations are being used to assess an existing building, generally soil vapour probes should be installed within 2 to 3 m of the building (and no more than 10 m from the building), but outside the zone of disturbance along the foundation wall. Probes on at least two sides of the building should be installed.

- Consider the influence of utilities and possible preferential pathways on soil gas sampling design.

Planning and Preparation:

- **Health and Safety Plan, Permits, Approvals and Utility Clearances**: Develop separate project Health and Safety Plan (this is beyond scope of this SOP), obtain all necessary permits and/or approvals in advance of drilling activities. Review and locate underground and aboveground utility locations.

- **Evaluate Potential Safety Issues**: Evaluate safety issues and whether integrity of building envelope, structure, and underground utilities could be affected. As necessary, review available building plans, contact knowledgeable persons (e.g., structural engineer) and perform geophysical testing. Subslab probes should not be installed through slabs where there is post-tensioned steel unless all necessary pre-cautions have been taken.

- **Select Probe Type**. The main options include:

  1. Probes installed in boreholes constructed using conventional drilling techniques.
  2. Probes installed using direct push techniques.
  3. Probes driven into the subsurface (typically temporary probes) either by hand, electric rotary hammer or direct push rig.

  The main advantages of probes installed in drilled boreholes or direct push is that a filter pack and seal may be constructed. An added advantage of probes installed in boreholes is that soil stratigraphy may be inspected prior to installation, while this is not possible with driven probes.

  For direct push technology, soil cores may be obtained, so consideration should be given to first obtaining a soil core to evaluate soil conditions, prior to pushing the cone at a second location and either installing an implant post-run or in an open borehole (providing it does not collapse).

  Driven probes have potential advantages in terms of access and cost relative to probes installed using drill rigs, but screens have a greater tendency to clog when in fine-grained soil, which also may fracture during driving of the probe. Consequently, driven probes are not recommended for fine-grained soils. Obtaining repeat samples is precluded when temporary driven probes are used.

- **Select Drilling Method**: Drilling methods will vary depending on geologic materials, target depth and access constraints. Methods that create smaller boreholes with the least amount of disturbance are highly preferred (e.g., Geoprobe, auger). Rotary sonic methods are acceptable but the use of air or water should be avoided to the extent possible. When using water, consider the possible generation of trihalomethanes such as chloroform and effect on soil vapour concentrations. Air rotary or hydro-vac methods should not be used unless there are no other alternatives.

  The drilling method may also depend whether multiple probes are to be installed in a single borehole. Since typical hollow stem augers have an inside

diameter of 107 mm (4 ¼ inches), it is possible to install three 19 mm (¾ inch) probes inside the augers, although care must be taken to avoid bridging when placing filter and seal materials. If a proper installation cannot be achieved for multiple probes, install probes in separate boreholes. Soil gas probes may also be installed in an open borehole, but only if the hole does not collapse. Care must be taken to install a proper filter pack and seal.

Select Method of Driven Probe Installation: There are smaller commercial retractable tip probes that can be driven to 2 to 3 m depth in looser soils using a slide hammer or an electric rotary hammer. One example is the AMS Retract-a-Tip which comes equipped with 22 mm O.D. rods and 50-mm long screen. Deeper probe deployments are possible are possible with direct push rigs, but dense soils or cobbles may preclude their use. One example is the Geoprobe PRT system (31.5 mm diameter O.D. rods) (Geoprobe, 2006). If a system that utilizes new tubing (preferred practice) threaded down the rods for collection of each new sample (e.g., Geoprobe PRT system), the connection between the PRT tubing and sampler should be leak tested after the sampler is retrieved.

- **Select Probe and Sampling Train Materials**. The probe and sampling train should be constructed of inert and non-porous materials. Stainless steel, Teflon® and PVC are acceptable materials. Nylaflow® (nylon) tubing is acceptable for most volatile chemicals, but excessive sorption of naphthalene onto Nylaflow® has been shown and thus it should not be used for this or similar compounds. No glues, tape, or other materials that could emit volatiles should be used. Only new materials should be used for probes, except when using temporary steel probes.

- **Select External Soil Gas Probe Construction**. The material type, diameter, screen length, and connections should be determined:

  - Common permanent external probes are continuous rigid PVC to ground surface (with short slotted section) or stainless steel mesh screens (“implants”) attached to flexible tubing to ground surface.

  - A probe diameter of 25 mm (1 inch) or smaller should be used to minimize the purge volumes and reduce the potential for short-circuiting.

  - Short screens (0.1 to 0.3 m length) should generally be used unless there are thick vadose zones (i.e., greater than about 10 m) where longer screens may be appropriate or where the objective is large volume composite soil gas sampling.

- For probes constructed of rigid PVC pipe, 19 mm (¾ inch) diameter pipe is recommended. Screens may consist of No. 10 to No. 40 slot pipe. Riser pipe connections should be flush-threaded and no glue should be used.

- For probes constructed of implants, a common diameter is 12.5 mm (½ inch), with length of 0.15 to 0.3 m. Typically 6 mm (¼ inch) diameter tubing is used to connect the implant to ground surface. Smaller diameter tubing can lead to excessive frictional losses when conducting pneumatic tests.

- Couplings should be air-tight Swagelok™ compression-fittings, barbed-fittings, or threaded fittings. When barbed-fittings are used, push tubing over a minimum of three barbs. Slip fittings should not be used.

- Probes should be completed with an air-tight cap (for PVC pipe) and valve at surface to prevent atmospheric air from entering the probe.

- Probes should be labeled without using volatile organic compound (VOC) emitting markers.

- **Select Subslab Soil Gas Probe Construction**. The material type, diameter, screen length, and connections should be determined:

  - Common subslab probe designs are a probe installed in a sealed drill hole or core hole in the slab (permanent), or an expanding plug-type probe (typically temporary).

  - Probes should consist of inert materials such as steel or brass tubes and Teflon fittings. A variety of subslab probes designs are possible, a design by USEPA (2004) is shown in Figure 1. When not in use, the probe is sealed with a recessed threaded cap. For sampling, the threaded cap is replaced with a fitting with threads on one end and ¼-inch compression or barbed fitting on the other end.

  - Expanding plug type probes should be installed in a properly size drill hole with smooth walls.

  - Concrete grout should consist of cement, aggregate and water, and should not contain any additives that could contain VOCs. Since regular concrete may develop shrinkage cracks over time, an expanding or swelling concrete designed to seal wet cracks in concrete floors may provide for better performance.

- A bentonite seal or other non-VOC containing products such as polyethylene glue or bees-wax should be placed around the temporary probe. A bentonite seal may also be placed around a permanent probe when sampling as an additional protective measure.

Material Handling, Storage and Decontamination:

- **Handling and Storage**: Probe materials (e.g., PVC pipe, tubing and implants) should be delivered to the site wrapped in plastic. Use care when storing materials on-site and when installing probes to avoid contamination. Do not expose probe materials to vehicle exhaust or other point sources of contamination.

- **Decontamination of Temporary Steel Probes:** Steel probes such as retractable soil gas sampling tips should be thoroughly washed with a hot-water soap solution followed by a distilled-deionized water rinse. Probes should be completely dry prior to reuse, as water droplets on the inside of the probe could affect soil gas concentrations. See SOP #2 for testing of blanks.

Installation of Probes or Driven Probes:

Probes Installed in Boreholes

1. Log the borehole as drilling proceeds. Adjust the depth of the soil gas probe, if warranted based on the soil stratigraphy observed and field screening results.

2. Place a thin sand layer (2.5 to 5 centimetres) at the base of borehole when installing probe so that it is not in direct contact with native soil to avoid clogging.

3. Insert the probe through the drill rods or in open hole. Install filter pack and seal while removing the rods as described below.

4. Place a filter pack comprised of coarse sand or fine gravel around the screen. Extend the filter pack 0.15 m above the top of the screen. If gravel is used, a thin sand layer between the gravel and bentonite should be used.

5. Install a bentonite seal above the filter pack consisting of dry granular bentonite (16 mesh). The bentonite seal should be a minimum 0.3 m thick. Place seal in two to three lifts that are a few centimeters thick and hydrate with distilled-deionized water.

6. Seal the remainder of the borehole annulus to near to ground surface using a thick slurry of powdered bentonite and water (“Volclay Grout”) installed using a tremie pipe, or use granular bentonite.

7. Use a tamping rod and weighted tape to verify position of filter pack and seal.

8. Where more than one probe is installed within a single borehole, install a minimum 0.15-m thick bentonite seal between probes constructed of either a slurry or granular bentonite. The seals between multi-level probes should be tested by pumping from one probe with a minimum vacuum of 10 inches H_20 column and monitoring adjacent probes for vacuum. A faulty seal will result in a rapid increase in vacuum in adjacent probes to significant levels.

9. Probes should be protected using a well cover or other similar protective casing for security and weatherproofing. The well covers should be appropriate secured in place with concrete.

10. As warranted, probes should be protected from disturbance (which can lead to short-circuiting during sampling) using appropriate methods (e.g. bollards, concrete blocks).

Probes Installed using Direct Push Technology

1. Larger size rods[1] should be used to facilitate the installation of a proper filter pack and seal. Never allow the borehole to collapse around the probe when using direct push technology to install probes.

2. Push probes to desired depth.

3. Lower implant and connect to expendable drive tip with threaded connection.

4. Use same procedure for installing filter pack and seal as for probes installed in boreholes.

Driven Probes

1. Drill small pilot hole where there are asphalt or concrete surfaces, as required.

2. Install probes vertically using a hydraulic ram or slide hammer (do not use a sledge hammer).

3. Minimize post-installation disturbance to probes.

4. Driven probes should not be used in soils that will fracture (e.g., certain types of clay).

[1] For Geoprobe systems use DT-21 dual-tube system with 2.125 inch OD and 1.5 inch ID rods. See www.geoprobe.com\ Direct Push Installation of Devices for Soil Gas Sampling and Monitoring [Tech.Bulletin No. MK3098]

5. If cobbles or other obstructions cause the rods to deflect, the installation should be abandoned, grouted, and re-tried at new location.

6. Place a bentonite seal around the probe at ground surface.

7. Retract sleeve and start sampling process.

Sub-Slab Probes

1. Probes that are installed directly below a slab are described below. For deeper probes, use the applicable procedure described above.

2. Drill hole in concrete using a heavy duty electric rotary hammer drill. Avoid the use of gasoline powered drills. Collect concrete dust during drilling using a dry/wet vacuum cleaner.

3. After drilling the hole and prior to installation of the probe, the hole should be quickly sealed (e.g., using a rubber stopper) to minimize disturbance to subslab vapour concentrations.

4. Install stainless steel or brass insert (Figure 1) and connect fitting on insert to valve. Use non-VOC emitting concrete grout.

5. After installing the probe, close the valve to the probe and allow time for the concrete seal to set before collecting a sample. For fast-setting concrete, about one hour may be sufficient. If the hole has stayed open for any appreciable time and there are pressure gradients between the building and subsoils, a longer waiting period between installation and sampling may be warranted to allow soil vapour concentrations to return to equilibrium.

Leak Testing:

A leak test of the probe seal should be performed at all new soil gas probe locations. Procedures for leak tests are described in SOP #6.

Decommissioning of Boreholes and Probes:

All open boreholes equal to or greater than 25 mm diameter should be sealed with bentonite grout placed using a tremie pipe if not used for installing permanent soil gas probes.

Holes created by driven probes that are less than 25 mm diameter should be sealed by pouring bentonite grout down the hole.

When subslab probes are decommissioned, the holes should be sealed by filling them with non-shrinking cement grout or other appropriate material in order to prevent soil gas from entering the building.

Documentation:

Each external soil gas probe installation should be documented on a separate borehole log form indicating the soil stratigraphy and probe construction details. Subslab probe installations do not require logs.

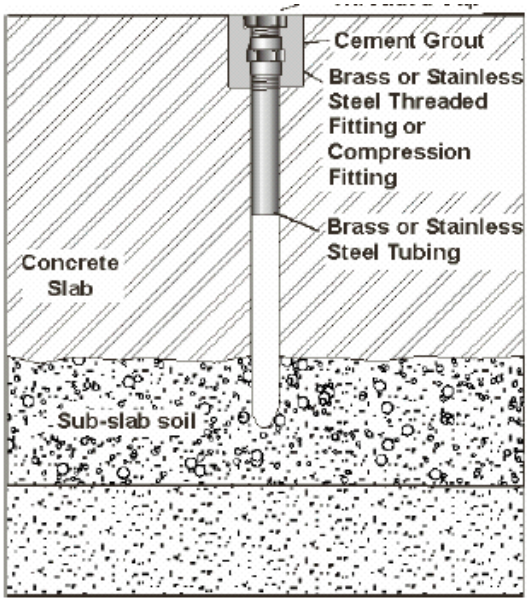


FIGURE 1: USEPA (2004) Recommended Design for Subslab Probes

REFERENCES

United States Environmental Protection Agency (USEPA). 2004. *Standard Operating Procedure (SOP) for Installation of Sub-Slab Vapor Probes and Sampling Using US EPA Method to Support Vapor Intrusion Investigations.* ORD, Ada, OK, Draft – February 12, 2004.

SUGGESTED OPERATING PROCEDURE NUMBER 5: SOIL GAS SAMPLING

SCOPE This suggested operating procedure (SOP) provides guidance on the collection of soil gas samples for chemical analysis. The scope of this procedure addresses soil gas sampling methods and sampling containers or devices used to obtain samples. Additional information on soil gas analysis is provided in Volume 1 of *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as *"Guidance Manual"*).

WHY? The term soil gas refers to the air present in soil pore spaces. Soil gases may be generated through the following processes: (i) partitioning that occurs when chemicals volatilize into soil gas from non-aqueous phase liquids (NAPLs), dissolved chemicals in groundwater, and chemicals sorbed onto soil particles; (ii) anaerobic decomposition of organic chemicals, waste material (e.g., refuse) or native organic matter and generation of biogenic gases (e.g., methane, carbon dioxide, hydrogen sulphide), and (iii) aerobic biodegradation of hydrocarbons or native organic matter and generation of carbon dioxide and consumption of oxygen.

HOW? The soil gas sampling process is summarized in Figure 1. Soil gas samples may be analyzed in the field using hand-held instruments such as a photoionization detector (PID), flame ionization detector (FID), combustible gas detector or landfill gas monitor (e.g., oxygen, carbon dioxide, methane, hydrogen sulphide). Soil gas samples may also be analyzed for specific compounds of interest using more advanced analysis methods, typically at an off-site analytical laboratory. When soil gas data is used in support of a human health risk assessment, chemical analysis of soil gas for specific compounds of interest to low detection limits (i.e., low ppbV levels) is typically required. However, field instruments can be effective as screening tools to locate more contaminated zones and to minimize overall analytical costs.

There are several different methods available for the collection and analysis of soil gas samples for laboratory analysis. The two main options are sampling using active sorbent tubes and canisters. The choice of analytical method will depend on project objectives, sampling methods, detection limits required, and data quality objectives. In the case of field analytical methods, soil gas samples are typically collected in gas-bags or syringes.

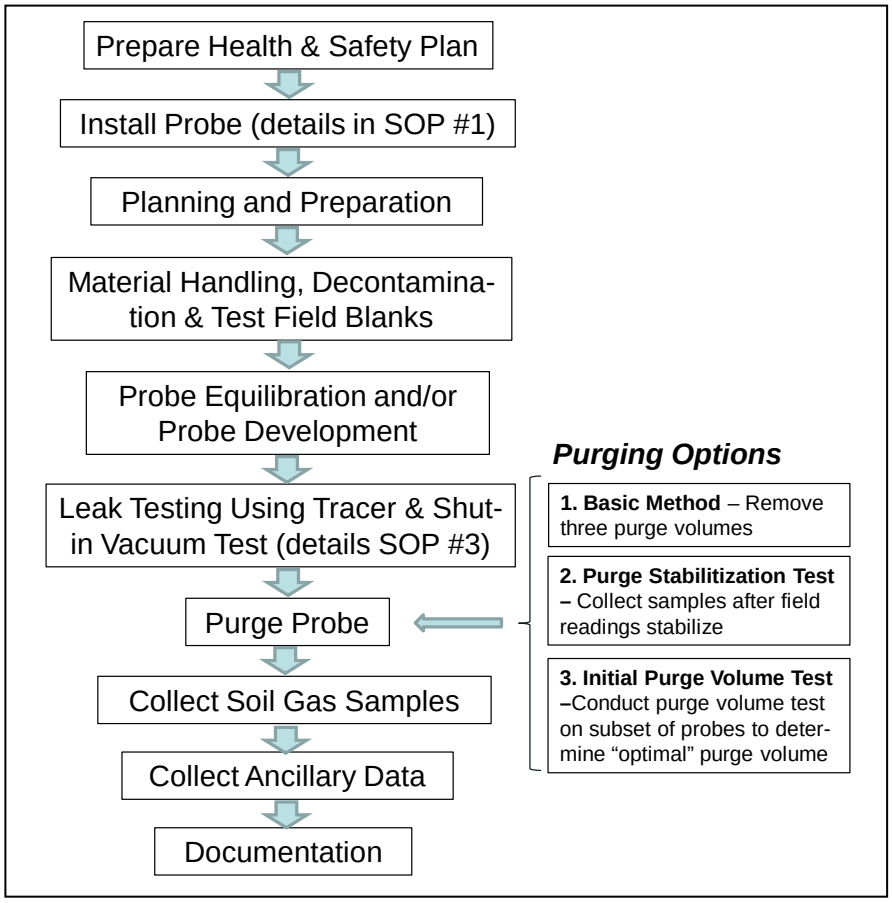


FIGURE 1: Soil Gas Sampling Process

PROCEDURE

Planning and preparation:

- ☐ **Prepare project Health and Safety Plan** (beyond the scope of this SOP).

- ☐ **Review soil gas probe installation details:** Determine if existing probes are adequate to achieve current project objectives and are in good condition. If groundwater monitoring wells are to be sampled, review well logs for screen completion depths relative to the water table and construction methods (e.g., filter pack and seal), and determine whether well is vented at surface (modifications to well and additional purging may be required).

- ☐ **Select Analytical Method and Sampling Device**: Identify the chemicals of potential concern (COPCs), and determine the sampling method and device, analytical method, detection limits, and data quality objectives. The typical sampling options are described in Table 1. Note that SKC Tedlar® bags are being phased out by SKC and replaced by Flexfilm, which is an acceptable alternative for fixed gas analysis. Gas-bags should not be used for low-level (i.e., ppbV) volatile organic compound (VOC) analysis.

Table 1: Summary of Common Analytical Methods for Soil Gas

Method	Sampling Device	Compounds Analyzed	Sample Holding Time
US EPA Method TO-15	Summa polished or fused silica lined (FSL) evacuated canisters (typically 1-6 L)	Broad range of VOCs from propane to naphthalene.	30 days[1]
US EPA Method TO-17	Thermally desorbable sorbent tubes collected using low flow pumps	Wide volatility range from light molecular weight VOCs such as 1,3-butadiene to 4-ring PAHs if multi-bed sorbent tubes are used.[2]	30 days, if stored at 4°C
Modified NIOSH 1501 or OSHA 7	Solvent extracted charcoal tube	Typically BTEX and other petroleum hydrocarbons. Higher Detection Limits than TO-17.	Contact laboratory
ASTM D1946-90 (2006)[3] or D1945-03(2010)[4]	Gas-bag, or Summa or FSL canister	Fixed gases and light molecular weight hydrocarbons.	1 to 3 days for bags; 30 days for canisters
ASTM D5504	Gas-bag or FSL canister[5,6]	Reduced sulphur compounds.	24 hours for bags; 24 hours for FSL canisters

Notes:

1. Recommend that canisters be used for sampling within 15 days of preparation by the laboratory.
2. Method performance is dependent on sorbent tube. Specialty multi-bed sorbents have been developed with good performance over relatively wide range of molecular weight compounds and moisture conditions.
3. Hydrogen, oxygen, nitrogen, carbon monoxide, carbon dioxide, methane, ethane, ethylene.
4. Hydrogen, oxygen/argon, nitrogen, carbon monoxide, carbon dioxide, hydrogen sulphide, helium, C1-C6 alkanes.
5. Note that researchers have reported inconsistent recoveries of reactive sulfur compounds from older fused silica lined (FSL) canisters and that a gas-bag may be a better option.
6. Silco or FSL canisters are designed for polar or more reactive compounds. Summa canisters are electro-polished stainless steel and do not perform as well for such compounds.

□ **Gas-Bag Collection:** For gas-bags, use a vacuum chamber (or "lung box") to collect samples. This avoids passing soil gas through a pump and possible bias due to cross-contamination from pump and/or pump leakage.

□ **Determine Sample Volume, Flow Rate and Time**: Based on the expected concentrations and required detection limits, determine the sample volume, flow rate and time (duration) requirements. The sample flow rate should be checked in the field.

□ **Determine Pump Requirements**: For sorbent tubes, use personal air sampling pump (e.g., SKC 224-PCXR4 or Gillian[2]) air sampling pump with low-flow adapter calibrated to required flow rate (generally 50-200 ml/min). For gas-bags, an air sampling pump is also suitable except when there are high vacuums (greater than about 20 inches water), which may occur when collecting soil gas samples from low permeability soil. Use a more powerful pump such as a SKC QuickTake 30 pump to collect bag samples for vacuums greater than about 20 inches water column. No pump is required for canister collection.

□ **Select Sampling Train Materials**. The sampling train should be constructed of inert and non-porous materials. Stainless steel and Fluorinated ethylene propylene i.e. Teflon® or Teflon-lined tubing are acceptable materials. Nylaflow® (nylon) tubing is acceptable for most volatile chemicals, but excessive sorption of naphthalene onto Nylaflow has been shown and thus it should not be used for this or similar compounds. No glues, tape, or other materials that could emit volatiles should be used. New materials should be used for sampling train at each probe, unless stainless steel tubing is used where cleaning is demonstrated through testing of blanks. Couplings should be air-tight Swagelok™ compression-fittings or barbed-fittings. When barbed-fittings are used, push tubing over a minimum of three barbs. Slip fittings should not be used.[3]

□ **Identify Field Quality Control (QC) Procedures and Samples**: Identify field QC procedures including flow and vacuum check, leak testing and purging requirements. Determine sample container certification and required field QC samples including duplicate samples, equipment blanks and trip blanks. If practical, test QC samples early in field program so that adjustments can be made if warranted. Make arrangements for the laboratory to supply ultra-high purity (UHP) nitrogen to field, if needed. Experience indicates canisters occasionally leak (evidenced by vacuum lower than about 27 inches Hg when canisters arrive on site) or are in poor condition (stripped threads) and gas-bags leak (evidenced by deflation after sampling). Therefore, order extra canisters and bags.

□ **Determine Purge Volumes**: The purge volume should take into account filter pack and may be calculated from:

[2] Note all references to products are for convenience only and not an endorsement.
[3] Vacuum grease is sometimes applied by the manufacturer to gas-bag valves, but should not be used for low-level analysis.

$$\text{Probe Volume } (cm^3) = \pi*1/4*[\theta_a*(D_B^2-D_P^2)*L_S + D_P^2*L_P + D_T^2*L_T]$$

where θ_a is air-filled porosity (0.25 is a reasonable assumption), D_B is the diameter borehole (cm), D_P is the diameter of the probe (cm), L_S is the length of sand pack (cm), L_P is the length of the probe (cm), D_T is the diameter of the tubing (cm), L_T is the length of the tubing (cm).

- □ **Temporal and Seasonal Considerations**: Consider possible temporal and seasonal variations when determining when to sample. Do not conduct sampling during and after moderate to heavy rain (i.e., greater than about 0.5 centimetre [cm]). Generally wait at least one day or longer depending on soil type.

- □ **Cold Weather Considerations**: Most field instruments and pumps are not designed to operate when temperatures are below freezing. Keep field detectors in an environment where temperature > 0^oC. Keep pumps warm in insulated coolers or insulated lunch bags with heat packs. If warm soil gas cools in tubing, condensation may occur and adversely affect the sampling and analysis process (e.g., reduced retention for sorbent tubes). Watch for signs of condensation. Sorbents and tubing may also be kept warm in insulated lunch bags (or possibly other ways) to reduce condensation. Do not collect soil gas samples from frozen ground. Such samples are not expected to be representative due to reduced volatilization.

- □ **Schedule the Work**: Conduct probe performance testing, leak tests and field screening prior to collecting samples for laboratory analysis. It may be advantageous to screen all probes then return to select probes to collect samples for laboratory analysis. For holding time sensitive analyses, coordinate shipping and receipt by laboratory (consider impact of weekends). Helium transport requires training and typically placarding, although small canisters may be exempt.

- □ **Assemble equipment needed.** Some of the equipment needed for soil gas sampling is: 1) vacuum chamber, 2) air-sampling pump, 3) flow meter (e.g., primary flow calibrator), 4) vacuum measurement devices (digital manometer or magnehelic gauges), 5) sampling train, 6) sampling containers (gas-bags, sorbent tubes, canisters), 7) field instruments, and 8) calibration and leak tracer gases.

Material Handling, Storage, Decontamination and Field Blanks:

- ❑ **Handling and Storage**: Sampling train materials should be wrapped in plastic or in food-grade plastic bags during delivery to site and storage on site. Use care when storing and handling materials and sampling devices to avoid contamination. Wear clean, medical grade nitrile gloves for sampling. Do not expose sampling materials to vehicle exhaust or other point sources of contamination.

- ❑ **Decontamination:** For ***laboratory analysis*** (i.e., ppbV levels), it is strongly recommended that dedicated new materials be used for each new probe (there

is generally no need to decontaminate new materials properly handled).[4] Sampling materials to be re-used for laboratory analysis should be thoroughly washed with a hot-water soap solution followed by tap-water and distilled-deionized water rinses. Retractable screen type soil gas probes may additionally require prior cleaning with brushes to remove soil. The valves and fittings should be completely dry prior to reuse, as water droplets could affect soil gas concentrations. A low-temperature bake (e.g., 70°C) may be used to dry stainless steel and brass. Cleaning procedures should be verified through testing of equipment blanks (see below). When sampling train materials are to be re-used for ***field screening analysis*** (i.e., ppmV levels), decontamination of relatively inert materials such as stainless steel, brass and Teflon may not be required; however, blank samples should be tested.

- **Field Blanks for Field Screening (ppmV levels)**: Where field screening and ppmV analyses are performed, collect and test a field blank to verify that sampling materials are clean. Draw ambient air through the probe (prior to installation)[5] and sampling train into a gas-bag and measure the concentration in the bag using a PID. If there is any detectable concentration, the probe and sampling train should be cleaned or replaced. The background PID levels in ambient air must be non-detect for this procedure to apply.

- **Field Blanks for Laboratory Analysis (ppbV levels)**: When re-using soil gas probes and/or sampling trains where laboratory analyses (i.e., ppbV levels) are to be performed, the following procedure should be followed: (i) connect a Summa canister containing zero ultra-pure air or nitrogen to one end of the sampling train, (ii) connect the other end of the sampling train to an evacuated Summa canister, (iii) simultaneously open both canister valves and then fill the canister at 100 to 200 ml/min. Submit the canister sample for laboratory analysis. A sorbent tube may also be used to obtain a field blank sample; however, the regulator and valve on the gas canister must be capable of delivering a constant flow rate between 100 and 200 ml/min, and a flow gauge must be used to measure the flow rate. A minimum of 10 percent of the probes and sampling trains that are re-used should be tested using the above procedure.

  When using new materials that are properly stored and handled, there is less potential for cross-contamination during sampling. The testing of equipment blanks of new materials is considered optional.

Probe Equilibration and/or Probe Development

- ☐ Soil gas probes should be developed by removing air entrained during installation and/or allowed to re-equilibrate via diffusion prior to sampling.

[4] Sampling tips or implants in contact with cutting oils should be soaked in isopropyl alcohol and then rinsed as described above.
[5] This procedure is generally only applicable to temporary probes

- A minimum of three probe volumes of air (consisting of the probe volume, tubing volume and air-filled pore volume of the sand pack) should be removed during development. Otherwise, the probe should be allowed to re-equilibrate prior to sampling. Development also provides for "conditioning" of PVC probes (studies have shown some sorption onto PVC occurs).

- The time required for equilibration will depend on the disturbance caused during installation. Recommended minimum equilibration times are provided in Table 2.

Table 2: Recommended Minimum Equilibration Times

Probe Type	Equilibration Time
Driven probes (AMS, Geoprobe PRT)	20 minutes
Probes installed in small diameter borehole (<50 mm), no fluids (air or water) used for drilling	1 day
Probes installed in larger diameter borehole (>50 mm), no fluids (air or water) used for drilling	2 days
Probes installed in hydro-vac hole (not recommended, but may be H&S or client requirement)	1 week
Probes installed in borehole where fluids (air or water) used for drilling (not recommended)	Conduct field screening over several weeks until concentrations stabilize

Flow and Vacuum (Probe Performance) Check:

- The probe performance test is used to verify that an acceptable gas flow rate and vacuum can be achieved and the calculated soil-air permeability is consistent with geologic materials in which the probe is screened. If the vacuum is much higher than expected, the probe may be plugged or within the saturated zone.[6] If the vacuum is much lower than expected, there may be short circuiting. When interpreting results, recognize that soil moisture (and hence precipitation events) affects soil-air permeability.

- The flow and vacuum measurements may be used to estimate the soil-air permeability using mathematical models for soil gas flow (Appendix I).

[6] Criteria for typical vacuums for different geologic media are being developed.

- □ The flow and vacuum check is conducted by measuring the vacuum at the desired flow rate. If the vacuum exceeds 10 inches of water, a lower flow rate should be used to reduce the vacuum, where practical (note that it is acceptable to obtain samples at higher vacuums). A vacuum of greater than about 20 inches water column also requires a specialized pump.

- □ As a minimum, allow the vacuum generated during performance testing to dissipate before collecting a soil gas sample for analysis. If a relatively large volume of soil gas is removed or high pumping rate is employed during the performance test (which may cause a local disequilibrium), the probe should be allowed to re-equilibrate using similar criteria described above.[7]

Leak Tracer Test and Shut-in Vacuum Test

- □ The purpose of the leak tracer test and shut-in vacuum test is to verify that leakage of the sampling train is within acceptable limits. The leak tracer test may be conducted during purging as described below (see Figure 5 for photo of sampling kit).

- □ Conduct leak tracer test using helium at each new probe being sampled, and at 10% of probes for each subsequent monitoring round. High purity or "zero" grade helium that >= 99.995% pure is recommended. Industrial or balloon grade helium is not recommended, but if required to use, consider collecting a helium sample for laboratory analysis.

- □ Place plastic shroud over the sample probe and valve and slowly fill the shroud until the helium concentration measured using ppm level detector (e.g., Dielectric MGD-2000) is a minimum of 10% ("top up" helium during test, as necessary). Purge probe, then collect soil gas sample in gas-bag and measure helium concentration. The purge volume prior to collection of sample should be as a minimum equal to or greater than sample volume that will subsequently obtained for laboratory analysis. Quantify leakage as follows:

  Leakage (%) = He Conc. Soil Gas/ He Conc. Shroud * 100

- □ If Leakage > 2%, fix or replace probe or sampling train and re-check leakage.

- □ Conduct shut-in vacuum tests twice daily by creating at least 10 inches water column vacuum in sampling train. Close valve at probe and furthest down-stream end of sampling train and monitor the change in vacuum over time. There should be no more than 5% loss in vacuum over 5 minutes.

[7] The sampling radius may be used to evaluate the potential for breakthrough of atmospheric air. The radius is calculated assuming the soil gas sampling zone is a sphere. The equation for volume of sphere is; 4/3 *PI *R^3. The radius (cm) is calculated as follows; R = [3 * V / (4*PI*θ_a)]^0.33 where V is sample volume (cm^3) and θ_a is the air-filled porosity. Assuming sample volume of 10L and air-filled porosity of 0.1, a radius of 28 cm is calculated.

Purging and Sampling:

General requirements are described below while specific considerations for canisters and sorbent tubes are described in subsequent sections. Schematics showing different sampling configurations are shown in Figures 2 and 3. The use of smaller diameter probes (generally equal or less than 19 mm diameter) is recommended to reduce purge volumes and sampling times.

1. The valve on the probe should be closed at all times unless the probe is being purged or sampled.

2. Measure the static pressure between the probe and ambient air using a manometer with resolution of 0.01 inches H_2O. The static pressure may provide useful information on possible pressure gradients and advective soil gas transport (optional).

3. Assemble the sampling train and check that fittings and connections are tight. Use the minimum length of tubing practical, to minimize the sorption of chemicals to the tubing.

4. For subslab sampling, purge gases should be vented outdoors to avoid contamination of indoor air.

5. Start leak tracer test as described above.

6. Connect the sampling train and equipment (pumps, gauges, *etc*) to the probe, open valves and purge at a nominal rate of 20 to 200 ml/min. If a larger diameter probe is sampled (e.g., monitoring well screened across the water table), a purge rate of up to 5 L/min may be used. Record the vacuum during purging and reduce the flow rate, as practical, if the vacuum is greater than 10 inches water column.

7. Purge volume criteria options are as follows:

   i. ***Basic Method***: Purge three probe volumes including the filter pack pore-space and then collect sample. Purge minimum 1 L of soil gas. If there is data on probe stabilization volumes, subsequent purge volume calculation may ignore filter pack volume if supported by data.

   ii. ***Purge Stabilization Test***: Collect sample after soil gas concentrations (PID, oxygen, carbon dioxide) stabilize (i.e., consecutive readings are within approximately 10 percent). Obtain minimum of three samples at approximately one purge volume increments. This method is considered best practice in all cases, and should be conducted when soil gas samples are collected from monitoring wells or larger diameter probes (greater or equal to 25 mm) or from probes installed in holes drilled by air rotary, sonic or hydro-vac holes.

   iii. ***Initial Purge Volume Test***: Initially conduct purge volume test on subset of probes and obtain soil gas concentrations after one, three and ten purge volumes. Desired purge volume typically corresponds to the purge volume

for which maximum PID concentrations are obtained. Use this purge volume for subsequent probes.

8. Once purging is complete, stop the flow of the pump and close the valve immediately upstream of the pump.

9. Allow the vacuum inside the probe to dissipate to atmospheric conditions (record the time for vacuum to dissipate).

10. An optional equilibration time of ½ to 1 day may be warranted depending on several factors: if large soil gas volumes (>~10 L) were removed during purging and leak checking, if probes are shallow (< ~1 m) because large purge volumes increase the potential for atmospheric air reaching the probe, or if there are layered low permeability soil deposits where there may be slow chemical desorption/diffusion out of soil deposits.

11. Once atmospheric conditions have been reached, connect the sampling device to the sampling train, open the valve and collect the sample at a flow rate between 20 and 200 ml/min. The procedure will depend on the sample collection method: (i) *Gas-bags*: collect sample using vacuum chamber using same sample train used for purging; (ii) *Evacuated canister*: close valve at probe, disconnect tubing from pump, and connect to canister (minimize tubing length); (iii) *Sorbent tube*: close valve at probe, disconnect tubing from pump, place sorbent in-line (minimize tubing length upstream of sorbent tube).

12. If multiple samples are required, allow vacuum to dissipate between sample collection. Collect samples for different analyses in the same order using the same procedure.

Screening Using Field Instruments:

1. Be aware of the capabilities and limitations of detectors when selecting field instruments and sample volume requirements (Table 3). Key points are:

    a) Use appropriate detector for contaminant type. Photoionization detectors (PID) are appropriate for a broad range of organic vapours (and some inorganics) depending on the lamp energy, combustible gas detectors are often used for petroleum hydrocarbon sites, and multi-gas detectors are used when there are potential concerns associated with biogenic gases.

    b) Be aware of potential cross-sensitivity and bias. For example, infrared methane detector response is biased upward by other light hydrocarbons and solvents, and helium detector response is biased upward by methane (see Golder SABCS guidance). For infrared detectors, take readings with and without charcoal filter.

    c) Calibrate and bump-test instruments in accordance with manufacturer's specifications. Keep calibration records in project files.

Combustible gas detectors when used for petroleum hydrocarbon sites should be calibrated to hexane, and when used for landfill or similar sites should be calibrated to methane. Record whether methane elimination switch is off or on. Note that methane elimination does not completely eliminate methane response and also reduces the response slightly for other compounds.

d) Combustible gas detectors (catalytic) are inaccurate at high hydrocarbon concentrations when concentrations approach and exceed the lower explosive limit of methane.

e) Be aware of response times for different detectors, which have implications for minimum sample volumes.

2. Use a new gas-bag for each location where gas-bags are submitted for laboratory analysis. When gas-bags are used for field screening they may be re-used, but should be cleaned prior to re-use by filling the bag with ambient air and then emptying the contents of the bag two times. After cleaning, measure the air concentrations in the bag using a PID. If the concentrations are not representative of ambient air (PID should read zero ppm), fill and empty the bag another three times. If the bag is still not clean after five cleaning cycles, discard the bag.

3. Collect soil gas samples in a 1-litre gas bag using a vacuum chamber to eliminate the potential for cross-contamination from the sampling pump.[8]

4. Field readings from gas bags should be measured within one hour of sample collection due to potential leakage and permeation.

Sampling Using Sorbent Tubes:

1. Determine the type of sorbent required, detection limits, pumps and quality control procedures and samples. The sample volume is a key parameter that is dependent on analyte and anticipated concentrations. It is often helpful to provide PID data to the laboratory and to then determine required sample volume in consultation with the laboratory. The sample volume should be sufficiently large to provide required detection limit but less than the safe sampling volume (SSV) for the sorbent to avoid breakthrough. The minimum sampling time to achieve a desired detection limit may be calculated as follows:

[8] Landtec GEM-2000 or equivalent may be used to collect samples directly from probes if vacuum is less than 10 inches water column. Project manager to confirm acceptability.

$t_{sample} = 1000 * (DL_{lab}) / (DL_{desired} * Q)$

Where:

t_{sample} = duration of sample in minutes

DL_{lab} = detection limit that can be achieved by laboratory in µg

$DL_{desired}$ = desired detection limit in $\mu g/m^3$

Q = sampling flow rate in L/minute

2. Calibrate pumps to desired flow rate using the type of sorbent tube that will be used. If two sorbent media are being used, samples are collected in parallel using Y-connections. Each side of the Y-connector must be calibrated separately for the specific sorbent tube used (see Figure 4).

3. Recharge the pumps fully prior to use and be aware of battery limitations as pumps used for sorbent sampling typically operate for a maximum of 8 hours on battery. If longer sampling durations are required, pumps may need to be plugged into an A/C power source.[9]

4. When samples are ready to be collected, for thermal tubes used for Method TO-17, remove caps from metal tube. For tubes used for NIOSH or OSHA methods, cut off the ends of the sorbent tube using a clean glass cutter. Cut the glass such that a 2 to 3 mm opening is created. Follow proper health and safety protocols while cutting the glass.

5. Connect the sorbent tube in-line between the probe and pump. Tubes used for Method TO-17 use Swagelok connections. Tubes used for NIOSH and OSHA methods must be connected using flexible silicon tubing to create an air-tight seal. Butt sorbent tube to sample tubing such that there is minimal contact between soil gas and the silicon tubing. Since sorbent tubes typically have a front and back section, they must be connected in the correct direction (often the tubes have an arrow indicating the direction of flow). If using more than one type of sorbent tube in parallel, be sure that the sampling tubes are in the correct location, as each side of the splitter is calibrated separately to the tube being used.

6. Once the sorbent tubes have been connected to the probe, open the valves of the sampling train and turn on the pump. Record the exact start time and stop time of the sample collection, and record the pump identification number for each sorbent tube.

7. The pump flow rate must be checked in the field during sampling since flow rates vary depending on permeability of the soil. Use a primary flow calibrator

[9] Generators are not recommended as an AC Power Source due to potential air emissions.

or similar instrument with accuracy within +/- 5 percent. The actual field flow rate should be used for calculation of concentrations.

8. After sampling is complete, stop the pump and close the valves. Disconnect the sorbent tubes and snuggly place an air-tight cap on each end of the sampling tube. Fill out label with pen (no Sharpies) and place label on tube.[10] As required, place tubes in a protective case to prevent breakage during shipping.

9. Field quality control samples should at a minimum consist of field duplicate samples and trip blanks. Sorbent tubes in series, distributed volume pair samples and equipment blanks may be required depending on project requirements.

   i. **Field duplicates**: Samples are obtained using a splitter or Y-connector provided by the laboratory and certified as clean (tubes in parallel). The flow rate for each tube should be calibrated separately and should be approximately equal for each tube. Samples may also be collected successively; however, additional variability to the sampling process may be introduced.

   ii. **Field transport blanks:** Are obtained by opening the ends of sorbent tube for a short period of time (5 minutes), leaving the tube open to atmosphere, sealing the tube and transporting the trip blank with other samples being analyzed.

   iii. **Sorbent tubes in series**: Chemical breakthrough of the sorbent is a potential issue for soil vapour due to humidity and often elevated concentrations. For NIOSH and OSHA methods, collection and analysis of the "front" and "back" of the sorbent tube is mandatory. If the concentration in the back tube exceeds 10% of the concentration in the front, breakthrough is considered to have occurred and generally results are not considered valid. For Method TO-17, there is no front and back of the tube, but two tubes may be collected in series.

   iv. **Distributed volume pairs:** The sample set-up is identical to duplicate analysis, except that samples are collected at different flow rates, intended to determine if breakthrough occurred (this method is specified in TO-17).

   v. **Equipment blanks:** Are required if non-dedicated probes or sampling train are used (see above).

   vi. **Frequency**: Field duplicate and trip blanks are recommended at a frequency of one in ten samples. If the batch size is less than 10 samples, a field duplicate and trip blank should generally still be collected.

10. Submit samples under signed chain-of-custody.

[10] While not ideal, laboratories indicate that this does not affect analysis.

Sampling Using Summa or Silco Canisters:

1. Determine the type and volume of canister required, detection limits, flow controllers, quality control procedures and quality control samples.

2. The sampling rate is regulated by either mass flow controller or critical orifice. Mass flow controllers provide for a more uniform flow rate and should be used for sampling durations longer than two hours. Critical orifices provide for a less uniform rate and may be used for durations less than two hours (mass flow controllers are also acceptable for shorter durations). Communicate to laboratory the altitude and temperature under which sampling will occur because mass flow controllers are affected by pressure and temperature and may need to be adjusted as part of controller preparation. Some controllers or orifices come with a dedicated vacuum gauge; this is useful for monitoring the flow rate during sampling, which is proportional to the rate at which the vacuum drops.

3. Prior to sampling, check the canister vacuum by attaching a vacuum gauge to the top of the canister. An oil-filled vacuum gauge is typically provided by the laboratory but often accuracy of such gauges is poor. As required, bring more accurate hand-held digital manometer to field. Prior to connecting the gauge, double check that the control knob on the side of the canister is fully closed. Using a wrench, remove the valve cap on the top of the canister, and attach the gauge. When attached correctly, it should not be possible to turn the gauge assembly (follow the laboratory instructions for tightening). After taking the reading, close the control knob tightly, and disconnect the gauge.

4. Some laboratories provide a gauge that is part of the flow controller. In this case, the sample collection begins at the same time as the vacuum is checked. Attach the canister to the soil gas probe prior to checking the vacuum. To check the vacuum, open the control knob and record the vacuum.

5. The canister vacuum should be between 27 and 30 inches mercury near sea level. As altitude increases, the vacuums measured will decrease – check with laboratory for correction to use. Typically, canisters with less than 27 inches mercury should not be used.

6. After checking the vacuum, attach the particulate filter and flow controller (unless it is attached to the vacuum gauge), also using a wrench. When attached correctly, it should not be possible to turn the flow controller assembly.

7. Connect the canister to the probe using air-tight fittings. Conduct shut-in vacuum test using hand-pump with valve, connected to t-fitting or collect canister sample under helium-filled shroud to verify air-tight connections.

8. When ready to sample, open the control knob on the side of the canister to begin sample collection, and record the start time of the sample collection.

9. After sampling is complete, check the vacuum again. There should be a residual vacuum left in the canister that ideally is between 4 and 6 inches mercury, but should be no more 10 inches mercury. For short duration soil vapour sampling (i.e., typically less than 2 hours), if there is no vacuum left in the canister at the end of the sampling process, the data is still considered valid (there is no mandatory minimum vacuum requirement in method TO-15); however, results should be flagged. For longer duration air sampling (i.e., typically 8 or 24 hours), there should be a vacuum remaining for sample to be considered valid.

10. Correct canister pressures for temperature differences using Ideal Gas Law; PV = nRT where P = pressure (atm), V = volume (L), R = gas constant (L atm / (mol·K), and T = temperature (K). This is particularly important for canisters collected at cold temperatures where at ambient temperature a residual vacuum may be measured, which may not exist when the canister air is at laboratory room temperature.

11. Fill out label with pen (no Sharpies) and attach label to canister.

12. The vacuum should be measured upon receipt by the laboratory. This data should be obtained and reported.

13. Field quality control samples should at a minimum consist of field duplicate samples. Field blanks and equipment blanks may be required depending on project requirements.

    i. **Field duplicates**: Samples are obtained using a splitter or Y-connector provided by the laboratory and certified as clean. The splitter and a single flow controller upstream of the splitter should be provided by the laboratory. Samples may also be collected successively; however, some additional variability to the sampling process may be introduced.

    ii. **Field transport blanks:** Field blank are typically collected one of two ways: 1) fill blank canister in field with ultra-pure nitrogen from second canister supplied by the laboratory using a short piece of clean Teflon tubing, or 2) handle blank in same way as other samples (measure vacuums) but do not fill canister in field and instead laboratory fills canister upon receipt. A field blank is another test of laboratory canister cleaning procedures and may be warranted depending on whether the laboratory is batch or individually certifying canisters as clean and level of quality assurance required for the project.

    iii. **Equipment blanks:** Are required if non-dedicated probes or sampling train are used (see above).

    iv. **Frequency**: Field duplicates are recommended at a frequency of one in ten samples. If the batch size is less than 10 samples, a field duplicate should generally still be collected (project manager decision).

14. Submit samples under signed chain-of-custody.

Storage and Handling of Soil Gas Samples:

1. Soil gas samples obtained using steel canisters, gas bags, glass cylinders or syringes should not be placed in a chilled cooler for transport since volatiles may condense out the vapour phase at lower temperatures. Samples should not be subjected to excessive heat.

2. Gas-bags, glass cylinders and syringes should be placed inside a container immediately after collection to avoid possible photo-oxidation reactions.

3. For sorbent tubes, cool storage (approximately 4°C) in sealed containers is required. Sorbent tubes should be stored in a sealed plastic container containing a bed of activated carbon to minimize the potential for adsorption of ambient VOCs and keep moisture away from tubes.

4. All soil gas samples should be transported in separate containers from soil and groundwater samples, and separate from pumps.

5. All soil gas samples should be submitted to the analytical laboratory under signed chain-of-custody. Additional requirements apply to “legal” samples.

6. Gas-bag samples may be shipped by air but should only be filled approximately half-full to avoid problems with pressure changes. Confirm requirements with laboratory. Ground transport is preferable if holding times can be met.

Ancillary Data:

1. Record qualitative weather conditions during sampling. This should include approximate temperature, sunshine, cloud cover, precipitation, wind (strong, moderate, slight), frost and snow cover. For subslab sampling, note the indoor temperature.

2. For cold weather or northern sampling, if possible, determine the depth of frost (or permafrost). It may be possible to estimate the depth of frost penetration through test pits or installation of thermistors in soil gas probes. Sampling of soil gas from frozen ground will likely be non-representative.

3. Obtain weather data from a nearby meteorological station. Where feasible, obtain temperature, barometric pressure, wind speed and direction, and precipitation data from three days prior to sampling to one day after sampling (to determine trends in barometric pressure).

4. Note other site conditions that could influence soil gas data including ground surface cover near probe (e.g., asphaltic pavement, concrete, condition of concrete, dirt, grass, *etc*.) and site remediation activities (e.g., operation of soil venting, air sparging, oxidation, or groundwater pumping systems) or other possible emission sources.

Documentation:

The soil gas sampling and analysis program should be documented on a Soil Gas Sampling Form and field note book. The information that should be recorded includes the sampler's name, date and time, type of probe sampled, leak tracer test results, flow rate and pressure data, purge volumes and sampling rate, field screening instruments used, pumps used, calibration data and ancillary described above. For canister sampling, the canister and flow controller identification number should be noted. Take photographs.

REFERENCES

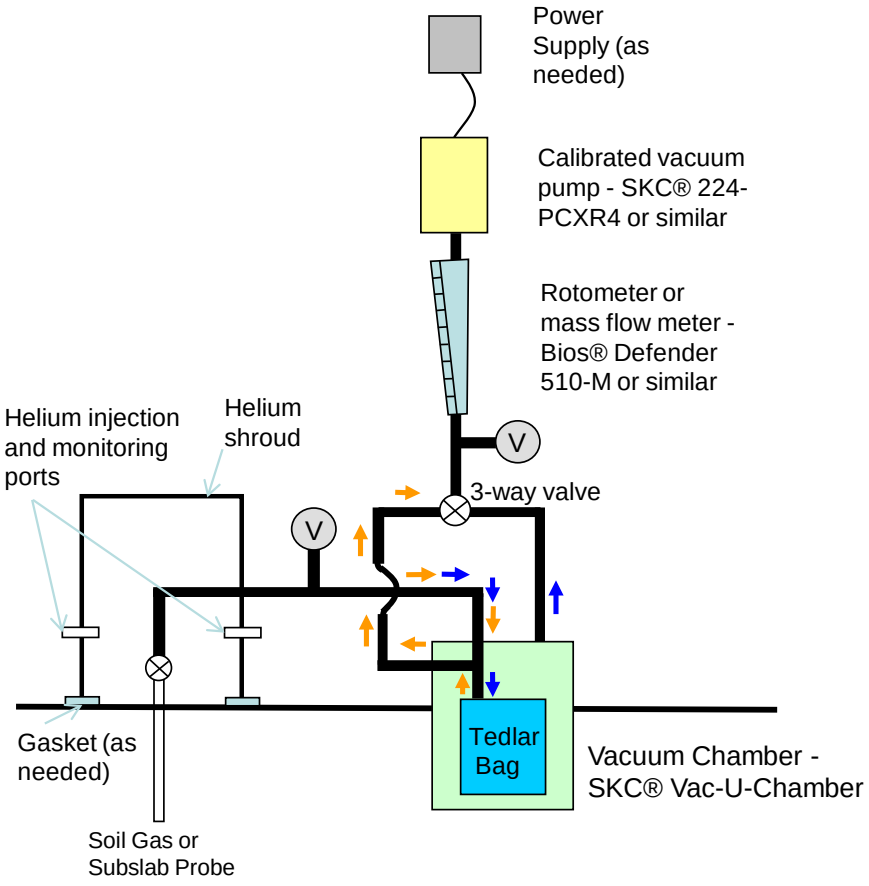
Electric Power Research Institute (EPRI). 2005. *Reference Handbook for Site-Specific Assessment of Subsurface Vapor Intrusion in Indoor Air*. Palo Alto, California, 1008492.

Hayes, H.C., D.J. Benton and N. Khan. 2006. *Impact of Sampling Media on Soil Gas Measurements*. AWMA "Vapor Intrusion – The Next Great Environmental Challenge", Philadelphia, PN, January 25-27.

TABLE 3: Common Field Detectors for Soil Gas

Instrument	Compounds Detected	Potential Advantages	Potential Disadvantages
Detector Tubes	Aliphatics, aromatics (e.g., benzene tubes), alcohols, inorganics (e.g., HCN, H_2S)	• Inexpensive • Easy to use • Immediate results • Specific compounds can be detected (although may be cross-sensitivities)	• Low sensitivity • Cross-sensitivity to other compounds • Affected by humidity, sample flow rate, temperature extremes • Limited shelf life
Portable Photo-ionization Detector (PID)	Organic vapours, most sensitive to aromatics, somewhat less sensitive to aliphatics, does not detect methane, detects some inorganics (H_2S, ammonia), response dependent on lamp energy	• Relatively inexpensive • Easy to use • Rapid detector response • Immediate results • Can obtain intrinsically safe instruments	• Low sensitivity (ppmV level), unless ppbV instrument used • Non chemical specific • Instrument response affected by humidity, cold temperatures (<0°C), dust and electrical currents (power lines) • Biased low when CH_4 levels > about 1%
Portable Flame Ionization Detector (FID)	Organic vapours, most sensitive to aliphatics, somewhat less sensitive to aromatics, detects methane	• Rapid detector response • Measures a wide range of organic vapours including methane • Less affected by humidity and dust than PIDs • Some FIDs have lower detection limits than PIDs	• Low sensitivity (ppmV level) • Non chemical specific • More operator training needed than PID, requires H_2 gas (may be shipping issues) • Instrument response may be affected by wind and cold temperatures (<0°C) • Inconsistent readings when low O_2 (< 15%) and high CO_2
Explosi-meter	Platinum catalytic detector - Any flammable gas (e.g., methane) or vapour (e.g., gasoline)	• Relatively inexpensive • Rapid detector response • Easy to use • Responds to any flammable gas, less sensitive to environmental effects than PIDs and FIDs • Generally range is 0.1 % to 100 % of LEL of methane or hexane, although ppmV instruments also available	• Not intended for very low level analysis • Non chemical specific • Inaccurate readings when O_2 less than about 12% v/v (depending on instrument) • Detector prone to aging, poisoning, moisture • Inaccurate readings when combustible gas concentrations are high (approach or exceed the LEL of methane or hexane)
Multi-gas Detector for mixed gases	Infrared, electrochemical, galvanic detectors - Landfill gases such as CH_4, H_2S, CO_2, O_2 Wide variety of options available	• Easy to use, some instruments designed to sample against vacuum • Rapid detector response • Specific gases can be detected • Infrared CH_4 detectors less prone to interference than catalytic type detectors, and cannot be poisoned	• Low sensitivity (generally % level) • May be cross sensitivities, for example, can be very significant positive bias in infrared methane concentrations when other light hydrocarbon or solvents are present • Performance dependent on type of detector
Mercury meters	Mercury	• Direct measurement device	• Low sensitivity, e.g., Ohio Lumex RA-915+ vapor Analyzer can detect Hg to 0.002 $\mu g/m^3$, Jerome 431-X can detect to 3 $\mu g/m^3$

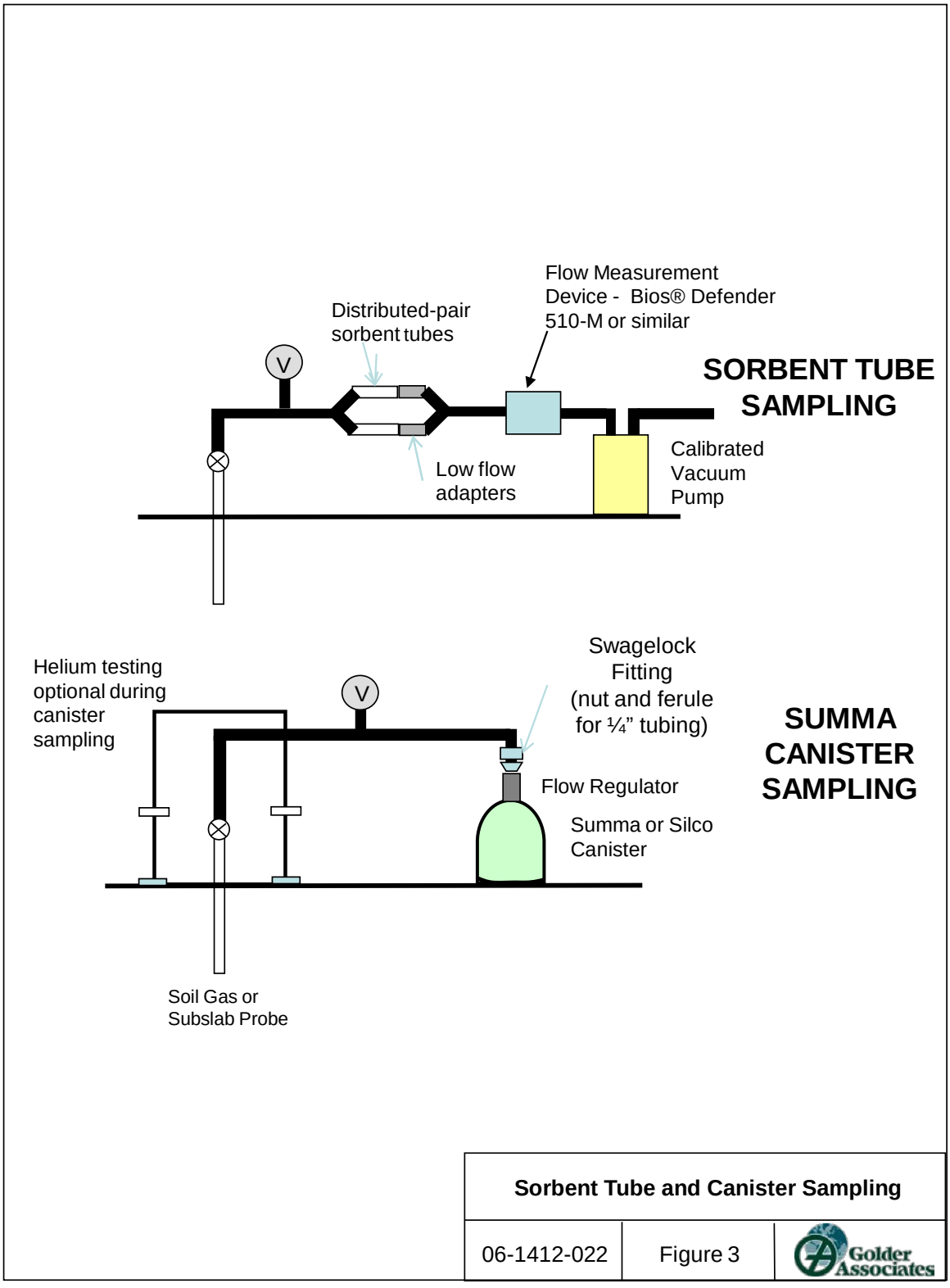
1. A field screening instrument recently introduced to the market is called a z-Nose™, an "electronic nose" that utilizes a GC and surface acoustic wave (SAW) quartz microbalance to quantify individual chemicals to ppbV sensitivity.



Legend

- Purging
- Sampling
- Valve
- V — Magnehelic Vacuum Gauge or Digital Manometer - Dwyer® Series 475 digital manometer or similar

Soil Gas Probe Purging and Collection of Tedlar Bag Sample		
06-1412-022	Figure 2	Golder Associates



Sorbent Tube and Canister Sampling		
06-1412-022	Figure 3	Golder Associates

FIGURE 4: Flow Rate Calibration of Sorbent Tubes (courtesy ALS, Vancouver, BC)

FIGURE 5: Golder Soil Gas Sampling Kit and Leak Tracer Test

Appendix I: Soil-Air Permeability Testing

Flow and vacuum measurements may be used to estimate the soil-air permeability using mathematical models for soil gas flow. Typically, the vacuum is measured for several different flow rates (i.e., step test). For a small diameter, short probe (e.g., ½ inch diameter, 6 inch long implant), a model for flow to a point (Garbesi *et al.*, 1995) may be used:

$$k = \mu Q / (S \Delta P_f) \quad [1]$$

For a larger, longer probe, a model for 1-D radial flow to a well (Johnson *et al.*, 1990) may be used:

$$Q = H^* \pi * (k/\mu) * P_p * (1-(P_{atm}/P_p)^2) / \ln(R_p/R_i)) \quad [2]$$

Re-arranging Equation 2 for the soil-air permeability yields:

$$k = Q * \mu * \ln(R_p/R_i) / [H * \pi * Pp * (1-(P_{atm}/P_p)^2)] \quad [3]$$

There are two unknowns in the above equation; the soil-air permeability and radius of influence for soil gas flow. Fortunately, equation 3 is not sensitive to the radius of influence. As a rough rule-of-thumb, the radius-of-influence can be set equal to the depth of the probe.

When there are higher pressures, soil gas flow is influenced by frictional losses at the pore walls, referred to as slip flow. There is an empirical correction, the Klinkenberg correction, which may be applied to correct for slip flow:

$$k = k_{cor} (1 + b / P) \quad [4]$$

For small diameter tubes, frictional losses may be significant and should be factored in the above calculations. For example, for ¼ inch tubing, frictional losses may become significant for flows greater than about 1 L/min. For 1 inch pipe, frictional losses will tend not to be significant at the flow rates commonly used for pneumatic testing of soil gas probes. Methods for estimating frictional losses can be found in textbooks or on-line tools (e.g., http://www.engineeringtoolbox.com/darcy-weisbach-equation-d_646.html)

Parameters

ΔP_f = pressure difference between surface & probe tip (g/cm-sec^2)
S = shape factor, for spherical pressure source, S = 4Πr; r = probe radius (cm)
k = permeability (cm^2)
k_{cor} = permeability corrected for Klinkenberg effect (cm^2)
μ = viscosity (g-cm/sec)
R_p = radius probe (cm)
R_i = radius influence (cm)
P_p = pressure probe (g/cm-sec^2)
Q = flow (cm^3/sec)
H = height well screen (cm)
P = pressure (atm)
b = empirical correction factor (0.05)
1.013E6 g/cm-sec^2 = 1 atm

REFERENCES

Garbesi, K., R.G Sextro, A.L. Robinson, J.D. Wooley, J.A. Owens, and W.W. Nazaroff. 1996. *Scale dependence of soil permeability to air: Measurement method and Field Investigation*. Water Resources Research, Vol. 32, No. 3, pages 547-560.

Johnson, P.C., C.C. Stanley, M.W. Kemblowski, D.L. Byers, and J.D. Colthart. 1990. *A practical approach to the design, operation, and monitoring of in-situ soil venting systems*, Ground Water Monit. Rev., 10(2), 159-178, 1990.

Appendix II. Unit Conversions

Soil vapour analytical results are typically reported in units of either volume per volume (e.g., parts per billion volume [ppbv]) and mass per volume (e.g., micrograms per cubic metre [µg/m^3]). The conversion of a gas concentration in ppbV units to µg/m^3 units is made assuming an ideal gas:

$$PV = nRT$$

where:

P [atm] = atmospheric pressure (1 atm)

V [L] = volume

n = moles of air

R [L-atm/mol-K] = universal gas constant = 0.0821

T [K] = standard temperature (273 K)

At standard temperature and pressure (i.e., 273 K and 1 atm), one mole of air will occupy a volume equal to 22.4 litres. For a ppbV concentration, there will be one mole of chemical per 109 moles of air. The conversion for ppbV to µg/m^3 is:

$$\left(C\frac{\mu g}{m^3}\right) = (C\ ppbv) \times \frac{1\ mol\ COC}{10^9\ mol\ air - ppbv} \times \frac{1\ mol\ air}{22.4\ L} \times \frac{273\ K}{298\ K} \times \frac{10^3\ L}{1\ m^3} \times MW\ \frac{g}{mol\ COC} \times \frac{10^6\ \mu g}{g}$$

$$\left(C\frac{\mu g}{m^3}\right) = (C\ ppbv) \times \frac{1}{22.4} \times \frac{273}{298} \times MW$$

The temperature commonly used for the above conversion is 20^{o}C (293K) since this is the temperature at which laboratory testing is conducted. Therefore, substitute 293 (or relevant temperature) in place of 298 for above equation.

SUGGESTED OPERATING PROCEDURE NUMBER 6: SOIL GAS PROBE LEAK TESTS

SCOPE This suggesting operating procedure (SOP) provides a suggested procedure for conducting leak testing of a soil gas probe and sampling train. Additional information on soil vapour sampling is provided in Volume 1 of the *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as *"Guidance Manual"*).

WHEN? A leak test should be conducted on each new soil gas probe. For each subsequent monitoring round, a leak test should generally be conducted on a subset of probes (e.g., 10 to 20%). Leak tests should also be conducted when assessing the suitability of previously installed soil gas probes that may have been damaged over time. A leak test of each sampling train should also be conducted on a regular basis by conducting a vacuum shut-in test.

WHY? Leak testing is conducted to test the seal of the soil gas probe to assess whether there is an introduction of atmospheric air into soil gas probes (referred to as "short-circuiting") and to test the connections of the sampling train. These two tests are performed using different methods as described below. Leakage may occur seals are improperly constructed over when probes or seals become damaged over time. There may be greater potential for leakage in low permeability soils when high vacuums are generated during sampling.

HOW? **Soil Gas Probe Seal**: A leak test of a soil gas probe seal is performed by applying a tracer compound at the base of the probe (typically within a shroud) at ground surface and then analyzing a soil gas sample from the probe for the tracer compound. The leakage is defined as follows:

Leakage (%) = Tracer conc. in soil gas / Tracer conc. in shroud * 100

When leakage is greater than 2%, the probe and/or sampling train should be repaired or replaced and leak check repeated prior to sampling. For gaseous tracers (e.g., helium), the starting concentration is the measured concentration under a shroud that is used to encapsulate the tracer gas (described below). For liquid tracers, the initial leak compound concentration is a theoretical estimate based on the vapour pressure of the compound at ambient temperature.

The two common types of tracers (gaseous and liquid) and basic test procedures are as follows:

- Enclosing the probe in a shroud filled with tracer gases (e.g., propane, butane, helium or sulphur hexafluoride (SF_6)); and

- Wrapping a towel soaked with a volatile liquid compound to the potential leaking areas (e.g., 2-propanol (rubbing alcohol) or pentane).

The potential advantages of using a tracer gas such as helium and SF_6 are that sensitive field instruments are available to enable real-time measurements in the field and direct quantification of potential leaks. There currently are advantages with using helium compared to SF_6 since detectors are more readily available at lower cost (one option for SF_6 is Ion Science GasCheck G-3); however, in some areas, helium is difficult and costly to obtain. By using a shroud, the connection between the probe and sampling train can also be tested. The potential disadvantages are that the test is somewhat cumbersome to perform and may require certifications and/or training for transportation and use of gases. High purity or "zero" grade helium that is at least 99.995% pure should be used. If balloon or industrial grade helium is required to be used, consider collecting a sample for laboratory analysis.

Leak tracer tests should be conducted during the purging and field testing process. An optional step is to collect canister samples for US EPA Method TO-15 analysis while conducting the leak tracer tests and requesting helium analyses from the laboratory. The laboratory should be advised prior to sample collection because helium is typically used for gas chromatographic methods and the laboratory will need to use an alternate gas.

The presence of chlorinated solvents in the soil gas sample may interfere with SF_6 measurements, so care should be taken when using SF_6.

The advantages of liquid tracers are that they are easy to apply and may be quantified to low levels using laboratory analysis (e.g., USEPA Method TO-15). The potential disadvantages include: 1) this method is generally not amenable to obtaining real-time data (unless there is field laboratory with this capability); 2) there may be liquid permeation through very small cracks in the sampling train (a process that is different than gas migration); 3) care must be taken with handling with liquid tracers since small spills can cause cross-contamination, and 4) higher concentrations of the leak compound may interfere with analyses for other volatile organic compounds (VOCs) and result in raised detection limits. As a result of these disadvantages the use of liquid tracers is not recommended. Additional information on leak testing is provided in ITRC (2007), CRWQCB (2003) and Hartman (2007, 2002).

Since helium has a number of positive features for use as a tracer compound, a more detailed procedure for leak tracer testing using helium is provided in procedure section below.

PROCEDURE

Leak Detection Test of a Soil Gas Probe using Helium:

1. Construct a shroud for conducting the leak detection test. The shroud should consist of a rigid enclosure made of an inert material such as stainless steel or rigid plastic (e.g., 10- to 20-litre pail) and should be large enough to sufficiently encapsulate and enclose the element being tested (i.e., the probe and annulus, or the probe and sampling train fittings). There should be three small openings in the shroud: two at the top, one to place the sampling train through, and the other to use as a sampling port for measuring the helium concentration inside the shroud; and one near the bottom to be used for filling the shroud with the helium gas. As needed, a soft gasket may be placed around the bottom of the shroud to create a seal against the ground surface.

2. Obtain a pressurized canister of high purity or "zero" grade helium that is at least 99.995% pure. Obtain regulator for controlling the flow of the helium. Follow appropriate health and safety procedures when transporting and working with helium gas. Note that the transportation of pressurized canisters of helium falls under the *Canadian Transportation of Dangerous Goods Regulation*. Obtain a helium detector capable of measuring concentrations ranging from 0.01% (or less) to 100% (e.g., Dielectric Technologies Model MGD-2002).

3. Place the shroud around the element to be tested and seal any significant openings in the test apparatus using inert sealing materials (e.g., bentonite or Silly Putty®).

4. Slowly fill the shroud will helium gas until the concentration of helium within the shroud reaches 20 to 30% helium (minimum recommended concentration is 10%). Take caution to fill the shroud slowly and to not over-pressurize the enclosure. Stop the flow of helium once the concentration of helium in the shroud reaches the desired concentration. "Top up" helium as required during the test.

5. Purge the soil vapour probe being tested, and collect a soil vapour sample in a Tedlar™ bag. Minimum purge volume while flooded with helium is volume of subsequent sample to be obtained for laboratory analysis. Measure the concentration of helium in the sample and calculate the Leakage as defined above. As a general rule, a Leakage less than 2% is considered to be acceptable. If the Leakage is greater than 2% repair or replace the probe.

Sample Train Leak Test

Sample Train Leak Test: There are at least three ways in which the sampling train can be tested for leaks: (i) "shut-in" vacuum test, (ii) leak tracer line test, and (iii) application of a tracer compound to connections.

A shut-in test involves creating a vacuum in the sampling train and monitoring vacuum over time to confirm that the vacuum does not dissipate. The applied vacuum should be a minimum of 10 inch H_20 water column. The decline in vacuum should be less than 10% over 5 minutes. If a pressure test is conducted, a soapy-water solution can be used to identify any couplings that may be leaking.

The leak tracer line test, described by API (2005), involves testing of sampling equipment for potential leaks using a tracer gas (e.g., diluted helium) of known concentration that is drawn through the sampling equipment at the approximate vacuum anticipated during sampling.

Liquid tracers can be applied by wrapping a towel soaked in the tracer around the fitting or an aerosol product such as difluoroethane (i.e., "Dust-off") can be sprayed over the fittings. The potential disadvantages of this method of testing the sampling train are the same as those described above with respect to testing the probe seal leak using liquid tracers.

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SUGGESTED OPERATING PROCEDURE NUMBER 7: COLLECTION OF IN SITU WATER QUALITY MEASUREMENTS

SCOPE This suggested operating procedure (SOP) is to recommend methods for collecting *in situ* water quality measurements using multiparameter meters to ensure quality control in field operations and uniformity among different field teams. Additional information regarding *in situ* water quality sampling is provided in Chapter 9 of Volume 1 of the *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as *"Guidance Manual"*). For more information refer to Section 6.5 of *Protocols Manual for Water Quality Sampling in Canada* (www.ccme.ca/files/Resources/water/protocols_document_e_final_101.pdf).

APPLICATION This SOP describes the methods for measuring *in situ* water quality parameters, such as dissolved oxygen (DO), temperature, pH, conductivity, and turbidity, in support of human health and ecological risk assessments. Methods developed and used for sampling large bodies of freshwater are in most cases applicable for sampling ocean/marine waters, but it is important to be aware of two additional considerations: consideration of timing and access due to tidal fluctuations, and collection of salinity data as well as other water quality parameters.

WHEN? *In situ* water quality measurements are typically collected for the following purposes:

1. As supporting analyses to help characterize the fate and effects of contaminants of potential concern (COPCs);
2. To assess characteristics of the water body that are relevant to regulatory targets (e.g., DO measurements are an indicator of stream health); and
3. To rule out water quality concerns as a potential cause of any adverse effects predicted by human health and ecological risk assessment.

WHY? *In situ* water quality parameters (e.g., pH) can influence the form of a chemical. Initial surveys of general water quality conditions across the study area and throughout the water column can be used to guide COPC sampling decisions. Depending on the source and nature of the COPC, specific conductance (conductivity) can be a useful surrogate to assess the degree of mixing within a water body. Additionally, *in situ* water quality can help identify stressors other than COPCs (e.g., oxygen depleted areas). Stream water quality monitoring is conducted in support of remediation activities designed to restore habitat and water quality to levels supporting healthy aquatic communities.

HOW? *In situ* water quality parameters can be monitored using a variety of water quality meters. The choice of sampling method and equipment primarily depends on:

water body characteristics (e.g., shallow *vs.* deep); receptors of concern (e.g., bottom dweller *vs.* shallow strata for valued ecosystem components [VECs]); water quality measurements required for study; discrete sample events *vs*. continuous data logging; weather/temperature conditions (e.g., excessive heat *vs.* freezing conditions); and logistical and safety limitations (e.g., sampling from bridges *vs*. wading).

TYPES

Hand-held Meter: Real-time monitoring data are typically collected by hand-held deployment at the monitoring location, boat, or platform. Hand-held meters are useful for short-term monitoring in both wadeable and deep water conditions. *In situ* assessment typically consists of a single evaluation of water quality parameters at a given stream location. When measuring DO, adequate water exchange across probe membrane is necessary for proper readings. Depending on the equipment used, movement of the probe within the water column may be required. This practice is not necessary for fast-moving water or for instruments equipped with self-contained circulation units. Adequate exchange should also be provided when measuring pH. Refer to manufacturer specifications for meter or parameter-specific procedures. *In situ* assessment can also be conducted using a meter with a mechanical/electrical winch or reel type device; use of these methods depends upon water depth and monitoring period.

Data Logger: For extended monitoring periods, data loggers may be attached to a stationary object at the monitoring location. Unattended meters must be pre-programmed to log monitoring data in the absence of the meter operator. Unattended deployments are useful for collecting data at regular intervals over extended monitoring periods. It is critical that the meter be correctly programmed following the manufacturer's procedures for unattended deployment to ensure that all necessary data are successfully logged. It is important that the logger remain in the precise position throughout the period of deployment. It is good practice to download data from the data logger at regular intervals to avoid any data loss. The field log book should include operator name, programming assumptions and parameters, the meter identifier, the date and time of initial deployment, date and time of retrieval, deployment location, and depth of assessment.

In fast moving waters, it may be necessary to attach a weight constructed of inert materials to hand-held or unattended probes. Collar and/or cable weights, which can be obtained from most equipment manufacturers, can be secured in such a way so as not to interfere with probe operation.

COLLECTION

In situ water quality monitoring entails observing monitoring data *via* a meter display unit or laptop computer during data collection. Data may be recorded in a field log book or logged to the internal memory of the meter. Logged data should be backed up in a separate location as soon as possible.

An important consideration in water quality assessment is the handling and calibration of the selected meter. Always calibrate hand-held meters and data loggers before use. Instruments are typically calibrated on a daily basis. An end-of-day meter check using known standards is useful to document lack of meter drift for each parameter. To ensure reliable operation, follow the equipment manufacturers' directions for transport, cleaning, storage, calibration, and operation. Before using, calibrate the meter probe for the specific procedure or COPC. Exercise care with multimeter probes to avoid cross-contamination of calibration standards. Immerse the meter probe into each calibration standard, thoroughly rinse in distilled or deionized water, and then remove excess water by shaking, blotting dry, or using a lint-free wipe. Conductivity standards are much more sensitive to cross-contamination/dilution than other standards. Conductivity standards are easily diluted, and this parameter may affect other parameters (specifically DO). Therefore, conductivity should always be the first parameter calibrated. The recommended order for calibration of the individual probes on a multiparameter meter is as follows: 1) conductivity; 2) pH; 3) DO; and 4) turbidity (USEPA, 2013).

Essential Information

- ☐ Conduct daily calibrations before meter use. Use standards appropriate to the meter and the study area being assessed (e.g., use pH buffers bracketing values observed in the field).
- ☐ Follow user manual procedures to ensure that all required probes are functioning. If a particular parameter is not needed, the sensor should be turned off in order to conserve battery power.

SAMPLING CHECKLIST

Equipment Checklist:

- ☐ Multiparameter meter (e.g., Horiba, YSI, Hydrolab)
- ☐ Buffers or calibration standards as specified by the manufacturer
- ☐ Probe cleaning solutions and wipes as specified by the manufacturer
- ☐ Meter cable marked with length increments for water depth
- ☐ Field notebook and permanent markers
- ☐ Global Positioning System (GPS) unit
- ☐ Non-powdered nitrile-type gloves

- ☐ Waders
- ☐ Personal protection equipment (PPE)
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Boat, if needed, and personal floatation devices for all field personnel
- ☐ First aid kit
- ☐ Camera
- ☐ Quality Assurance Program Plan (QAPP)
- ☐ Sampling and Analysis Plan (SAP)
- ☐ Health and Safety Plan (HASP)

Planning and Preparation:

- ☐ Review site-specific information, such as: regional land use patterns; site layout and topography; general information on the watershed; water bodies present; water depth; distribution, thickness, and type of sediment; nature of the shoreline; potential onshore and offshore sources of contaminants; ecological habitats and VECs; use of the study area for fishing, shellfish harvest, and boating or recreation; and current and anticipated future use of the water.
- ☐ Conduct site reconnaissance visit to aid in the preparation of the QAPP, SAP, and HASP
- ☐ Prepare QAPP
  - Define and document the data quality objectives, including specific types of Quality Assurance/Quality Control (QA/QC) samples required to meet those data quality objectives.
  - Determine and plan the field equipment quality control procedures that will be conducted for the project, including contingency plans for equipment replacement and calibration errors.
- ☐ Prepare SAP
  - Review the study area features and devise a sampling location map that provides representative coverage.

- Select monitoring methods and equipment based on study area-specific conditions and data quality objectives.

- List equipment, supplies and procedures related to sample labelling, preservation, decontamination, handling, and shipping.

☐ Prepare HASP (including an emergency plan)

- Determine and plan safety requirements specifically associated with sampling requirements based on study area features, COPCs, and personal health and safety precautions (e.g., personal floatation devices and other boat safety requirements, if applicable, chemical resistant clothing and gloves, respiratory protection, ear and eye protection , *etc.*).

- Check proper functioning and integrity of PPE, including respirators and filters.

- Determine and plan safety requirements specifically associated with wading and in-water (i.e., boat) activities.

- Hard hats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet.

- File a field plan and boat plan (if appropriate) with a land-based supervisor.

- Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should carry an adequate supply of water or other liquids for protection against dehydration in hot weather.

☐ Reserve, order, and pack all required equipment

- Obtain and prepare the necessary monitoring equipment and documents.

- For efficiency and to reduce field decontamination activities, monitoring equipment should be cleaned and decontaminated at the laboratory or field office before going to the sampling site.

- Ensure that the water meter is functional and that power supply and laptop batteries are fully charged.

- Pack water quality meter user manuals, power supply, and calibration materials for ready availability at the study area.

- Reserve a boat and make arrangements for qualified boat operator.

- Prepare a field notebook for documenting all activities related to field sampling. Information to be recorded in the field notebook includes, but is not limited to, weather conditions, progress toward accomplishing sampling objectives, deviations from the field sampling plan, sample identification numbers consistent with what is recorded on the laboratory chain-of-custody forms, sample observations, preservatives used and shipping protocol.

☐ If access to private property will be required, obtain written access permission from landowners

☐ Obtain First Aid/CPR certification and appropriate safety training for all field personnel

In Situ Data Collection:

Proper safety precautions must be observed when collecting *in situ* surface water data.

IMPORTANT: **Never compromise your personal safety or that of a field partner to collect a sample.** Always plan ahead to avoid falling and drowning hazards. Always wear appropriate safety gear such as personal floatation devices. When working with winches, cables and similar machinery, gloves, hard hats, safety glasses, and steel-toed boots are important safety items. A qualified boat operator is required for all transportation or sampling from a boat. Boat operations must conform to all requirements in federal and provincial laws.

Depending on the depth and size of a water body, a boat may be required to access sample points. If the sampling trip involves the use of a boat, the weather forecast and/or marine conditions should be obtained prior to departure. If conditions are poor, the sampling trip should be postponed.

1) All field staff must be familiar with the project HASP.

2) A clean pair of new, non-powdered, nitrile-type gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling.

3) Calibrate the meter using the standard or buffer specified by the manufacturer on an appropriate frequency consistent with the sampling plan.

4) When assessing turbidity, make note of the general levels of suspended solids in the sampling area. Turbidity measurements are sometimes imprecise, especially in the presence of high suspended solids concentrations.

5) When assessing pH, be certain the meter was calibrated with standards bracketing the range of pH values observed at the study area. In low conductivity waters or in studies especially designed to monitor pH, special buffers and additional quality assurance methods may be necessary.

6) When assessing DO concentrations, follow the manufacturer's recommended practice to ensure adequate exchange across probe membranes, account for study area elevation, ambient/water temperature, and the salinity of the water during probe meter calibration.

7) Allow *in situ* measurement values to stabilize, if feasible, prior to recording. Recording of averages may be necessary in unstable conditions (i.e., meter bounce).

IMPORTANT: Evaluation of *in situ* parameters within well mixed waters is essential to determining alterations in fate and effects of COPCs. Additionally, *in situ* parameters should be verified after collecting any samples for *ex situ* analysis.

8) Conduct *in situ* assessments following collection of surface water samples to minimize in-stream activities that may compromise sample integrity. Conduct *in situ* assessments before collection of sediment samples will release suspended matter into areas of *in situ* assessment. Assess *in situ* parameters immediately upstream of sediment collection areas, to avoid suspended sediments influencing the assessment.

9) Measure water depth to determine the appropriate depth(s) of sample collection. Meter cables can be marked to facilitate sample collection at proper depths.

10) Clean the meter and probe and decontaminate following the instructions provided by the manufacturer.

11) All information regarding the sampling event should be accurately recorded in field notes, in order to support the subsequent preparation of a sampling event report.

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SUGGESTED OPERATING PROCEDURE NUMBER 8: NEAR SURFACE WATER DISCRETE SAMPLES BY DIRECT DIP

SCOPE This suggested operating procedure (SOP) recommends methods for collecting near-surface discrete samples of surface water using the direct-dip technique. The technique is generally applied in wadeable streams or in larger bodies of water where a surface sample is required. Additional information on near-surface surface water sampling is provided in Chapter 9 of Volume 1 of the *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as *"Guidance Manual"*). For more information refer to Section 6 of *Protocols Manual for Water Quality Sampling in Canada* (www.ccme.ca/files/Resources/water/protocols_document_e_final_101.pdf)

APPLICATION This SOP describes the methods for collecting near surface discrete samples of surface water to support human health and ecological risk assessments. Methods developed and used for sampling in large bodies of freshwater are in most cases applicable for sampling ocean/marine waters, but it is important to consider timing and access due to tidal fluctuations.

WHEN? Near-surface discrete sampling of surface water is typically collected for the following purposes:

1. To measure concentrations of contaminant of potential concern (COPC) for characterizing exposure in human health and ecological risk assessments;
2. To understand patterns of COPC fate and transport that may affect exposure pathways in human health and ecological risk assessment;
3. To test the toxicity of COPCs in surface water to laboratory test organisms (e.g., fish, aquatic invertebrates); and/or
4. To evaluate the effectiveness of various surface water treatment technologies.

WHY? The purpose of this procedure is to establish a uniform method of collecting near-surface discrete samples of surface water using the direct-dip technique, primarily to assess potential human health and ecological risks associated with surface water exposure. Another example SOP for surface water sampling is available from United States Environmental Protection Agency (USEPA, 2013).

HOW? Surface water samples may be collected using a variety of methods. The choice of sampling method depends primarily on: COPC characteristics (e.g., highly soluble *vs*. insoluble); water body characteristics (e.g., shallow *vs*. deep); COPC source characteristics (e.g., whether the known or suspected source is likely to result in diffuse discharges from along the sides of a channel or bank, or whether from a single point or outfall); valued ecosystem components (VECs) (e.g., bottom dweller *vs.* shallow strata); life cycle of sensitive VEC (e.g., fish fry *vs.* adults); volume of surface water required; and logistical and safety limitations (e.g., sampling from bridges *vs.* wading, ice present *vs.* ice absent).

This SOP addresses collection of discrete samples by the direct dip method.

TYPES Discrete samples provide information on conditions at discrete times and locations. Discrete samples are the least involved, simplest type of surface water samples to collect. Discrete samples can be collected by direct dipping of sample containers into surface water, or with mechanical devices, such as van Dorn bottles. This SOP pertains exclusively to near-surface, surface water sample collection by the direct-dip technique. Additional surface water sampling methods are discussed in other SOPs. The simplicity and cost effectiveness of discrete samples make them appealing in many regards, but they provide temporally and spatially discrete information. Thus, individually, they do not account for variations in COPC concentrations that may occur over time due to changes in hydrological conditions (e.g., flow rates, discharge rates), contaminant release patterns, or other factors. Consideration of multiple discrete samples collected at different times and from different locations can, however, provide insight into many such factors. Discrete samples are well suited to identify maximum COPC concentrations in judgmental sampling programs.

COLLECTION It is imperative that materials in the sampling equipment do not contaminate the water samples or otherwise alter sample integrity. For example, plastic sample containers cannot be used to collect samples to be analyzed for sorptive trace organic compounds or organic compounds used as plasticizers (e.g., bis(2-ethylhexyl)phthalate). Polyvinyl chloride (PVC) and PVC cemented joints can be a source of chloroform and various organic compounds such as toluene, acetone, methyl ethyl ketone, and others (CCME, 1993).

Essential Information

- ☐ Identify key data uses and project goals that will influence whether single discrete samples or composited near-surface grab samples are required.
- ☐ Confirm that the direct dip technique is suitable for the surface water and analytes of interest. This technique is best suited to sampling shallow, well mixed surface water.
- ☐ Confirm that the materials of construction of the collection vessel (when not the actual sample bottle) are appropriate for the COPCs. As a rule of thumb, if the materials comprising in the portion of vessel that contacts the sample are the same as those of the sample containers, this requirement has been met.
- ☐ Determine the volume of surface water required for each sample, in order to achieve sampling objectives.
- ☐ Determine sample preservation requirements, based on the selected analytical methodology's specifications.

Generally, the use of glass, stainless steel, polypropylene tubing, and Teflon® materials that come in contact with the sample provide high quality samples. However, stainless steel can be a source of chromium, nickel, and other metals, if prolonged sample contact is allowed. The use of glass or Teflon® sample containers and contact materials is preferred for collection of samples for analysis of organic compounds, while polypropylene plastic is sufficient for collection of samples for heavy metals analyses when samples are immediately acidified upon collection. Use of plated or painted sampling equipment can contaminate samples. Consideration must always be given to supporting equipment and its associated operation in compromising sample integrity. For example, when sampling from a boat, oils and other hydrocarbons released from the engine can compromise samples. Additional guidance related to proper contact materials for sampling equipment is provided by the Ontario Ministry of the Environment and Energy (MOEE, 1996) and USEPA (2013). In particular, MOEE (1996) provides information related to assessing the temperature stability of sampling equipment. Supporting information related to chemicals associated with various types of sampling equipment and the operation of equipment is provided by CCME (1993).

SAMPLING CHECKLIST

Equipment Checklist:

- ☐ Pre-cleaned, pre-labelled (with site name, number and location) sample containers provided by the analytical laboratory, including any specialized sample container needs (e.g., volatile organic analysis (VOA) vials for

analysis for volatile organic compounds (VOCs); large volume containers for toxicity testing or treatability testing)

- ☐ Transfer container made of similar material as the sample containers
- ☐ Pole or mechanical device to hold or extend sample container
- ☐ Peristaltic pump, tubing and mesh, or syringe and filters to filter samples for dissolved metals analysis
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Non-powdered nitrile-type gloves
- ☐ Field notebook and permanent markers
- ☐ Chain-of-custody forms and seals
- ☐ Boat (depending on site conditions)
- ☐ Waders
- ☐ Personal protective equipment (PPE)
- ☐ Cooler, packing material and ice packs (for maintaining samples at ≤ 10°C in transit [but not frozen])
- ☐ Global Positioning System (GPS) unit
- ☐ Bathymetric map (detailed description of submerged terrain) or nautical charts (primarily for safe navigation) if applicable
- ☐ Garbage bags
- ☐ First aid kit
- ☐ Camera
- ☐ Quality Assurance Program Plan (QAPP)
- ☐ Sampling and Analysis Plan (SAP)
- ☐ Health and Safety Plan (HASP)

Planning and Preparation:

☐ Review site-specific information, such as regional land use patterns; site layout and topography; general information on the watershed; water bodies present; water depth; distribution, thickness, and type of sediment; nature of the shoreline; potential onshore and offshore sources of contaminants; ecological habitats and VECs; use of the study area for fishing, shellfish harvest, and boating or recreation; and current and anticipated future use of the water.

☐ Conduct site reconnaissance visit to aid in the preparation of the QAPP, SAP, and HASP

☐ Prepare QAPP

- Define and document the data quality objectives, including specific types of Quality Assurance/Quality Control (QA/QC) samples required to meet those data quality objectives.

- Determine and plan the field quality control samples that will be obtained for the project, including field duplicate samples, field blanks, equipment blanks, and trip blanks (see Chapter 3 of Volume 1 of the Guidance Manual for further details). Where feasible, test quality control samples during the site reconnaissance visit, so that adjustments can be made, as warranted.

- Duplicate samples generally should be collected at a rate of 10%, in order to assess sample location variability.

IMPORTANT: Use trip and field blanks, especially if sampling sites are subject to activity such as boat traffic or other use, or if the sampling sites have a high potential for transport of wind-borne COPCs. Ensure the laboratory analyzes matrix spikes if sampling high ionic strength waters or waters high in suspended solids.

☐ Prepare SAP

- Review the study area features and devise a sampling location map that provides representative coverage.

IMPORTANT: If the sampling objective is to obtain a sample that is representative of the water body at all depths, confirm that the surface water to be sampled with direct dip technique is shallow and sufficiently well mixed. Design the sampling and analysis plan to provide the most representative data. For example, collect samples in shallow riffle areas of small streams to ensure adequate mixing.

- Identify COPCs and expected depth of occurrence. Select sampling and analytical methods and equipment based on study area-specific conditions and data quality objectives.

- Based on project objectives, determine sample volume requirements. Verify that the selected sampling method can produce the required volumes. If not, re-evaluate options for sampling methods.

- Correspond with the analytical laboratory(ies) concerning sampling requirements, appropriate sample containers, holding times, preservation methods for individual COPCs, and shipping. Select sampling, handling, and shipping practices that will achieve those holding times, particularly for COPCs with short holding times.

IMPORTANT: Consult with the analytical laboratory concerning requirements related to sample containers, sample volume, holding times, and preservation. Develop an SAP that specifies practices that will prevent exceeding established holding times. The SAP should specify holding times for individual COPCs, especially for COPCs with short holding times that may dictate some aspects of field logistics. Coordinate with laboratory regarding shipping methods and processing.

- List equipment, supplies and procedures related to sample labelling, preservation, handling, shipment, and equipment decontamination.

☐ Prepare HASP (including an emergency plan)

- Determine and plan safety requirements specifically associated with sampling requirements based on study area features, COPCs, and personal health and safety precautions (e.g., personal floatation devices and other boat safety requirements, if applicable, chemical resistant clothing and gloves, respiratory protection, ear and eye protection , *etc*.).

- Check proper functioning and integrity of PPE, including respirators and filters.

- Determine and plan safety requirements specifically associated with wading and in-water (i.e., boat) activities.

- Hard hats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet.

- File a field plan and boat plan (if appropriate) with a land-based supervisor.

- Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should carry an

adequate supply of water or other liquids for protection against dehydration in hot weather.

☐ Reserve, order, and pack all required equipment

- Obtain and prepare the necessary sampling and storage equipment, sample containers, materials, and documents.
- For efficiency and to reduce field decontamination activities, all sampling equipment should be cleaned and decontaminated at the laboratory or field office before going to the sampling site. If possible, a separate set of cleaned and decontaminated equipment should be available for each sampling site.
- Reserve a boat and make arrangements for qualified boat operator.
- Prepare a field notebook for documenting all activities related to field sampling. Information to be recorded in the field notebook includes, but is not limited to, weather conditions, progress toward accomplishing sampling objectives, deviations from the field sampling plan, sample identification numbers consistent with what is recorded on the laboratory chain-of-custody forms, sample observations, preservatives used and shipping protocol.

☐ If access to private property will be required, obtain written access permission from landowners

☐ Obtain First Aid/CPR certification and appropriate safety training for all field personnel

Sample Collection:

Proper safety precautions must be observed when collecting surface water samples.

IMPORTANT: **Never compromise your personal safety or that of a field partner to collect a sample.** Always plan ahead to avoid falling and drowning hazards. Always wear appropriate safety gear such as personal floatation devices. When working with winches, cables, and similar machinery, gloves, hard hats, safety glasses, and steel-toed boots are important safety items. A qualified boat operator is required for all transport or sampling from a boat. Boat operations must conform to all requirements in federal and provincial laws.

1) These dangers include, but are not limited to, strong water currents, slippery substrate, roots or sharp objects beneath the water's surface that may cause a fall or other personal injury. Additional concerns include the use of heavy objects on a vessel and overhead hazards from winches if used. If sampling in water that is

greater than three feet deep, all personnel are required to wear personal floatation devices.[21]

2) Ensure that weather conditions will not create unsafe conditions during the field sampling effort.

3) All field staff must be familiar with the project HASP.

4) Samples may be collected from a boat, from a bridge, or by wading (if water is shallow). If sampling a small stream from a boat or by wading, sample from downstream to upstream locations to minimize the potential for sample contamination due to sediment re-suspension. If sampling from a bridge, collect samples from the upstream side of the bridge to minimize the influence of any trace constituents (e.g., metals) associated with road runoff from the bridge.

5) Take precautions to minimize disturbance of the surface water (e.g., propeller wash) prior to sampling.

6) If sampling is supported by assessment of *in situ* parameters (pH, dissolved oxygen, *etc*.), assess such parameters following the procedures outlined in the applicable SOP. Conduct *in situ* assessments after collecting surface water samples, in order to minimize in-stream activities that may re-suspend sediment and associated chemicals that may compromise sample integrity. Collect surface water samples before collecting sediment samples.

7) Upon arriving at a sampling station, face upstream and allow sufficient time for any re-suspended sediment to travel downstream of the sample location. When sampling from a boat, collect all samples from the upstream side of the boat and upwind of the motor to minimize sample contamination from metals and/or petroleum hydrocarbons associated with boat operation and gas fumes. Allow sufficient time for fumes or oils associated with boat operation to clear from the area. Note that sample containers affixed to poles or other mechanical devices can be used as a direct-dip sampling technique, provided that other sampling guidelines outlined herein are followed.

8) A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling.

[21] DFO and other organizations may have their own safe work practices and requirements for working near or in water. Project-specific safety requirements should be researched and documented in the HASP.

9) For samples collected for analytes that do not require chemical preservatives, slowly submerge the open sample container (open end facing upstream) under the water surface upstream of the field technician's location, cap the container, and shake the sample container to rinse all inside surfaces of the sealed container. Discard the rinse water downstream of the sample location, allowing time for the water to disperse. Again facing upstream, slowly submerge the sample container (open end first) approximately 0.1 metre (m) below the water surface (or one-half the total depth of water in shallow streams) and allow it to fill with water, sealing the sample container while it is still under water. Avoid skimming the surface unless specifically required as part of project-specific goals. As soon as practical, place the sample(s) on ice. If multiple sample containers are to be filled at the same location, fill all containers while minimizing disturbance before exiting the site to place samples on ice.

10) For analytes requiring samples to be collected in containers with chemical preservatives, use a clean transfer container, of the same material as the sample container, to collect the water sample in the same manner as described above (including rinsing the transfer container), and transfer the sample directly to the sample container with the chemical preservative.

11) Fill additional sample containers following the above procedures to obtain the required sample volume. Gently shake sample bottles with preservatives to provide full mixing of the sample and preservative.

12) Measure the water depth and conduct *in situ* analyses, if applicable.

13) Document sample location using a GPS unit, or using manual measurements to a known location (e.g., indicate sample location on a topographic map).

14) If this sampling methodology is to be used to collect samples for VOC analysis (using either stainless steel or Teflon® equipment), samples should be collected with as little agitation or disturbance as possible. The VOA vial should be filled to the top and without bubbles or headspace after it is capped. If a bubble or bubbles are present, the vial should be refilled. The VOA vial may be either preserved with sodium bisulphate or hydrochloric acid or they may be unpreserved. Note: unpreserved samples have shorter hold times and may be subject to biodegradation, thus a preservative is used unless the following situation occurs. If the surface water sample contains a high concentration of dissolved calcium carbonate, there may be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles. This will render the sample unacceptable. In this case, unpreserved vials should be used and arrangements must be confirmed with the laboratory.

15) Filtering is required for samples designated for dissolved metals analysis, but not for those designated for total metals analysis. For those samples designated for dissolved metals analysis only, prior to sample shipment and as soon as possible after collection, filter samples using a peristaltic pump or hand pump and 0.45-micron mesh filter. Ideally, samples are filtered in the field at the collection site immediately after sample collection. Place the resulting filtrate into a pre-preserved sample container, labelled for dissolved metals analysis.

16) Label each sample with the following information: date, time of sampling, sample ID, analytical method and/or parameter group(s) to be analyzed for, sampler initials, and method of preservation (if any).

17) Follow proper procedures related to holding conditions (i.e., storage temperature > 0 to 6°C), chain-of-custody, and shipping.

18) A description of how the samples were packed in the field, what preservatives were used, and how they were shipped to the laboratory should be recorded.

19) Follow the decontamination procedures outlined in the sampling and analysis plan to clean equipment and dispose of all expendable materials appropriately. In general, a mild, surfactant detergent is sufficient. If organic metals or other inorganic COPCs are present, a 10 percent nitric acid wash solution is necessary for proper decontamination. If organic COPCs are of concern, an acetone wash with water rinse is needed. Note: Do not clean plastic or polyvinyl chloride sampling devices with acetone or other organic solvents that will compromise the structural integrity of the sampling device.

20) All information regarding the sampling event should be accurately recorded in field notes, in order to support the subsequent preparation of a sampling event report.

REFERENCES

Canadian Council of Ministers of the Environment (CCME). 1993. *Guidance Manual on Sampling, Analysis, and Data Management for Contaminated Sites Volume I: Main Report*. PN 1101. CCME National Contaminated Sites Program. December.

Ontario Ministry of the Environment and Energy (MOEE). 1996. *Guidance on Sampling and Analytical Methods for use at Contaminated Sites in Ontario*. Standards Development Branch. Toronto, Ontario. December.

U.S. Environmental Protection Agency (USEPA). 2013. *Operating Procedure for Surface Water Sampling*. Region 4, U.S. Environmental Protection Agency, Science and Ecosystem Support Division, Athens, Georgia. SESDPROC-201-R3. February 28, 2013. Available at; http://www.epa.gov/region4/sesd/fbqstp/

SUGGESTED OPERATING PROCEDURE NUMBER 9: SURFACE WATER DISCRETE SAMPLES WITH MECHANICAL COLLECTION DEVICES

SCOPE This suggested operating procedure (SOP) recommends methods for collecting surface water samples using mechanical discrete sample collection devices. This SOP describes the methods to be followed to collect discrete surface water samples from waters greater than approximately 1.5 metres (m) in depth, referred to throughout this SOP as subsurface water samples. The technique is applied when subsurface samples are needed, often at multiple depths. Such sampling is typically conducted from a boat, bridge, or other platform using a mechanical sampling device, such as a van Dorn bottle or Kemmerer sampler. Additional information on surface water sampling is provided in Chapter 9 of Volume 1 of the *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as "*Guidance Manual*"). For more information refer to Section 6 of *Protocols Manual for Water Quality Sampling in Canada* (http://www.ccme.ca/files/Resources/water/protocols_document_e_final_101.pdf)

APPLICATION This SOP describes the methods for collecting discrete samples of subsurface water using mechanical collection devices for the purposes of chemical, toxicological, and/or treatability analysis in support of human health and ecological risk assessments. Methods developed and used for sampling in large bodies of freshwater are in most cases applicable for sampling ocean/marine waters, but it is important to consider timing and access due to tidal fluctuations.

WHEN? Discrete subsurface water samples are typically collected for the following purposes:

1. To measure concentrations of contaminants of potential concern (COPCs) for characterizing exposure in human health and ecological risk assessments;
2. To understand patterns of COPC fate and transport that may affect exposure pathways in human health and ecological risk assessment;
3. To test the toxicity of COPCs in subsurface water to laboratory test organisms (e.g., fish, aquatic invertebrates); and/or
4. To evaluate the effectiveness of various subsurface water treatment technologies.

WHY? Subsurface water sampling may be conducted to: assess potential exposures (based on COPC concentrations) and effects (based on toxicity testing); delineate chemical distributions; determine the effectiveness of different treatment technologies; and evaluate progress toward remediation requirements.

HOW? Subsurface water samples may be collected using a variety of methods. The choice of sampling method depends primarily on: COPC characteristics (e.g., highly soluble *vs*. insoluble); water body characteristics (e.g., shallow *vs.* deep); valued ecosystem components (VECs) (e.g., bottom dweller *vs*. shallow strata); COPC source characteristics (e.g., whether the known or suspected source is likely to result in diffuse discharges from along the sides of a channel or bank, or whether from a single point or outfall); life history stage of sensitive VECs (e.g., fish fry *vs*. adults); logistical and safety limitations (e.g., sampling from bridges *vs.* from a boat, ice present *vs.* ice absent); and sample volumes required.

TYPES **Kemmerer and van Dorn Samplers:** These devices collect discrete samples at specified depths indicated by calibrated ropes affixed to the sampling device. The Kemmerer sampler is a cylinder with stoppers at both ends. The ends of the sampler are open while the sampler is lowered in a vertical position, allowing free passage of water through the cylinder. The van Dorn sampler is a similar device that is deployed vertically, in a horizontally-oriented position. For both sampler types, a messenger weight is sent down the rope when the sampler is at the designated depth, causing the stoppers to close the cylinder and collect the sample. Water is transferred into sample containers through a valve. Samplers constructed of plastic or rubber cannot be used for sampling all volatile and extractable organic compounds. Some newer devices are constructed of stainless steel or Teflon®, or are Teflon®-coated. Teflon® and Teflon®-coated devices are acceptable for most COPCs (Caution -Teflon may not be suitable for the collection of samples requiring analysis for perfluorinated sulphonates and carboxylates (PFOS and related compounds).

Kemmerer and van Dorn samplers are relatively simple to operate and are sufficiently rugged to be used in a wide range of sampling conditions. They are most often used to collect subsurface samples in lakes and ponds, but can also be applied to calm, deep-water habitats of rivers, streams, and marine environments. They are almost always deployed from a boat. They are generally ineffective in fast-water habitats because their depth of deployment cannot be accurately assessed. Furthermore, the inability to maintain a set position in the water column precludes consistent triggering of the sampling device by the messenger.

Peristaltic Pump: This device can be effectively used to sample a water column or very shallow areas where large sampling devices or direct dip methods are not feasible. The tubing intake is positioned in the water column at discrete depths by means of a conduit or weight. To weigh down the tubing, inert materials are tied to the tubing so that it is submerged below the inlet of the tubing. Lead or metallic weights should not be used when collecting metal samples. Vertical composite samples also may be collected by moving the tubing intake at a constant rate vertically up and down the water column. Prior to collecting the first sample,

several tubing volumes of water are pumped through the system to flush the tubing. For sampling purposes, pump volumes should not exceed 0.5 litres per minute (L/min) (USEPA, 1996) to avoid influence from outside the desired discrete sample location. Individual sample containers are filled *via* the discharge tube, while the inlet tube is held in the water column. The discharge tube should not touch the sample container. This procedure enables controlled filling of sample containers to allow for headspace or addition of preservatives.

Samples collected for volatile organic compound (VOC) analysis cannot be collected directly from the peristaltic pump discharge because the water has passed through the silastic® pumping mechanism, which potentially purges VOCs from the water sample. If a peristaltic pump is used for VOC sample collection, the sample must be collected by running the pump for several minutes, with the submerged inlet tube at the desired depth to fill the tubing with water representative of that interval. The pump is then turned off and the inlet tube is capped, pinched, or sealed prior to removal from the water column to avoid loss of sample. The pump speed is reduced to a slow pumping rate and the pump direction is reversed so that water is removed from the pump head tubing. The inlet tube is re-attached. With the pump still running in the reverse direction, water is pumped from the inlet tube into the volatile organic analysis (VOA) vials; care must be taken to prevent any water that passed through the pump head tubing from being incorporated into the sample. It is critical that the water sample collected for VOC analyses does not pass over the rollers of the peristaltic pump, which would risk sample purging. It is also important that sufficient tube length is provided to allow collection of the required sample volume. Additional information regarding the proper use of peristaltic pumps with VOCs is described in the U.S. Environmental Protection Agency's *Operating Procedure for Surface Water Sampling* (USEPA, 2013).

Double Check-Valve Bailers: If the data requirements do not necessitate a sample from a discrete interval of the water column, samples may be collected using double check-valve bailers. Bailers with an upper and lower check-valve can be lowered through the water column, and water is continually displaced through the bailer until the desired depth is reached, at which point the bailer is retrieved. Upon retrieval, the two check-valves seat prevent water from escaping or entering the bailer. The degree of water displacement through the body of the bailer depends upon the check-valve ball movement, which allows water to flow freely through the bailer body. A bailer is acceptable when a mid-depth sample is required. This technique may not be successful in strong currents.

COLLECTION It is imperative that materials in the sampling equipment not contaminate the water samples or otherwise alter sample integrity. For example, plastic sample containers cannot be used to collect samples to be analyzed for sorptive trace

organic compounds or organic compounds used as plasticizers (e.g., bis(2-ethylhexyl)phthalate). Polyvinyl chloride (PVC) and PVC cemented joints can be a source of chloroform, and various organic compounds such as toluene, acetone, methyl ethyl ketone, and others (CCME, 1993).

Generally, the use of glass, stainless steel, polypropylene tubing, and Teflon® materials that come into contact with the sample provides high quality samples. However, stainless steel can be a source of chromium, nickel, and other metals, if prolonged sample contact is allowed. The use of glass or Teflon® sample containers and contact materials is preferred for collection of samples for analysis of organic compounds, while polypropylene plastic is sufficient for collection of samples for heavy metals analyses when samples are immediately acidified upon collection. Use of plated or painted sampling equipment risks sample contamination.

Consideration must always be given to supporting equipment and its associated operation in compromising sample integrity. For example, when sampling from a boat, oils and other hydrocarbons released from the engine can compromise samples. Additional guidance related to proper contact materials for sampling equipment is provided by the Ontario Ministry of the Environment and Energy (1996), particularly related to assessing the temperature stability of sampling equipment. Supporting information related to chemicals associated with various types of sampling equipment and equipment operation is provided by CCME (1993).

Sampling devices used in estuarine and marine environments should be constructed of materials compatible with these more corrosive waters.

Essential Information

- ☐ Confirm that the materials of construction of the sample device are appropriate for the COPCs. As a rule of thumb, if the materials comprising the portion of the sampling device that contacts the sample are the same as those of the sample containers, this requirement has been met. Confirm that sample containers, preservatives, and holding times are appropriate for the COPCs.
- ☐ Use equipment blanks for sampling devices, especially if their appropriateness for a specific analyte has not been previously demonstrated.

SAMPLING CHECKLIST

Equipment Checklist:

- ☐ Boat (depending on site conditions)
- ☐ Personal floatation devices for all field personnel
- ☐ Personal protection equipment (PPE)
- ☐ Mechanical discrete sampler, such as a van Dorn or Kemmerer bottle
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Pre-cleaned sample containers provided by the analytical laboratory, including any specialized containers (e.g., VOA vials for analysis of VOCs; large volume containers for toxicity testing or treatability testing)
- ☐ Clean transfer container
- ☐ Peristaltic pump, tubing and mesh or syringe and filters to filter samples for dissolved metals analysis
- ☐ Field notebook and permanent markers
- ☐ Non-powdered nitrile-type gloves
- ☐ Garbage bags
- ☐ Global Positioning System (GPS) unit
- ☐ Bathymetric map (detailed description of submerged terrain) or nautical charts (primarily for safe navigation) if applicable
- ☐ First aid kit
- ☐ Camera
- ☐ Syringe with filter
- ☐ Chain-of-custody forms and seals
- ☐ Cooler, packing material and ice packs (for maintaining samples at ≤ 10°C in transit [but not frozen])
- ☐ Quality Assurance Program Plan (QAPP)

☐ Sampling and Analysis Plan (SAP)

☐ Health and Safety Plan (HASP)

Planning and Preparation:

☐ Review site-specific information, such as regional land use patterns; site layout and topography; general information on the watershed; water bodies present; water depth; distribution, thickness, and type of sediment; nature of the shoreline; potential onshore and offshore sources of contaminants; ecological habitats and VECs; use of the study area for fishing, shellfish harvest, and boating or recreation; and current and anticipated future use of the water.

☐ Conduct site reconnaissance visit to aid in the preparation of the QAPP, SAP, and HASP

☐ Prepare QAPP

- Define and document the data quality objectives, including specific types of Quality Assurance/Quality Control (QA/QC) samples required to meet those data quality objectives.

- Determine and plan the field quality control samples that will be obtained for the project, including field duplicate samples, field blanks, equipment blanks, and trip blanks (see Chapter 3 of Volume 1 of this guidance manual for further details). Where feasible, test quality control samples during the site reconnaissance visit, so that adjustments can be made, as warranted.

- Duplicate samples generally should be collected at a rate of 10%, in order to assess sample location variability.

IMPORTANT: Use trip and field blanks, especially if sampling sites are subject to activity such as boat traffic or other use, or if the sampling sites have a high potential for transport of wind-borne COPCs. Use matrix spikes if sampling high ionic strength waters or waters high in suspended solids.

☐ Prepare SAP

- Review the study area features and devise a sampling location map that provides representative coverage.

- Identify COPCs and expected depth of occurrence. Select sampling and analytical methods and equipment based on study area-specific conditions and data quality objectives.

- Based on project objectives, determine sample volume requirements. Verify that the selected sampling method can produce the required volumes. If not, re-evaluate options for sampling methods.

- Correspond with the analytical laboratory(ies) concerning sampling requirements, holding times, preservation methods for individual COPCs, and shipping. Select sampling, handling, and shipping practices that will achieve those holding times, particularly for COPCs with short holding times.

IMPORTANT: Consult with the analytical laboratory concerning requirements related to sample volume, holding times, and preservation. Develop an SAP that specifies practices that will prevent exceeding established holding times. The SAP should specify holding times for individual COPCs, especially for COPCs with short holding times that may dictate some aspects of field logistics. Coordinate with laboratory regarding shipping methods and processing.

- List equipment, supplies and procedures related to sample labelling, preservation, handling, shipment, and equipment decontamination.

☐ Prepare HASP (including an emergency plan)

- Determine and plan safety requirements specifically associated with sampling requirements based on study area features, COPCs, and personal health and safety precautions (e.g., personal floatation devices and other boat safety requirements, if applicable[22], chemical resistant clothing and gloves, respiratory protection, ear and eye protection , *etc*.).

- Check proper functioning and integrity of PPE, including respirators and filters.

- Determine and plan safety requirements specifically associated with boat activities.

- Hard hats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet.

[22] DFO and other organizations may have their own safe work practices and requirements for working near or in water. Project-specific safety requirements should be researched and documented in the HASP.

- File a field plan and boat plan (if appropriate) with a land-based supervisor.

- Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should carry an adequate supply of water or other liquids for protection against dehydration in hot weather.

☐ Reserve, order, and pack all required equipment

- Obtain and prepare the necessary sampling and storage equipment, sample containers, materials, and documents.

- For efficiency and to reduce field decontamination activities, all sampling equipment should be cleaned and decontaminated at the laboratory or field office before going to the sampling site. If possible, a separate set of cleaned and decontaminated equipment should be available for each sampling site.

- Reserve a boat and make arrangements for qualified boat operator.

- Prepare a field notebook for documenting all activities related to field sampling. Information to be recorded in the field notebook includes, but is not limited to, weather conditions, progress toward accomplishing sampling objectives, deviations from the field sampling plan, sample identification numbers consistent with what is recorded on the laboratory chain-of-custody forms, sample observations, preservatives used and shipping protocol.

☐ If access to private property will be required, obtain written access permission from landowners

☐ Obtain First Aid/CPR certification and appropriate safety training for all field personnel

Sample Collection:

Proper safety precautions must be observed when collecting subsurface water samples.

IMPORTANT: **Never compromise your personal safety or that of a field partner to collect a sample.** Always plan ahead to avoid falling and drowning hazards. Always wear appropriate safety gear such as personal floatation devices. When working with winches, cables, and similar machinery, gloves, hard hats, safety glasses, and steel-toed boots are important safety items. A qualified boat operator is required for all transport or sampling from a boat. Boat operations must conform to all requirements in federal and provincial laws.

Depending on the depth and size of a water body, a boat may be required to access sample points. If the sampling trip involves the use of a boat, the weather forecast and/or marine conditions should be obtained prior to departure. If conditions are poor, then the sampling trip should be postponed.

1) All field staff must be familiar with the project HASP.

2) A clean pair of new, non-powdered, nitrile-type gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling.

3) If sampling a stream from a boat, as practical, sample from downstream to upstream sample locations to minimize the potential for sample contamination due to sediment re-suspension. Similarly in marine or estuarine environment, sample from down-current to up-current. If sampling from a bridge, collect samples on the upstream side of the bridge to minimize the influence of any trace constituents (e.g., metals) associated with the bridge.

4) Take precautions to minimize disturbance of the surface water (e.g., propeller wash) prior to sampling.

5) If sampling is supported by assessment of *in situ* parameters (pH, dissolved oxygen, *etc.*), assess such parameters following the procedures outlined in the SOP #7 for *in situ* water quality assessment. Conduct *in situ* assessments following collection of discrete subsurface water samples to minimize in-stream activities that may compromise sample integrity. Collect subsurface water samples before collecting sediment samples.

6) After positioning and anchoring the boat at the sample location, allow sufficient time for any re-suspended sediments to settle or travel away from the sample location. Measure water depth to determine the appropriate depth(s) of sample collection. The deployment ropes associated with many mechanical sampling devices are calibrated in metre or half-metre depths to facilitate sample collection at proper depths. Collect all samples from the upstream side of the boat and upwind of the motor to minimize sample contamination from metals and/or petroleum hydrocarbons associated with boat operation and gas fumes. Allow sufficient time for fumes or oils associated with boat operation to clear from the area.

7) Using a pre-cleaned van Dorn bottle or similar device (e.g., Kemmerer sampler) made of contact materials appropriate for the COPCs, set the sampling device to the open sampling position, following manufacturer's instructions. Use gloved hands whenever opening and setting the trigger devices on such equipment, using caution not to transfer contaminants, such as sediment or grease. Van Dorn bottles are often constructed of PVC and rubber materials. Such materials preclude sampling for highly sorptive compounds, VOCs, and many other organic compounds that may be associated with rubber and plastic materials. In such cases, alternative techniques are applied, or appropriate materials of contact are obtained (e.g., Teflon®, stainless steel, *etc.* as appropriate).

8) If this sampling methodology is to be used to collect samples for VOC analysis (using Teflon® or stainless steel equipment), samples should be collected with as little agitation or disturbance as possible. The VOA vial should be filled to the top and without bubbles or headspace after it is capped. If a bubble or bubbles are present, the vial should be refilled. The VOA vial may be either preserved with concentrated hydrochloric acid or they may be unpreserved. If the subsurface water sample contains a high concentration of dissolved calcium carbonate, there may be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles. This will render the sample unacceptable. In this case, unpreserved vials should be used and arrangements must be confirmed with the laboratory

9) Filtering is required for samples designated for dissolved metals analysis, but not those designated for total metals analysis. For those samples designated for dissolved metals analysis only, prior to sample shipment and as soon as possible after collection, filter samples using a peristaltic pump or hand pump and 0.45-micron mesh filter. Ideally, samples are filtered in the field at the collection site (on the vessel or shore side) immediately after sample collection. Place the resulting filtrate into a pre-preserved sample container, labelled for dissolved metals analysis.

10) Deploy the sampling device from the upstream side of the boat; rinse the sampling device at least two times with site water before sampling near the surface. When sampling in deep water and once the device is deployed to the proper sampling depth, allow it to maintain position for 30 seconds to allow flushing *in lieu* of rinsing before sample collection. Deploy the sample messenger to trigger the sampling device, and slowly retrieve the sampler.

11) Proceed with filling other sample containers, taking caution not to over fill any containers with sample preservatives. As samples approach the tops of sample containers, especially small volume containers, the lid of the respective sample container can be used to hold small sample volumes to transfer the final small sample volumes into sample containers.

12) Immediately place samples on ice.

13) Measure the water depth and conduct *in situ* analyses, if applicable.

14) Document sample location using a GPS unit, or using manual measurements to a known location (e.g., indicate sample location on a topographic map).

15) Prior to shipment and as soon as possible after collection, filter samples designated for dissolved metals analysis using a pump (e.g., peristaltic) or syringe and a 0.45-micron mesh filter. Filtration at the collection site immediately after sample collection is much preferred and required in some jurisdictions. Place the resulting filtrate into a pre-preserved sample container labelled for dissolved metals analysis.

16) Label each sample with the following information: date, time of sampling, sample ID, analytical parameters (e.g., metals), sampler initials, and method of preservation.

17) Follow proper procedures related to holding conditions (i.e., storage temperature > 0 to 6°C), chain-of-custody, and shipping.

18) Following all sampling events, clean and decontaminate mechanical sampling devices following manufacturer's recommendations and specific recommendations for the COPCs, as described in the sampling and analysis plan. In general, a mild surfactant detergent is sufficient. If organic metals or other inorganic COPCs are present, a 10 percent nitric acid wash solution is necessary for proper decontamination. If the COPCs are organic compounds, an acetone wash with water rinse is needed. Note: Do not clean plastic or polyvinyl chloride sampling devices with acetone or other organic solvents that will compromise the structural integrity of the sampling device.

19) All information regarding the sampling event should be accurately recorded in field notes, in order to support the subsequent preparation of a sampling event report.

REFERENCES

Canadian Council of Ministers of the Environment. 1993. *Guidance Manual on Sampling, Analysis, and Data Management for Contaminated Sites Volume I: Main Report.* PN 1101. CCME National Contaminated Sites Program. December.

Ontario Ministry of the Environment and Energy. 1996. *Guidance on Sampling and Analytical Methods for use at Contaminated Sites in Ontario*. Standards Development Branch. Toronto, Ontario. December.

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U.S. Environmental Protection Agency. 2013. *Operating Procedure for Surface Water Sampling*. Region 4, U.S. Environmental Protection Agency, Science and Ecosystem Support Division, Athens, Georgia. SESDPROC-201-R3. February 28, 2013. http://www.epa.gov/region4/sesd/fbqstp/

SUGGESTED OPERATING PROCEDURE NUMBER 10: COLLECTION OF SURFACE AND SUBSURFACE SEDIMENT DISCRETE SAMPLES

SCOPE This suggested operating procedure (SOP) recommends methods for collecting discrete sediment samples in surface sediment (i.e., generally 0 to 10 centimetres [cm]) and subsurface (i.e., generally greater than 10 cm) within wadeable (i.e., shallow water depths which allow safe access) and deep water, to ensure quality control in field operations and uniformity among field teams. Additional information on sediment sampling is provided in Chapter 10 of Volume 1 of the *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as *"Guidance Manual"*). For more information refer to Section 7 of *Protocols Manual for Water Quality Sampling in Canada* (http://www.ccme.ca/files/Resources/water/protocols_document_e_final_101.pdf).

APPLICATION This SOP describes the methods for collecting discrete sediment samples for the purposes of physical and chemical characterization and/or toxicological evaluation with aquatic organisms in support of human health and ecological risk assessments. Methods developed and used for sampling in large bodies of freshwater are in most cases applicable for sampling ocean/marine waters, but it is important to consider timing and access due to tidal fluctuations.

WHEN? Discrete sediment samples are typically collected for three purposes:

1) To determine the nature and concentration of contaminant of potential concern (COPC) exposures for human health and ecological risk assessments *via*;
    - Determination of the concentration of the COPC; and
    - Physiochemical characterization.
2) To characterize the relationship between COPC in sediment and potential adverse effects in aquatic organisms for ecological risk assessments *via*:
    - Toxicological evaluations; and
    - Benthic community evaluations; and
    - Collection of benthos for evaluation of COPC concentrations in tissue.
3) To understand patterns of chemical fate and transport that may affect exposure pathways in human health and ecological risk assessment.

WHY? Sediment sampling can be conducted to assess potential risks, delineate extent of contamination, and evaluate progress toward remediation requirements. Discrete sediment samplers are easy to operate, readily available, cost effective, and versatile.

HOW? Discrete sediment samples can be collected using a variety of methods ranging from basic manual hand devices (e.g., manual hand scoops, push tubes, grab samplers and corers) to mechanical jaw devices. The choice of appropriate sampling equipment depends on: study objectives (i.e., human health or ecological risk assessment); information from site reconnaissance (i.e., location of depositional areas); COPCs (i.e., equipment construction material compatibility); size of the study area or subarea; analytical testing requirements (e.g., sample volume required); biological analyses requirements (i.e., bioassays and benthic community studies); sediment characteristics (e.g., particle size); statistical considerations (i.e., minimum sample size needed); accessibility (i.e., boat or safe wading access); and budget.

TYPES

Surface and Subsurface Sediment – shallow water depth:

Scoop: A scoop constructed of non-reactive (inert) materials can be used to collect surface sediment samples. Scoops are generally used to sample sediment from wadeable ponds, lakes, wetlands, low current streams, estuaries, and tide pools. Scoops are inexpensive and easy to handle. A limitation of using scoops is the probable loss of fine grained material on retrieval of the sampler; therefore their use is recommended only for sampling sediments that are exposed to air or in shallow areas with minimal flow, where loss of fine-grained sediment can be minimized.

Push Tube: Push tubes are generally used to sample surface sediment in wadeable ponds, lakes, wetlands, low current streams, estuaries, and tide pools. Push tubes also can be used by divers in nonwadeable freshwater and marine waterbodies. Push tubes can be made of Teflon®, plastic, or glass and are available in many diameters (United States Environmental Protection Agency [USEPA], 2001). They are useful in soft, uniform sediment from which a relatively undisturbed sediment sample is desired (e.g., for volatile organic compound [VOC] analyses). Sample volume is determined by the diameter of the push tube and the depth to which the tube can be manually inserted into the sediment. One limitation of using push tubes is the difficulty of retaining sediment during extraction. Sediment retention can be improved by one or a combination of the following techniques: 1) core catchers (a finger-like trap that allows advancement of the push tube into sediment, but hinders fallout) can be used; 2) immediately prior to extraction, an end cap can be carefully placed on the bottom of the push tube (this is easier in softer sediment and shallow samples, as it requires the sampler to be able to physically reach the bottom of the tube); and 3) prior to extraction, the exposed end of the push tube can be filled with water and an end cap applied to create a vacuum to decrease the potential for fallout.

Mechanical: The Ekman sampler is a common type of mechanical sampling device. Ekman samplers consist of a small set of jaws or a bucket which, when the sampler is lowered and reaches the sediment surface on the bottom of the

water body, shut to contain a section of the sediment surface (USEPA, 2001). This type of mechanical device is usually attached to a pole or handle, enabling shallow sample collection (a rope with messenger is used for deeper waters). See Chapter 10 of Volume 1 of the *Guidance Manual* for additional information on Ekman samplers. Other light samplers that can be used are either the petite Ponar or the mini-shipek – which function on a similar principle as the Ekman sampler. In addition

Surface and Subsurface Sediment – greater water depth:

Mechanical: The most common mechanical device used to sample sediment from deep water is the Ponar sampler, which is available in petite and standard size and can sample up to a 0.05-square metre (m^2) area (USEPA, 2014). During descent, it creates less turbulence than other mechanical samplers, because a screen over the sample compartment permits water to pass through the sampler. The Ponar sampler is typically deployed from a boat using a winch. The petite Ponar sampler is smaller and lighter than the Ponar sampler and can be easily operated by one person in most conditions. It collects smaller samples (0.023 m^2) and is ideal for hazardous chemical sampling, because it is easier to decontaminate in the field (USEPA, 2014). The Shipek sampler is used, as frequently as the Ponar, in the Great Lakes. Other types of mechanical sediment samplers include the Ekman, van Veen, and Peterson samplers. The latter three types of samplers are commonly used in marine environment. Additional information on mechanical sediment samplers, including the Ponar and petite Ponar, is provided in Chapter 10 of Volume 1 of the *Guidance Manual*.

Additional information on sediment sampling methods is available in USEPA (1995: 2001; 2014), Clark (2003), Ontario Ministry of Environment and Energy ([MOEE] 1996), U.S. Navy (1997), and the sediment sampling section of Florida Department of Environmental Protection (2009).

SAMPLING CHECKLIST
Equipment Checklist:

- ☐ Stainless steel or disposable plastic scoop, push tube, or mechanical discrete sampling device, such as an Ekman or Ponar sampler
- ☐ Stainless steel bowls/pans (i.e. a material that is compatible with the COCP to avoid sample contamination) or disposable aluminum pans and sediment mixing utensils (e.g., stainless steel or Teflon spoon or spatula). Glass or other inert materials may need to be considered – see text box below.
- ☐ Certified pre-cleaned sample containers with labels
- ☐ Cooler, packing material and ice packs (for maintaining samples at ≤ 10°C in transit [but not frozen])

- ☐ Site and associated maps
- ☐ Sediment sampling logs
- ☐ Waterproof pens/markers
- ☐ Non-powdered nitrile-type gloves
- ☐ Tape measure
- ☐ Multi-parameter water quality meter (if required)
- ☐ Global Positioning System (GPS) unit
- ☐ Bathymetric map (detailed description of submerged terrain) or nautical charts (primarily for safe navigation) if applicable
- ☐ Stakes and flagging
- ☐ Field notebook
- ☐ Camera
- ☐ Hip or chest waders
- ☐ Personal protection equipment (PPE)
- ☐ Boat or sampling platform (for deep water sampling)
- ☐ Winch or sampler retrieval device (for deep water sampling or use with heavy sampling devices such as Ponar, Ekman, Shipek, van Veen)
- ☐ Personal floatation devices for all field personnel
- ☐ First aid kit
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater

Sampling Equipment and Sample Container Considerations

Consideration and care should be given to the materials that may contact the sediment sample during collection (i.e., sampling equipment, containers), in order to minimize the potential for cross-contamination or alteration of sample integrity. For example, plastic sample containers or metal sampling equipment can be sources of trace organic compounds and metals, respectively. Generally, relatively non-reactive (inert) materials—glass, stainless steel, and Teflon®—should be used for sample collection to obtain samples that represent the aquatic environment. Laboratories typically supply appropriate sampling containers that are certified by the laboratory as pre-cleaned. Proper decontamination and cleaning of sampling equipment between uses is essential to prevent cross-contamination.

- ☐ Quality Assurance Program Plan (QAPP)
- ☐ Sampling and Analysis Plan (SAP)
- ☐ Health and Safety Plan (HASP)

Planning and Preparation:

- ☐ Review site-specific information, such as regional land use patterns; site layout and topography; general information on the watershed; water bodies present; water depth; distribution, thickness, and type of sediment; nature of the shoreline; potential onshore and offshore sources of chemicals; ecological habitats and valued ecosystem components (VECs); use of the study area for fishing, shellfish harvest, and boating or recreation; and current and anticipated future use of the water.
- ☐ Conduct site reconnaissance visit to aid in the preparation of the QAPP, SAP, and HASP

IMPORTANT: Prior to initiation of sampling, consider field logistics – identify activities that must be done on the boat or immediately in the field, as opposed to those that could be performed later, on land or at a laboratory or in the office. It is often safest and most efficient to preserve samples in the field and to sort samples in a field trailer or in the laboratory. Check that all field personnel have the proper training and experience to carry out the sampling.

- ☐ Prepare QAPP
  - Define and document the data quality objectives, including specific types of Quality Assurance/Quality Control (QA/QC) samples required to meet those data quality objectives.

- Determine and plan the field quality control samples that will be obtained for the project, including field duplicate samples, field blanks, equipment blanks, and trip blanks.

- Duplicate samples generally should be collected at a rate of 10%, in order to assess sample location variability.

☐ Prepare SAP

- To the extent possible, clearly define the study area boundaries to constrain the sampling area and select geo-referenced sampling locations within the study area. Then, select appropriately matched reference areas with similar habitat conditions. In lotic (flowing) systems, a suitable reference area is often located immediately upstream of the study area, beyond the influence of the site. In lentic (static) systems, a suitable water body(ies) within the same watershed, but outside the area of influence, should be selected. The selection process and purpose of each reference site should be clearly stated.

- Identify COPCs and potential depth(s) of occurrence in sediment. Select sampling and analytical methods and equipment based on site-specific conditions and data quality objectives.

- Determine the minimum volume per sample or composite sample required for chemical analysis (sufficient volume must be collected to perform analysis on a dry weight basis). Determine the depth of sediment to be collected (e.g. top 3 cm *vs*. top 5 cm).

- Define the sediment sampling strategy. Factors to define within the SAP include: tidal conditions and sediment accessibility, targeted geo-referenced sampling locations, number of samples (i.e., collection of single samples per location or replicate samples), sampling effort (discrete samples *vs*. composite samples i.e., are samples composed of one Ponar or multiple Ponar "drops" which are then homogenized) and required sample volume per location. Additional information on these aspects of sediment sampling is provided in Chapter 10 of Volume 1 of the *Guidance Manual*.

- Review the study area features and habitat types and generate a sampling location map that provides representative coverage of the study area or meets specific project objectives (e.g., hot spot sampling).

- Coordinate with the analytical laboratory regarding methods for sample collection, processing, and shipping.

- Consider equipment decontamination procedures. As feasible, use disposable sampling devices to eliminate potential cross-contamination between sample locations. For those instruments that will be reused, the

SAP should specify proper decontamination methods to be used in the fields. Proper decontamination methods vary across COPCs and should be confirmed with the analytical laboratory in advance of sampling. All decontamination materials (e.g., washwater) must be collected and disposed of appropriately. In most cases, it is sufficient to wash samplers and gear with water from the study area.

- List equipment, supplies and procedures related to sample labelling, preservation, decontamination, handling, and shipping.

☐ Prepare HASP (including an emergency plan)

- Determine and plan safety requirements specifically associated with sampling requirements based on study area features, COPCs, and personal health and safety precautions (e.g., personal floatation devices and other boat safety requirements, if applicable, chemical resistant clothing and gloves, respiratory protection, ear and eye protection , *etc.*).

- HASP requirements, including requirements for PPE (e.g., chemical resistant clothing and gloves, respiratory protection, hard hats, ear and eye protection, *etc.*), are based on the site's features and the COPCs. All personnel should be aware of the potential dangers associated with sediment sampling. These dangers include, but are not limited to, strong water currents, slippery substrate, roots or sharp objects that may cause a fall or other personal injury. Additional concerns include the use of heavy objects on a vessel, the presence of "triggers" on some sampling devices that may release unexpectedly (resulting in injury to operators), and overhead hazards from winches or other equipment on the vessel. If sampling is conducted in water greater than three feet deep, sampling personnel are required to wear personal floatation devices.[23]

- Check proper functioning and integrity of PPE, including respirators and filters.

- Understand the physical demands required to properly operate field sampling equipment (e.g., winch operation) and plan accordingly.

- Hard hats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet.

- File a field plan and boat plan (if appropriate) with a land-based supervisor.

[23] DFO and other organizations may have their own safe work practices and requirements for working near or in water. Project-specific safety requirements should be researched and documented in the HASP.

- Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should carry an adequate supply of water or other liquids for protection against dehydration in hot weather.

☐ Reserve, order, and pack all required equipment

- Obtain and prepare the necessary sampling and storage equipment, sample containers, materials, and documents.

- For efficiency and to reduce field decontamination activities, all sampling equipment should be cleaned and decontaminated at the laboratory or field office before going to the sampling site. If possible, a separate set of cleaned and decontaminated equipment should be available for each sampling site.

- If necessary, reserve a boat and make arrangements for qualified boat operator.

- Prepare a field notebook for documenting all activities related to field sampling. Information to be recorded in the field notebook includes, but is not limited to, weather conditions, progress toward accomplishing sampling objectives, deviations from the field sampling plan, sample identification numbers consistent with what is recorded on the laboratory chain-of-custody forms, sample observations, preservatives used and shipping protocol.

☐ If access to private property will be required, obtain written access permission from landowners

☐ Obtain First Aid/CPR certification and appropriate safety training for all field personnel

☐ Weather forecasts and/or marine conditions reports should be obtained prior to departure for field sampling and sampling should be postponed if conditions are poor

Winter Sampling Safety

Sampling in winter presents extra elements of danger. Always proceed with caution over ice. Do not take unnecessary risks.

Check the ice for thickness with a rod or ice chisel every few steps (ice should be a minimum of 8 cm thick). Use the buddy system, and carry a length of rope (with a harness tied around your waist) to use as a life line. Avoid honeycombed ice and areas over rapids. Be aware that ice downstream from bridge supports and/or outlets may be thin as a result of modified flow patterns and de-icing agents.

IMPORTANT: **Never compromise personal safety or the safety of a field partner**. Always plan ahead to avoid hazards. Always wear appropriate PPE, including personal floatation devices. When working with winches, cables, and similar machinery, PPE should include gloves, hard hats, safety glasses, and steel-toed boots. Boat operations must conform to all requirements in federal and provincial laws, and a qualified boat operator is required for all sampling conducted from a vessel.

Sample Collection:

1) Select sediment sample location, mark location (e.g., stake or post) if feasible, and conduct georeferencing (i.e., GPS) and/or manual measurements to known physical locations for mapping and site modelling purposes. Log water depth at each station.

2) If surface water samples are also to be collected, collect surface water samples at each location before collecting sediment samples, taking care not to disturb the sediment layer.

3) If required, prior to sediment collection at each location, carry out water quality measurements using a multi-parameter meter, close to the sediment layer (e.g., approximately 1 metre above the sediment layer), taking care not to disturb the sediment layer.

4) To minimize the potential for cross-contamination from suspended sediment, begin sampling at the farthest downstream (or down-current) location and proceed successively upstream (or up-current). Stand facing the direction of flow and approach the location from the downstream direction. In static systems, areas where contaminant concentrations are expected to be greatest should be sampled last. Sampling should proceed from sites where lowest concentrations of contamination is expected to stations where greatest concentrations of contamination is expected to reduce the potential for sample cross-contamination, particularly if any component of the sampling device(s) must be reused.

5) Take precautions to avoid disturbing the sediment (e.g., propeller wash) prior to sampling.

6) Wear appropriate PPE for all areas of potential exposure (e.g., gloves, waders, and eye protection). All information regarding the sampling event should be accurately recorded in field notes and appropriate photographs should be taken in order to support the subsequent preparation of a sampling event report.

Sampler Use/Deployment for Sediment Sample Collection – shallow water depth:

Collect a discrete surface (i.e., generally 0 to 10 cm) sediment sample using a scoop (or equivalent), push tube, or mechanical sampler, as follows:

1) For scoops, collect a surface sediment sample by scooping a portion of the surface sediment, minimizing the loss of fine-grained sediment as much as possible.

2) For push tubes, gently push the tube into the sediment to the desired depth. Methods for retaining sediment are described earlier in this SOP. Carefully extract the push tube from the sediment.

3) For mechanical samplers, lower the sampler to the sediment surface and either automatically (e.g., messenger) or manually trigger the sampler jaws to close. Mechanical samplers can also be used to collect subsurface samples (i.e., sample depth greater than 10 cm); consult Appendix 10-1 of Volume 1 to see the depths and volume of sediment samples taken by various sediment sampling equipment.

Sampler Use/Deployment for Sediment Sample Collection – greater water depth:

1) Prepare the sampler for deployment; including checking for proper decontamination and operation of the sampling device (consult Appendix 10-1 of Volume 1 for selecting equipment for collection of surface or subsurface sediment samples).

2) Lower the sampler to the sediment surface, ensuring that it settles flat. Unless a camera or diver can verify that the sampler has settled flat, making sure that the cable is vertical, as much as possible, improves the outcome that the sampler has settled “flat”.

3) Activate the device to completely close the sampler and retrieve it from the water. Verify full closure of the jaws or sample device when the sampler reaches the surface (keep the sampler as horizontal as possible). Obstructions (e.g., rocks and/or foreign material) may hinder proper closure of the sample device. If closure verification cannot be established prior to surfacing the device, the acceptability of the sample must be determined when the sampling

device is inspected upon retrieval (verify that trap is fully closed, the presence of fined grained sediments on the surface is a good indication that washout has not occurred). If unacceptable, set aside the content for later disposal, and re-deploy the sampling device. It is important not to discard the sample back in to the water until all sampling is finished, to minimise contaminating subsequent samples. If acceptable, pour, decant, or siphon excess water out of the sampling device. Effort should be made to minimize the loss of fine-grained material from the surface of the sediment.

4) If composite samples are being collected from a site the boat operator must make every effort to return to the same sampling location: within a radius of 5 m of the original targeted georeferenced location.

Sample Collection (continued):

7) If sampling for VOCs or other analytes or parameters sensitive to changes in reduction-oxidation (redox) conditions or excessive handling (e.g., acid volatile sulphide /simultaneously extracted metals [AVS/SEM] and toxicity testing), place the sediment sample directly from the scoop or sampler device into the sample container, minimizing disturbance as much as possible.

8) If sampling for VOCs, sample may be preserved back at the lab if appropriate sample container and strict holding times are met, or can be preserved in the field with methanol or sodium bisulphate preservative (extrude ca. 5g of sediment into sample vial containing preservative). Alternatively, hermetically sealed samples may be collected if appropriate sampling devices are used. Please consult with your analytical laboratory for assistance prior to collecting these samples. The overall goal is to minimize loss of VOCs due to volatilization.

9) For composite sampling only;

   i. Remove the sample (or portion of sample from a designated depth interval) from the sample device and place into an inert bowl or pan.

   ii. If necessary, collect additional discrete sediment samples to obtain additional sample volume from the location (approximately five or six, if possible) and empty each discrete sample into the bowl or pan. Composite samples cannot be used for VOC analysis

10) As much as practicable, remove all visible non-representative sediment material, (e.g., twigs, shells, leaves, stones, wood chips, and vegetation). In general, a particle size of 2 millimetres (mm) or less serves as the basis to discriminate between sediment and non-sediment material (USEPA, 2001). More importantly, the goal is to collect representative sediment (i.e., particle size) for the study area. Record, in field notebook, all removed non-representative material.

11) Photograph the sediment sample (and any discarded materials e.g., rocks, wood chips,) and record the photo number in the sediment sampling log.

12) For composite sampling only (if mixing the sample in the field), gently mix the discrete samples using a scoop or spoon until the sample appears homogeneous; avoid excessive mixing to maximize sample integrity.

IMPORTANT: Certain chemicals and analyses are sensitive to the active mixing that occurs in order to homogenize a composite sample. For example, VOCs volatilize if the sediment is handled excessively. Analytes that are sensitive to redox conditions, like AVS and SEM, should not be subsampled from a composite sample. These sensitive parameters should be collected as discrete samples. Place the sediment sample directly from the scoop or sampler device into the sample container, minimizing disturbance as much as possible. Do not homogenize. For those COPCs that are not typically sensitive to mixing, it is good standard practice to only mix the sample until homogenized—avoid over-mixing the sample.

13) Place homogenized sediment into the appropriate sample containers according to the protocols for the COPCs. In general, fill sample containers in the following sequence unless the SAP specifies priority COPCs (e.g., organic carbon is an essential part of the study design/objectives):

    a. VOC
    b. Semi- and non-volatile compounds (e.g., polychlorinated biphenyls [PCBs])
    c. Inorganics (i.e., metals)
    d. Miscellaneous parameters (e.g., particle grain size and organic carbon)

14) Label each container with: the date, time of sampling, sample identification, the COPCs, the initials of the sampling personnel, and the method of preservation (if relevant).

15) Record sample and sample location characteristics including, but not limited to, lithology (colour, texture, odour), anoxic content, presence of sheen and/or debris, date, time, and name of person logging sample; sample location number and coordinates; project designation; relative depth of water and surface elevation; estimated sediment penetration; sediment sample depth interval; percent sample recovery; and other comments (problems with the sampling and/or why areas were excluded from sampling).

16) If elevated chemical concentrations are expected in some samples, those samples should be stored separately from samples expected to contain trace chemical concentrations. Reference samples should be stored in designated, separate coolers.

17) Follow proper procedures for sample preservation, holding conditions (i.e., storage temperature > 0 to 6°C), chain-of-custody, and shipping. Reference applicable sampling designs to verify any unique procedures for storage or transportation (e.g., light sensitivity or vertical storage).

18) If relevant and useful, photograph each sample location and record the photograph number in the field notebook (this step may not be required for sample stations located on large lakes or in the open marine environment).

19) Clean and decontaminate all reusable sampling devices and support materials or equipment which may have come in contact with sample material between each sampling location. In general, a mild, surfactant detergent is sufficient. If organic metals or other inorganic COPCs are present, a 10 percent nitric acid wash solution is necessary for proper decontamination. If organic COPCs are of concern, an acetone wash with water rinse is needed. Note: Do not clean plastic or polyvinyl chloride sampling devices with acetone or other organic solvents that will compromise the structural integrity of the sampling device.

20) All information regarding the sampling event should be accurately recorded in field notes, in order to support the subsequent preparation of a sampling event report.

IMPORTANT: If an oily or tarry residue is present after washing with a surfactant detergent solution and deionized water, wash the equipment thoroughly with an organic solvent (methanol, acetone, hexane or other) using a separate brush to remove any particulate matter or surface film. Collect the rinseate for proper disposal.

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SUGGESTED OPERATING PROCEDURE NUMBER 11: COLLECTION OF SEDIMENT CORE SAMPLES

SCOPE This suggested operating procedure (SOP) recommends methods for collecting sediment core samples to ensure quality control in field operations and uniformity among field teams. Core samplers are recommended for sampling programs where it is critical to maintain the integrity of the sediment profile and to obtain information regarding vertical distribution of selected parameters. Additional information on sediment core sampling is provided in Chapter 10 of Volume 1 of the *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as *"Guidance Manual"*). For more information refer to Section 7 of *Protocols Manual for Water Quality Sampling in Canada* (http://www.ccme.ca/files/Resources/water/protocols_document_e_final_101.pdf).

APPLICATION This SOP describes methods for collecting sediment core samples for the purposes of sediment deposit profiling and physical and chemical analyses, in support of human health and ecological risk assessments. Methods developed and used for sampling in large bodies of freshwater are in most cases applicable for sampling ocean/marine waters, but it is important to consider timing and access due to tidal fluctuations.

WHEN? Sediment cores are generally collected for the following purposes in risk assessment:

1. To sample sediment for the vertical evaluation of sediment characteristics and distribution of contaminants of potential concern (COPCs);

2. To characterize historical concentrations of COPCs in the sediment bed; and

3. To understand sedimentation processes that may affect migration and exposure pathways in human health and ecological risk assessment.

WHY? Coring devices collect vertical columns of sediment. Compared to discrete samplers, coring devices create minimal water turbulence during descent (U.S. Environmental Protection Agency [USEPA], 2001). Thus, core samplers leave fines and chemicals at the sediment-water interface minimally disturbed. Processing cores while on the vessel or shore side is relatively simple, as sediment cores are collected in liners that are capped and can be delivered directly to the laboratory for analysis. Care should be taken when transporting sediment cores, so as not to disturb the sediment in the core. If this is not possible due to fine nature of the sediment, the cores will have to be processed in the field, immediately after collection. Sediment coring is useful for preserving the sequential layering of sediment to obtain a historical profile of sediment deposition. Therefore, sediment core sampling may be conducted to evaluate progress toward remediation requirements in the case of monitored natural recovery. Sediment coring data can also be used to evaluate potential concerns if

deeper sediment is disturbed and mixed with surface sediment. In addition, core samplers may be used where it is important to maintain an oxygen-free environment, as the closed cell design of the sampler effectively limits the exchange of oxygen (USEPA, 2001). The disadvantage of coring devices is that a limited sample volume is obtained, and many cores may be required to obtain the required volume of sediment for analysis.

HOW? In general, core samplers fall into three broad categories: gravity core samplers, piston core samplers, and vibracore samplers. Core samples may be collected using a variety of methods. The choice of the appropriate sampling method depends on: depth of the water; sediment characteristics (e.g., particle size); and length of the core to be collected.

TYPES **Gravity Core Sampler:** The sample is collected using the force of gravity to penetrate the sediment. In general, the gravity core penetrates deeper into the sediment when the device is heavier and the water depth is sufficient to obtain the necessary velocity. Balance must be reached between the weight of the corer, the speed of the decent of the corer and the type of sediment. In soft, fine-grained sediment, gravity core samplers can reach depths of up to 3 metres (m) (USEPA, 2001). The box core sampler is one of the most commonly used gravity core samplers. When used properly, the box core sampler can obtain undisturbed sediment samples from the sediment-water interface (USEPA, 2001). A variety of replaceable internal liner materials are available including stainless steel, glass, Teflon®, polyvinyl chloride (PVC), or carbon steel, depending on the type of analyses and COPCs. Selection of liners should be considered prior to sampling to prevent cross-contamination.

A gravity check valve used in conjunction with the Benthos type gravity core sampler allows air and water to pass through the tube during the descent. The check valve closes to create negative pressure on the back of the sample as it is extracted from the substrate. The vacuum pressure acts to securely hold the complete sediment core inside the tube, with minimal sample loss upon retrieval (CCME, 1993; USEPA, 2001). A piston can be used instead of a check valve, but this does not allow water flow through the sampler during descent. In addition, use of a core catcher at the end of the corer can also help retain the core sample inside the liner to avoid sample loss.

Piston Core Sampler: Piston core samplers are used in relatively soft, fine-grained sediment to collect sediment cores up to 30 m deep (CCME, 1993). Like gravity core samplers, piston core samplers are lowered to the sediment surface using gravity. However, the piston, located inside the core barrel, stops at the sediment-water interface to avoid sediment disturbance. As the core barrel continues to penetrate the sediment, the piston creates a vacuum, reducing the resistance to the core barrel's penetration into the sediment and filling the void space of the core barrel (CCME, 1993). This action reduces the likelihood of sample disturbance or compression and allows the sampler to reach relatively

deeper sediment depths. These type of samplers are typically deployed by large boats or platforms due to the size, weight, and retrieval mechanism requirements.

Vibracore Sampler: This device minimizes compaction or spreading when sampling soft or loosely consolidated sediments. An electric motor creates a vibration range that enables the core tube to displace the sediment and advance with minimal sediment compaction. A vibracore sampler can collect cores up to 10 m or more in length (USEPA, 2001). The weight of this sampler usually requires a large vessel for transport, deployment, and retrieval from the sediment.

Additional information on sediment sampling methods is available in USEPA (1995; 2001; 2014), Clark (2003), MOEE (1996), U.S. Navy (1997), and the sediment sampling section of Florida Department of Environmental Protection (2009).

Essential Information

Depending on the softness of the sediment, coring devices can produce sediment cores that have spread or compacted as a result of driving the device into the sediment. Spreading (a condition that occurs when the sediment is pushed to the side) and compaction (a condition that occurs when the sediment is pushed downward during core tube advancement) both affect the physical integrity of the core sample. Consult the USEPA's *Operating Procedure for Sediment Sampling* (USEPA, 2014) for more information about addressing spreading and compaction.

COLLECTION Core sampling can be ineffective if the coring device compresses the sample, resulting in insufficient sample volume. This limitation is greatly influenced by the diameter of the core tube and the composition of the sediment. Therefore, the diameter of the core device is determined by the type of substrate to be sampled. For example, if the sediment has a high clay content, then a larger diameter coring device would allow for the sample to be collected with minimal compaction. On the other hand, if the sediment is primarily sand or loose material, a smaller diameter coring device may be needed to prevent loss of sample. The tube length may vary but is typically at least 25 centimetres (cm) longer than the desired sediment core length.

IMPORTANT: Prior to initiating sampling, consult with the analytical laboratory regarding requirements for sample volume, holding times, light sensitivity, temperature (e.g., frozen *vs.* chilled), transportation considerations (e.g., horizontal integrity), and preservation. Develop a Sampling and Analysis Plan (SAP) that specifies the holding times for COPCs, as well as recommended practices for avoiding exceeding holding times. Sampling, processing, and shipping practices required for analytes with short holding times may dictate some aspects of field logistics.

SAMPLING CHECKLIST

Equipment Checklist:

- ☐ Core sampler, such as a gravity, piston, or vibracore sampler
- ☐ Siphoning tube
- ☐ Core liner, made of material appropriate for the COPC(s)
- ☐ Basket-type core catcher
- ☐ Core liner caps
- ☐ Core-cutting tool or knife
- ☐ Stainless steel or Teflon (i.e. a material that is compatible with the COCP to avoid sample contamination) disposable scoops or spoons
- ☐ Stainless steel or glass bowls/pans or disposable aluminum pans
- ☐ Certified pre-cleaned sample containers with labels
- ☐ Cooler, packing material and ice packs (for maintaining samples at $\leq 10°C$ in transit [but not frozen])
- ☐ Container for collection and transportation of core samples
- ☐ Site and associated maps
- ☐ Sediment sampling logs
- ☐ Waterproof pens/markers
- ☐ Non-powdered nitrile-type gloves
- ☐ Tape measure
- ☐ Multi-parameter water quality meter (if required)

- ☐ Global Positioning System (GPS) unit
- ☐ Bathymetric map (detailed description of submerged terrain) or nautical charts (primarily for safe navigation) if applicable
- ☐ Field notebook
- ☐ Camera
- ☐ Boat or sampling platform
- ☐ Winch or sampler retrieval device (for deep water sampling or use with heavy sampling devices such as Ponar, Ekman, Shipek, van Veen)
- ☐ Personal floatation devices for all personnel
- ☐ First aid kit
- ☐ Personal protection equipment (PPE)
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Quality Assurance Program Plan (QAPP)
- ☐ Sampling and Analysis Plan (SAP)
- ☐ Health and Safety Plan (HASP)

Sampling Equipment and Sample Container Considerations

Consideration and care should be given to the materials that may contact the sediment sample during collection (i.e., sampling equipment, containers), in order to minimize the potential for cross-contamination or alteration of sample integrity. For example, plastic sample containers or metal sampling equipment can be sources of trace organic compounds and metals, respectively. Generally, relatively non-reactive (inert) materials—glass, stainless steel, and Teflon®—should be used for sample collection produce high quality samples. Laboratories typically supply appropriate sampling containers that are certified by the laboratory as pre-cleaned. Proper decontamination and cleaning of sampling equipment between uses, in particular the core tubes (liner), is essential to prevent cross-contamination.

Planning and Preparation:

- ☐ Review site-specific information, such as regional land use patterns; site layout and topography; general information on the watershed; water bodies present; water depth; distribution, thickness, and type of sediment; nature of the shoreline; potential onshore and offshore sources of contaminant; ecological habitats and valued ecosystem components (VECs); use of the study area for fishing, shellfish harvest, and boating or recreation; and current and anticipated future use of the water.

- ☐ Conduct site reconnaissance visit to aid in the preparation of the QAPP, SAP, and HASP

IMPORTANT: Prior to initiation of sampling, consider field logistics – identify activities that must be done on the boat or immediately in the field, as opposed to those that could be performed later, on land or at a laboratory or in the office. It is often safest and most efficient to preserve samples in the field and to sort samples in a field trailer or in the laboratory.

- ☐ Prepare QAPP

  - Define and document the data quality objectives, including specific types of Quality Assurance/Quality Control (QA/QC) samples required to meet those data quality objectives.

  - Determine and plan the field quality control samples that will be obtained for the project, including field duplicate samples, field blanks, equipment blanks, and trip blanks.

  - Duplicate samples generally should be collected at a rate of 10%, in order to assess sample location variability.

- ☐ Prepare SAP

  - To the extent possible, clearly define the study area boundaries to constrain the sampling area and select geo-referenced sampling locations within the study area. Then, select appropriately matched reference areas with similar habitat conditions. In lotic (flowing) systems, a suitable reference area is often located immediately upgradient of the study area, beyond the influence of the site. In lentic (static) systems, a suitable water body(ies) within the same watershed, but outside the area of influence, should be selected. The selection process and purpose of each reference site should be clearly stated.

  - Identify COPCs and select sampling and analytical methods and equipment based on site-specific conditions and data quality objectives.

- Determine the minimum volume per sample or composite sample required for chemical analysis.

- Define the sediment sampling strategy. Factors to define within the SAP include: tidal conditions and sediment accessibility, sampling locations, number of samples (e.g., single samples or site replicates; discrete or composite samples), and required sample volume per location. Additional information on these aspects of sediment sampling are provided in Chapter 10 of Volume 1 of the *Guidance Manual*.

- Review the study area features and habitat types and devise a sampling location map that provides representative coverage.

- Coordinate with the analytical laboratory regarding methods for sample collection, processing, and shipping.

IMPORTANT: Prior to sampling, consult with the analytical laboratory concerning requirements related to sample volume, holding times, and preservation. Develop an SAP that specifies practices that will prevent exceeding established holding times. The SAP should specify holding times for individual COPCs, especially for COPCs with short holding times that may dictate some aspects of field logistics. Coordinate with laboratory regarding shipping methods and processing.

- Consider equipment decontamination procedures. As feasible, use disposable sampling devices to eliminate potential cross-contamination between sample locations. For those instruments that will be reused, the SAP should specify proper decontamination methods to be used in the fields. Proper decontamination methods vary across COPCs and should be confirmed with the analytical laboratory in advance of sampling. All decontamination materials (e.g., washwater) must be collected and disposed of appropriately.

- List equipment, supplies and procedures related to sample labelling, preservation, decontamination, handling, and shipping.

☐ Prepare HASP (including an emergency plan)

- Determine and plan safety requirements specifically associated with sampling requirements based on study area features, COPCs, and personal health and safety precautions (e.g., personal floatation devices and other boat safety requirements, if applicable, chemical resistant clothing and gloves, respiratory protection, ear and eye protection, *etc.*).

- HASP requirements, including requirements for PPE (e.g., chemical resistant clothing and gloves, respiratory protection, hard hats, ear and eye protection, *etc.*), are based on the site's features and the COPCs. All personnel should be aware of the potential dangers associated with

sediment sampling. These dangers include, but are not limited to, strong water currents, slippery substrate, roots or sharp objects that may cause a fall or other personal injury. Additional concerns include the use of heavy objects on a vessel, the presence of "triggers" on some sampling devices that may release unexpectedly (resulting in injury to operators), and overhead hazards from winches or other equipment on the vessel. If sampling is conducted in water greater than three feet deep, sampling personnel are required to wear personal floatation devices.[24]

- Check proper functioning and integrity of PPE, including respirators and filters.

- Hard hats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet.

- File a field plan and boat plan (if appropriate) with a land-based supervisor.

- Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should carry an adequate supply of water or other liquids for protection against dehydration in hot weather.

☐ Reserve, order, and pack all required equipment

- Obtain and prepare the necessary sampling and storage equipment, sample containers, materials, and documents.

- For efficiency and to reduce field decontamination activities, all sampling equipment (e.g., core tubes) should be cleaned and decontaminated at the laboratory or field office before going to the sampling site. If necessary, reserve a boat and make arrangements for qualified boat operator and qualified winch operators.

- Prepare a field notebook for documenting all activities related to field sampling. Information to be recorded in the field notebook includes, but is not limited to, weather conditions, progress toward accomplishing sampling objectives, deviations from the field sampling plan, sample identification numbers consistent with what is recorded on the laboratory chain-of-custody forms, sample observations (including core length and identification of a clay plug), and shipping protocol.

[24] DFO and other organizations may have their own safe work practices and requirements for working near or in water. Project-specific safety requirements should be researched and documented in the HASP.

- ☐ If access to private property will be required, obtain written access permission from landowners
- ☐ Obtain First Aid/CPR certification and appropriate safety training for all field personnel
- ☐ Weather forecasts and/or marine conditions reports should be obtained prior to departure for field sampling and sampling should be postponed if conditions are poor

Winter Sampling Safety

Sampling in winter presents extra elements of danger. Always proceed with caution over ice. Do not take unnecessary risks.

Check the ice for thickness with a rod or ice chisel every few steps (ice should be a minimum of 8 cm thick). Use the buddy system, and carry a length of rope (with a harness tied around your waist) to use as a life line. Avoid honeycombed ice and areas over rapids. Be aware that ice downstream from bridge supports and/or outlets may be thin as a result of modified flow patterns and de-icing agents.

Sample Collection:

1) Select sediment sample location and conduct georeferencing (i.e., GPS) and/or manual measurements to known physical locations for mapping and site modelling purposes. When conducting location logging for deep water sediment collection, care must be taken, especially in flowing conditions, to establish a vertical reference point.

2) If required, prior to sediment collection at each location, record water quality measurements at the surface, mid-depth, and within 1 m from bottom using a multi-parameter meter, taking care not to disturb the sediment layer (the equipment required to obtain this type of information in deep water will create its own challenges).

3) In flowing systems, to minimize the potential for disturbing the sediment, begin sampling at the farthest downstream (or down-current) location and proceed successively upstream (or up-current). Stand facing the direction of flow and approach the location from the downstream direction.

4) Take precautions to avoid disturbing the sediment (e.g., propeller wash) prior to sampling.

5) Wear appropriate PPE for all areas of potential exposure (e.g., gloves, waders, and eye protection).

6) Prepare the sediment coring device for deployment, including ensuring proper decontamination and assembling of the device.

IMPORTANT: If unfamiliar with the area of investigation, reconnaissance sampling may be appropriate prior to deployment of intrusive substrate core samplers. Grab samplers can be used to verify the nature of the substrate and help determine the proper sampling equipment and technique.

7) Deploy the sediment coring device according to the manufacturer's operating instructions.

8) Withdraw the coring device from the sediment according to the manufacturer's operating instructions for the specific device.

9) Immediately place a cap on the bottom of the core liner to prevent sample loss keeping the core in a near vertical position to preserve its integrity. Acceptability of the sample must be determined when the sampling device is inspected upon retrieval. If unacceptable, set aside the content for later disposal, and re-deploy the sampling device. It is important not to discard the sample back in to the water until all sampling is finished, to minimise contaminating subsequent samples. If acceptable, pour, decant, or siphon excess water out of the sampling device. Effort should be made to minimize the suction of fine-grained material or disturbing the integrity of the upper part of the core.

10) Using a hacksaw or core cutting device, remove the upper (excess) portion of the core liner that does not contain sediment. If using a hacksaw, ensure that no plastic cuttings fall into the core.

11) Place a cap at the top of the core liner.

12) Label each core liner with: date, time of sampling, sample identification number, COPCs, length of core sample, initials of the samplers, and method of preservation.

13) Document the sample location using a GPS unit and/or manual measurements to a known location.

14) Although it is preferred to process the cores in the field (see next step), if sending the intact sediment core directly to the analytical laboratory, ensure that both ends of the core liner are sealed tightly and the cores are stored and/or transported, if all possible, in an upright position. Document the state of the core using photographs and written description. Transportation of cores must be carried out very carefully, so as to avoid disturbing (mixing) the sediment in the core and therefore, having a sample that is not representative of the sampled bottom.

15) If processing sediment sample(s) in the field, extrude the sediment sample according to the manufacturer's operating instructions for the specific device and/or open the core liner using a spatula or core-cutting tool. A procedure should be in place to carry out this operation. Photograph sample core and record the photo number in the sample log book.

16) Extrude and remove the appropriate sediment sample (or portion of sample from a designated depth interval) using a spatula or spoon.

IMPORTANT: Certain chemicals and analyses are sensitive to the active mixing that occurs in order to homogenize a composite sample. For example, volatile organic compounds (VOCs) volatilize if the sediment is handled excessively. Analytes that are sensitive to reduction-oxidation (redox) conditions, like acid volatile sulphide (AVS) and simultaneously extracted metals (SEM), should not be subsampled from a composite sample. These sensitive parameters should be collected as discrete samples. Place the sediment sample directly from the scoop or sampler device into the sample container, minimizing disturbance as much as possible. Do not homogenize. For those COPCs that are not typically sensitive to mixing, it is good standard practice to only mix the sample until homogenized—avoid over-mixing the sample.

17) If sampling for VOCs or other analytes or parameters sensitive to changes in redox conditions or excessive handling (e.g., acid volatile sulphide/ simultaneously extracted metals [AVS/SEM] and toxicity testing), place the sediment sample directly from the sampling device into the sample container, minimizing disturbance as much as possible.

18) For composite sampling only, transfer the core sediment into an inert bowl or pan using a spoon or plunger type device. If feasible, avoid collection of smear zone (i.e., interior sides of the core liner) sediment. If necessary, collect additional sediment core samples to obtain necessary sample volume from the location and transfer the core(s) into the inert bowl or pan. If multiple cores are required to satisfy sample volume requirements then care must be taken to collect the cores from the same location (i.e., within 5 m of the station coordinates).

19) Remove all visible non-representative sediment material, (e.g., twigs, shells, leaves, stones, wood chips, and vegetation). In general, a particle size of 2 millimetres (mm) or less serves as the basis to discriminate between sediment and non-sediment material (USEPA, 2001). More importantly, the goal is to collect representative sediment for the study area, regardless of particle size. Record, in field notebook, all removed non-representative material.

20) Photograph the sediment sample and record the photo number in the sediment sampling log.

21) For composite sampling only, gently mix the individual core samples using a scoop or spoon until the sample appears homogeneous; avoid excessive mixing to

maximize sample integrity. If possible, do not mix the sediment in the field. Carry out all homogenization in a laboratory environment if confident that core tubes will remain intact from time of sampling. Compositing cannot be used for VOC analysis.

22) Place homogenized sediment into the appropriate sample containers according to the protocols for the COPCs.

23) Label each container with: the date, time of sampling, sample identification number, COPCs, sediment sample depth interval (e.g., 0-5 cm, 10-20 cm) initials of the sampling personnel, core samples used (for composite sampling only).

24) Record sample and sample location characteristics including, but not limited to, lithology (colour, texture, odour), anoxic content, presence of sheen and/or debris, date, time, and name of person logging sample; sample location number and coordinates; project designation; relative depth of water and surface elevation; estimated sediment penetration; sediment sample depth interval; percent sample recovery; and other comments (problems with the sampling and/or why areas were excluded from sampling).

25) Follow proper procedures related to preservation, holding (i.e., storage temperature > 0 to 6°C), chain-of-custody, and shipping. Reference applicable sampling designs to verify any unique procedures for storage or transportation (e.g., light sensitivity or vertical storage).

26) Photograph of each sample location and record the photograph number in the field notebook.

27) Clean and decontaminate all reusable sampling devices and support materials or equipment which may have come in contact with sample material between each sampling location. In general, a mild, surfactant detergent is sufficient. If organic metals or other inorganic COPCs are present, a 10 percent nitric acid wash solution is necessary for proper decontamination. If organic COPCs are of concern, an acetone wash with water rinse is needed. Note: Do not clean plastic or polyvinyl chloride sampling devices with acetone or other organic solvents that will compromise the structural integrity of the sampling device.

IMPORTANT: If an oily or tarry residue is present after washing with a surfactant detergent solution and deionized water, wash the equipment thoroughly with an organic solvent (methanol, acetone, hexane or other) using a separate brush to remove any particulate matter or surface film. Collect the rinseate for proper disposal.

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SUGGESTED OPERATING PROCEDURE NUMBER 12: COLLECTION OF PORE WATER SAMPLES

SCOPE This suggested operating procedure (SOP) recommends methods for collecting porewater samples to ensure quality control in field operations and uniformity among field teams. Additional information on sediment porewater sampling is provided in Chapter 10 of Volume 1 of the *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as "*Guidance Manual*").

APPLICATION This SOP describes the methods for collecting porewater samples for the purposes of chemical characterization and toxicity testing, in support of ecological risk assessments. Methods developed and used for sampling in large bodies of freshwater are in most cases applicable for sampling ocean/marine waters, but it is important to consider timing and access due to tidal fluctuations.

WHEN? Sediment porewater is typically collected for the following purposes:

1. To characterize potential exposure and risk to benthic organisms from aqueous phase contaminants of potential concern (COPCs);

2. To identify groundwater discharge areas; and

3. To understand patterns of COPC fate and transport that may affect exposure pathways.

WHY? Porewater sampling is typically conducted to assess potential ecological risks and/or to determine the geochemical form, or speciation, of COPCs in the aqueous phase. Because geochemical speciation affects bioavailability, which affects toxicity, exposure of benthic organisms to porewater is an important consideration in ecological risk assessment. Porewater toxicity tests can complement information gained from bulk sediment and surface water toxicity evaluations.

HOW? Porewater in fine-grained, uncompacted sediment is most suitable for sampling (U.S. Environmental Protection Agency [USEPA], 2001), although porewater can also be successfully extracted from coarse-grained, sandy sediment. Porewater can be collected using either *in situ* or *ex situ* procedures. Porewater sampling procedures should minimize changes to *in situ* sediment conditions at the sampling location, in order to avoid changing chemical bioavailability and toxicity in the sample. According to the USEPA's *Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual* (USEPA, 2001), **all sediment collection and processing methods alter porewater chemistry to some degree. The extent to which alterations to porewater chemistry are a significant concern depends on environmental conditions, the geochemistry of the COPCs, and the objectives of the sampling program.**

Although both *in situ* and *ex situ* sampling methods may be adequate for site-specific sampling program objectives, it is crucial that the same sampling procedures and equipment be used at all sampling locations within a field program. This consistency of procedures and equipment within a field program will improve the risk assessor's ability to compare the significance of the results across sampling locations. In addition, the sediment depth at which porewater is extracted, using either *in situ* or *ex situ* procedures, should correlate with the depth of interest for the sampling program.

TYPES

In situ: Compared to *ex situ* methods, *in situ* porewater sampling methods are less subject to sampling and extraction related artefacts (USEPA, 2001). Thus, *in situ* methods are more likely to maintain the chemical integrity of the sample. However, *in situ* methods generally produce relatively small volumes of porewater and are often limited to wadeable or diver-accessible water depths. These logistical constraints restrict their use and applicability in some sediment monitoring studies, particularly in the marine environment. The principal methods for *in situ* collection of porewater involve deployed peepers or suction techniques.

Peepers

Peepers consist of small chambers that are covered by a membrane or mesh and inserted into the sediment. A typical mesh pore size is 0.45 microns, which is equivalent to the operational definition of dissolved chemical species. Thus, samples recovered following passage through a 0.45 micron filter are representative of dissolved or aqueous phase COPC concentrations. Although peepers can be deployed with a mesh pore size larger than 0.45 microns, the recovered sample subsequently must be passed through a 0.45 micron filter (such as an in-line syringe filter) to provide a measure of dissolved porewater chemistry. Likewise, porewater samples that appear turbid upon recovery should be filtered through a 0.45 micron filter. The peepers are filled with analyte-free water (typically distilled and deionized water) which, when deployed in sediment, equilibrates with the ambient porewater *via* passive diffusion. The equilibration time is a function of membrane/mesh pore size, peeper volume, sediment type, COPC concentration, and temperature and ranges from days to weeks (USEPA, 2001). Two to four weeks is a typical deployment period, although shorter deployments are possible if the researcher is interested in non-equilibrium conditions (such as for a time-series study). Peepers can be deployed individually or in arrays.

Suction

There are a variety of suction devices available for collecting porewater, including mini-piezometers, suction lysimeters, and the direct insertion of syringe needles into the sediment. A typical suction device consists of a syringe or tube of variable length, with ports located at the desired sampling depth. The device is inserted into the sediment to the desired depth and a manually-operated, spring-

operated, or vacuum gas suction is applied to directly retrieve the porewater sample (Carr and Nipper, 2003). It is important to note that, because it is difficult to draw a sample through a 0.45 micron filter, porewater collected by this method is typically drawn through a larger mesh filter (such as a glass fibre filter or glass frit) before being expressed through a 0.45 micron filter to recover only the dissolved fraction. A variation on this approach employs a porous cup or perforated tube with filters. The unit is inserted into the sediment for a period of time, allowing porewater to infiltrate the chamber before the samples are retrieved by suction (Carr and Nipper, 2003). Depending on the spatial resolution of the sampling, suction methods can yield smaller sample volumes than peepers. Moreover, chemical artefacts associated with sample handling are possible with suction methods, because they typically expose porewater to oxygenated conditions.

Ex situ: *Ex situ* porewater collection methods are often necessary when relatively large volumes of porewater are required (such as for toxicity testing), when *in situ* collection is not viable, or when rapid sampling is required (USEPA, 2001). While *ex situ* extraction can be conducted in the field or in the laboratory, extraction in the laboratory, just prior to analysis or testing, is preferable. Laboratory extraction maintains the sample in its original geochemical state throughout transport and storage and can be conducted in a glove bag under an inert nitrogen (N_2) atmosphere, if required. Centrifugation and squeezing (pneumatic pressure) are the most common techniques for the collection of porewater under the *ex situ* method (Carr and Nipper 2003). *Ex situ* porewater collection methods using centrifugation typically follow the collection of sediment by grab or core sampling. Because information on these sediment collection methods is provided in the relevant sediment SOPs of this guidance manual, the porewater collection procedures described in the following paragraphs focus on *in situ* collection.

Additional information about the porewater sampling techniques described above is provided in the USEPA's *Operating Procedure for Pore Water Sampling* (USEPA, 2013) and *Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual* (USEPA, 2001), as well as Carr and Nipper (2003).

IMPORTANT: Understanding the requirements and limitations of both *in situ* and *ex situ* sample collection will aid in the development of the proper sampling investigation.

POREWATER SAMPLING CHECKLIST

Equipment Checklist:

- ☐ Peepers (that have been pre-assembled by filling the peeper with deionized water, securing mesh or filter over the opening, and sealing in a plastic, zip-top bag for deployment) or suction sampling devices
- ☐ 0.45-micron filters
- ☐ Clean purge water container
- ☐ Peristaltic pump and Teflon® tubing
- ☐ Plastic, zip-top bags
- ☐ Tape
- ☐ Syringe or autopipette with acid-washed pipette tips
- ☐ Certified pre-cleaned sample containers with labels
- ☐ Cooler, packing material and ice packs (for maintaining samples at ≤ 10°C in transit [but not frozen])
- ☐ Waterproof pens/markers
- ☐ Non-powdered nitrile-type gloves
- ☐ Deionized water
- ☐ Diluted (0.01 Molar) acid solution
- ☐ A portable glove bag
- ☐ Nitrogen (N_2) or argon (Ar) for purging the glove bag
- ☐ Field table
- ☐ Field notebook
- ☐ Camera
- ☐ Global Positioning System (GPS) unit
- ☐ Bathymetric map (detailed description of submerged terrain) or nautical charts (primarily for safe navigation) if applicable
- ☐ Hip or chest waders

- ☐ Personal floatation device for all field personnel
- ☐ Boat
- ☐ First aid kit
- ☐ Personal protection equipment (PPE)
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Quality Assurance Program Plan (QAPP)
- ☐ Sampling and Analysis Plan (SAP)
- ☐ Health and Safety Plan (HASP)

Planning and Preparation:

- ☐ Review site-specific information, such as regional land use patterns; site layout and topography; general information on the watershed; water bodies present; water depth; distribution, thickness, and type of sediment; nature of the shoreline; potential onshore and offshore sources of contaminants; ecological habitats and valued ecosystem components (VECs); use of the study area for fishing, shellfish harvest, and boating or recreation; and current and anticipated future use of the water.
- ☐ Conduct site reconnaissance visit to aid in the preparation of the QAPP, SAP, and HASP

IMPORTANT: Prior to initiation of sampling, consider field logistics – identify activities that must be done on the boat or immediately in the field, as opposed to those that could be performed later, on land or at a laboratory or in the office. It is often safest and most efficient to preserve samples in the field and to sort samples in a field trailer or in the laboratory.

- ☐ Prepare QAPP
  - Define and document the data quality objectives, including specific types of Quality Assurance/Quality Control (QA/QC) samples required to meet those data quality objectives.
  - Determine and plan the field quality control samples that will be obtained for the project, including field duplicate samples, field blanks, equipment blanks, and trip blanks.

- Duplicate samples generally should be collected at a rate of 10%, in order to assess sample location variability.

☐ Prepare SAP

- To the extent possible, clearly define the study area boundaries to constrain the sampling area. Then, select appropriately matched reference areas with similar habitat conditions. In lotic (flowing) systems, a suitable reference area is often located immediately upgradient of the study area, beyond the influence of the site. In lentic (static) systems, a suitable water body(ies) within the same watershed, but outside the area of influence, should be selected. The selection process and purpose of each reference site should be clearly stated.

- Identify COPCs. Select sampling and analytical methods and equipment based on site-specific conditions and data quality objectives.

- Determine the minimum volume per sample required for chemical analysis.

- Define the porewater sampling strategy. Determine how porewater data will be spatially correlated with corresponding sediment data. Additional information on these aspects of porewater sampling are provided in Chapter 10 of Volume 1 of the *Guidance Manual*.

IMPORTANT: Consult with the analytical laboratory concerning requirements related to sample volume, holding times, and preservation. Develop an SAP that specifies practices that will prevent exceeding established holding times. The SAP should specify holding times for individual COPCs, especially for COPCs with short holding times that may dictate some aspects of field logistics. Coordinate with laboratory regarding shipping methods and processing.

- Review the study area features and habitat types and devise a sampling location map that provides representative coverage and/or meets the objectives of the project (e.g., hot spot identification).

- Coordinate with the analytical laboratory regarding methods for sample collection, processing, and shipping.

- Consider equipment decontamination procedures. As feasible, use disposable sampling devices to eliminate potential cross-contamination between sample locations. For those instruments that will be reused, the SAP should specify proper decontamination methods to be used in the fields. Proper decontamination methods vary across COPCs and should be confirmed with the analytical laboratory in advance of sampling. All

decontamination materials (e.g., washwater) must be collected and disposed of appropriately.

- List equipment, supplies and procedures related to sample labelling, preservation, decontamination, handling, and shipping.

☐ Prepare HASP (including an emergency plan)

- Determine and plan safety requirements specifically associated with sampling requirements based on study area features, COPCs, and personal health and safety precautions (e.g., personal floatation devices and other boat safety requirements, if applicable, chemical resistant clothing and gloves, respiratory protection, ear and eye protection , *etc.*).

- HASP requirements, including requirements for PPE (e.g., chemical resistant clothing and gloves, respiratory protection, hard hats, ear and eye protection, *etc.*), are based on the site's features and the COPCs. All personnel should be aware of the potential dangers associated with porewater sampling. These dangers include, but are not limited to, strong water currents, slippery substrate, roots or sharp objects that may cause a fall or other personal injury. Additional concerns include the use of heavy objects on a vessel, the presence of "triggers" on some sampling devices that may release unexpectedly (resulting in injury to operators), and overhead hazards from winches or other equipment on the vessel. If sampling is conducted in water greater than three feet deep, sampling personnel are required to wear personal floatation devices.[25]

- Check proper functioning and integrity of PPE, including respirators and filters.

- Hard hats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet.

- File a field plan and boat plan (if appropriate) with a land-based supervisor.

- Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should carry an adequate supply of water or other liquids for protection against dehydration in hot weather.

☐ Reserve, order, and pack all required equipment

[25] DFO and other organizations may have their own safe work practices and requirements for working near or in water. Project-specific safety requirements should be researched and documented in the HASP.

- Obtain and prepare the necessary sampling and storage equipment, sample containers, materials, and documents.

- For efficiency and to reduce field decontamination activities, all sampling equipment should be cleaned and decontaminated at the laboratory or field office before going to the sampling site. If possible, a separate set of cleaned and decontaminated equipment should be available for each sampling site.

- If necessary, reserve a boat and make arrangements for qualified boat operator.

- Prepare a field notebook for documenting all activities related to field sampling. Information to be recorded in the field notebook includes, but is not limited to, weather conditions, progress toward accomplishing sampling objectives, deviations from the field sampling plan, sample identification numbers consistent with what is recorded on the laboratory chain-of-custody forms, sample observations, preservatives used and shipping protocol.

☐ If access to private property will be required, obtain written access permission from landowners

☐ Obtain First Aid/CPR certification and appropriate safety training for all field personnel

☐ Weather forecasts and/or marine conditions reports should be obtained prior to departure for field sampling and sampling should be postponed if conditions are poor

Winter Sampling Safety

Sampling in winter presents extra elements of danger. Always proceed with caution over ice. Do not take unnecessary risks.

Check the ice for thickness with a rod or ice chisel every few steps (ice should be a minimum of 8 centimetres [cm] thick). Use the buddy system, and carry a length of rope (with a harness tied around your waist) to use as a life line. Avoid honeycombed ice and areas over rapids. Be aware that ice downstream from bridge supports and/or outlets may be thin as a result of modified flow patterns and de-icing agents.

IMPORTANT: **Never compromise personal safety or the safety of a field partner.** Always plan ahead to avoid hazards. Always wear appropriate PPE, including personal floatation devices. When working with winches, cables and similar machinery, PPE should include gloves, hard hats, safety glasses and steel-toed boots. Boat operations must conform to all requirements in federal and provincial laws, and a qualified boat operator is required for all sampling conducted from a vessel.

SAMPLE COLLECTION:

Peepers

1) For analytes sensitive to changes in oxidation/reduction (redox) conditions, deoxygenate the peeper assembly prior to deployment.

2) For porewater samples analyzed for dissolved metals, use of a mesh size greater than 0.45 micron will necessitate re-filtering the samples through a 0.45 micron filter prior to their transfer to sample containers.

3) In shallow water, deployment can be completed manually, but in deep water, a diver is usually required.

4) Take precautions to minimize disturbance of the sediment (e.g., propeller wash or excess foot traffic) prior to sampling.

5) Deploy the peepers by removing them from a sealed bag underwater, orienting them lengthwise instead of upright (i.e., orienting the peeper such that the plane of the mesh is vertical instead of horizontal), and burying them in the sediment to a depth consistent with the study's goals and objectives.

6) Deploy the peepers for a sufficiently long period of time for the samples to reach chemical equilibrium with the surrounding porewater. This process may take up to four weeks.

7) For peepers deployed in surface sediment, consider the use of a tracer (such as chloride [Cl^-]) to determine whether equilibrium has been reached within the peeper.

Note: Porewater samples collected for volatile organic compound (VOC) analysis should be removed from the peeper using Teflon or stainless steel sampling equipment. Samples should be collected with as little agitation as possible and laboratory vials should be filled such that there is no headspace or visible bubbles within the sample. Laboratory vials should be glass with Teflon® septa.

Porewater samples collected for metals analysis should be removed from the peeper using Teflon, High-density polyethylene (HDPE) or polycarbonate sampling equipment. Sampling equipment should be rigorously acid-cleaned and rinsed prior to use.

8) To retrieve the peepers following the equilibration period, carefully remove overlying sediment and then immediately seal peepers in zip-top plastic bags and bring to the water surface. Care should be taken to minimize water from the water column in plastic bag.

9) Porewater analytes are sensitive to changes in redox conditions, and the stabilization of the sample to preserve field conditions may be required prior to shipment. Stabilization can include the addition of acid or base (depending on the COPC of interest), or the precipitation of the COPC with specific compounds.

10) In a purged chamber or glove bag containing N_2 or argon, extract the porewater using a clean syringe or pipette and place the extracted porewater in a labelled sample container. A new (pre-cleaned) syringe or pipette tip should be used for each sample.

11) If using a tracer, collect a sample of surface water from the test area to compare its tracer concentration with the tracer concentration in the peepers.

12) If elevated chemical concentrations are suspected in some samples, those samples should be stored separately from samples containing trace concentrations. Reference samples should be stored in designated, separate coolers.

13) Follow proper procedures related to holding conditions (i.e., storage temperature of >0 to 6°C), chain-of-custody, and shipping.

14) Record porewater sampling information on a sampling form and/or in the project field logbook.

15) Document sample location using GPS unit and/or manual measurements to known locations.

16) Following all sampling events, clean and decontaminate sampling equipment, and dispose of used syringes and/or pipette tips. Equipment to be

decontaminated includes the disassembled peepers, glove bag (if not disposable), field table, autopipettes, and other trays or containers employed in organizing the contents of the glove bag. In general, a mild, surfactant detergent cleaning is sufficient for cleaning sampling equipment, but organic solvents may be necessary. Note: Do not clean plastic or polyvinyl chloride (PVC) sampling devices with acetone or other organic solvents that will compromise the structural integrity of the sampling device. Collect and contain all washwater and decontamination materials for appropriate disposal.

IMPORTANT: If an oily or tarry residue is present after washing with a surfactant detergent solution and deionized water, wash the equipment thoroughly with an organic solvent (methanol, acetone, or other) using a separate brush to remove any particulate matter or surface film. Collect the rinseate for proper disposal.

17) All information regarding the sampling event should be accurately recorded in field notes, in order to support the subsequent preparation of a sampling event report.

Suction

1) In flowing systems, begin sampling at the farthest downstream location and proceed toward successively upstream locations. Sampling in this manner will minimize the potential for re-suspended sediment to interfere with sample collection. Stand facing the direction of flow and approach the location from the downstream direction.

2) In static systems, sample areas of suspected elevated chemical concentrations last. Sampling from lowest to highest suspected concentrations will reduce the potential for sample cross-contamination, particularly if any component of the sampling devices must be reused.

3) Take precautions to avoid disturbing the sediment (e.g., propeller wash) prior to collection.

4) Non-powdered nitrile-type gloves should be worn for sampling and should be changed between sampling locations.

5) Drive the sampling device down to 2 cm to 3 cm beyond the target sample depth.

6) Expose a 3-cm section of the stainless steel screen (or equivalent) that corresponds to the target sample depth interval by pulling the drive point up 3 cm past the top of the protective sleeve.

7) Begin collecting porewater from the screened depth using a peristaltic pump and Teflon® tubing (using new tubing for each location).

8) Based on visual observations of fine sediment and/or turbidity caused by the sampling device, it may be necessary to purge the porewater prior to sampling by collecting porewater in a separate container until visually observed to be free of sediment and turbidity. The extent to which it is possible to collect sufficient porewater for purging as well as for analysis will depend on site characteristics, such as the grain size distribution of the sediment and redox conditions, as well as study objectives. The porewater sample recovered by suction will require filtration through a 0.45 micron filter prior to COPC analysis for dissolved chemical species. Transfer the purge water to the appropriate holding container for disposal.

9) Collect porewater samples from the upper end of the tubing into the proper laboratory-supplied containers for analysis, making sure that the discharge syringe or tubing does not contact the sample container.

10) Label each container with: the date, time of sampling, sample identification, the COPCs, the initials of the samplers, and the method of preservation.

11) Retract sampling device and estimate the depth of the porewater sample.

12) Prior to shipment and as soon as possible after the collection, field filter the samples designated for dissolved analyses using a peristaltic pump and 0.45-micron mesh filter. Place the resulting filtrate into a pre-preserved sample container, labelled appropriately. If laboratory containers contain preservatives such as acids, filtering must occur prior to transferring the sample to the laboratory container.

13) If elevated chemical concentrations are suspected in some samples, those samples should be stored separately from samples containing trace chemical concentrations. Reference samples should be stored in designated, separate coolers.

14) Follow proper procedures related to holding conditions (i.e., storage temperature > 0 to 6°C), chain-of-custody, and shipping.

15) Record porewater sampling information on a sampling form and/or in the project field logbook.

16) Document sample location using GPS unit and/or manual measurements to known locations.

17) Clean and decontaminate all reusable sampling devices. In general, a mild, surfactant detergent cleaning is sufficient, but organic solvents may be necessary. Note: do not clean plastic or PVC sampling devices with acetone or other organic solvents that will compromise the structural integrity of the sampling device. Collect and contain all washwater and decontamination materials for appropriate disposal.

Ex Situ Sampling (Centrifugation or Pneumatic Pressure)

1) Recover sediment grab sample or sediment core following appropriate SOP for sediment.

2) For grab samples, quickly transfer sample into laboratory-supplied sample container. For *ex situ* recovery of redox-sensitive porewater analytes, fill sample container completely to limit the diffusion of oxygen into the sediment sample.

3) For sediment cores, extrude or otherwise recover sediment sub-sample for laboratory extraction. For *ex situ* recovery of redox-sensitive porewater analytes extrude sample or open sediment core barrel with a portable glove bag. Fill sample container completely to limit the diffusion of oxygen into the sediment sample.

4) If elevated chemical concentrations are suspected in some samples, those samples should be stored separately from samples containing trace concentrations. Reference samples should be stored in designated, separate coolers.

5) Follow proper procedures related to holding conditions (i.e., storage temperature > 0 to 6°C), chain-of-custody, and shipping.

6) Record sampling information on a sampling form and/or in the project field logbook.

7) Document sample location using GPS unit and/or manual measurements to known locations.

8) Following all sampling events, clean and decontaminate sampling equipment, and dispose of core barrel liners (if used). Equipment to be decontaminated includes the grab sampler or coring devise, glove bag (if not disposable), field table, and trays or containers employed in organizing the contents of the glove bag (if used). In general, a mild, surfactant detergent cleaning is sufficient for cleaning sampling equipment, but organic solvents may be necessary. Note: Do not clean plastic or PVC sampling devices with acetone or other organic solvents that will compromise the structural integrity of the sampling device. Collect and contain all washwater and decontamination materials for appropriate disposal.

9) All information regarding the sampling event should be accurately recorded in field notes, in order to support the subsequent preparation of a sampling event report.

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SUGGESTED OPERATING PROCEDURE NUMBER 13: PLANT SAMPLING

SCOPE This suggested operating procedure (SOP) recommends methods for the collection of representative plant samples for chemical characterization. There are several variations of methods to collect multiple types of plant tissues. Specific procedures may apply to each method in collecting garden plants, other terrestrial plants, or aquatic plant tissue samples. Additional information on plant sampling is provided in Chapter 11 of Volume 1 of the *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as "*Guidance Manual"*). For more information refer to Section 11 of *Protocols Manual for Water Quality Sampling in Canada* (http://www.ccme.ca/files/Resources/water/protocols_document_e_final_101.pdf).

APPLICATION This SOP describes the methods for collection of terrestrial or aquatic plant tissue samples for chemical analysis to support human health and ecological risk assessments.

WHEN? Plant samples may be collected for the following purposes:

1. To characterize exposure to human receptors from contaminants of potential concern (COPCs) in garden vegetables for use in human health risk assessments; and
2. To characterize exposure to valued ecosystem components (VECs) from COPCs in native plants for use in ecological risk assessments.

WHY? Plant tissue may be sampled to characterize the nature and extent of COPC concentrations in terrestrial or aquatic plants in support of human health and ecological risk assessments. Dietary exposure may be directly measured using chemical analysis of plant food items or may be modelled if higher trophic level interactions need to be considered (i.e., ecological risk assessment).

HOW? Plant tissue samples may be collected using a variety of vegetation components. The choice of which plant tissue to collect and the associated method for sampling depend on: COPCs (i.e., pesticides *vs.* metals); vegetation component expected to retain the highest concentrations of COPCs; receptor types (e.g., human or ecological) and their preferred diets; and life history of receptors in relation to the distribution or growth patterns of the target plant tissue (i.e., seasonal or year-round use).

TYPES ***Garden (terrestrial) samples***: This type of sample is typically used in human health risk assessments to characterize residential scenarios with respect to dietary exposures to COPCs. Garden plant tissue samples are intended to be representative of the part(s) of the plant that are consumed, and they are usually

rinsed to remove dirt before consumption. Garden plant tissue samples include vegetation that is:

- Collected from a residential garden plot or agricultural field;
- Collected as individual plant samples, rather than as a composite of multiple plants of the same species; and
- Composed of a single type of plant tissue (the part consumed, i.e., only leaves, only fruit, only roots), rather than as a composite of several plant tissue types.

Other Terrestrial Plant Samples: This type of sample is most often used in ecological risk assessments and occasionally in human health risk assessments. Samples are usually composed of berries or other edible fruits, grasses, leaves, or tubers (depending on the dietary preferences of receptors). When used in support of ecological risk assessments, this type of sample is not rinsed, in order to simulate actual exposure patterns of VECs. When used in support of human health risk assessments, this type of sample is rinsed to simulate human exposure scenarios through produce consumption.

Aquatic samples: This type of sample is typically used in ecological risk assessments to measure or model dietary exposure to VECs. Aquatic plant samples include vegetation that is:

- Collected from a single sample location;
- Composed of multiple plants from the same sampling location; and
- Composed of all tissue types from a single plant or multiple plants collected at the sampling location.

Essential Information

The Sampling and Analysis Plan (SAP) should specify whether data will be used only for human health risk assessment or for both human and ecological risk assessment purposes. If data will only be used for human health risk assessment, samples should be collected at the same time that plants are normally harvested. The SAP should specify handling procedures (i.e., washing, peeling).

COLLECTION Prior to initiating sample collection, COPCs warrant careful consideration, in order to define practices that will prevent cross-contamination of the sample with residues from sampling or processing materials. If metals are COPCs, plastic sampling equipment should be used where possible and no sampling equipment should contain lead or be painted. Additional detailed procedures for various classes of target analytes are described in Health Canada's *Supplemental*

Guidance on Human Health Risk Assessment for Country Foods (Health Canada, 2007), in the U.S. Environmental Protection Agency's Region 8 Standard Operating Procedure for *Garden Vegetable Sampling at Residences for Determination of Risk-based Exposure to Metals* (USEPA 1999), Washington State's *Aquatic Plant Sampling Protocols* (Ecology 2001), and in the U.S. Department of Agriculture's Standard Operating Procedure for *Collection of Vegetation Samples* (USDA 2002).

SAMPLING CHECKLIST

Equipment Checklist for Terrestrial Plant Samples:

- ☐ Plant collection tools including a shovel (lead-free and unpainted) for collecting root tissue, rake for collecting aquatic plant tissue, and hand trowel (plastic or steel)
- ☐ Stainless steel scalpel, garden shears or clippers
- ☐ Zip-top plastic bags or paper bags or envelopes if specified by the laboratory
- ☐ Aluminum foil (if specified by the laboratory to prevent contact of tissue samples with plastic)
- ☐ Deionized water
- ☐ Non-powdered, nitrile-type gloves
- ☐ Plastic buckets (17-litre capacity) and vegetable brush (for washing vegetables)
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Field notebook and permanent markers
- ☐ Scale and ruler
- ☐ Garbage bags
- ☐ Cooler, packing material and ice packs (for maintaining samples at $4^{o}C$)
- ☐ First aid kit
- ☐ Personal protection equipment (PPE)
- ☐ Camera
- ☐ Quality Assurance Program Plan (QAPP)

- ☐ Sampling and Analysis Plan (SAP)
- ☐ Health and Safety Plan (HASP)

Equipment Checklist Aquatic Samples:

- ☐ Boat, if needed, and qualified boat operator
- ☐ Personal floatation devices
- ☐ Bathymetric map (detailed description of submerged terrain) or nautical charts (primarily for safe navigation) if applicable
- ☐ Field data sheets
- ☐ Weighted sounding line
- ☐ Weighted rake and retrieving line
- ☐ Zip-top plastic bags
- ☐ Aluminum foil (if specified by the laboratory to prevent contact of tissue samples with plastic)
- ☐ Clipboard
- ☐ Copy of monitoring procedures
- ☐ Deionized water
- ☐ Non-powdered nitrile-type gloves
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Field notebook and permanent markers
- ☐ Garbage bags
- ☐ Cooler, packing material and ice packs (for maintaining samples at 4°C)
- ☐ First aid kit
- ☐ PPE
- ☐ Camera
- ☐ GPS

☐ QAPP

☐ SAP

☐ HASP

Planning and Preparation:

IMPORTANT: Prior to initiating any sampling, coordinate with the analytical laboratory to determine whether using aluminum foil will cross-contaminate any samples to be analyzed for metals or other COPCs. If so, avoid using foil and simply place plants in the plastic bag and freeze. As with other biota samples, plants are typically shipped to the analytical laboratory fresh (un-dried). Discuss the plant analysis methods with the laboratory personnel ahead of time. Laboratory personnel may recommend drying (at low temperature) prior to analysis. Drying is most effectively done by the laboratory, rather than in the field.

☐ Review site-specific information, such as regional land use patterns; site layout and topography; general information on the watershed; water bodies present; water depth; distribution, thickness, and type of sediment; nature of the shoreline; potential onshore and offshore sources of contaminants; ecological habitats and VECs; use of the study area for fishing, shellfish harvest, and boating or recreation; and current and anticipated future use of the water.

☐ Use the reconnaissance survey to generally characterize relative plant densities and the approximate number of sampling locations and sample mass required for the target analytes

☐ Prepare QAPP

- Define and document the data quality objectives, including specific types of Quality Assurance/Quality Control (QA/QC) samples required to meet those data quality objectives. Duplicate samples generally should be collected at a rate of 10%, in order to assess sample location variability.

☐ Prepare SAP

- Complete a detailed review of project objectives and possible target analytes.
- Determine the type of plant and plant tissue type(s) most likely to reflect exposure to the target analytes.
- Determine the proper season for sampling. This usually comes when the plant is under stress from flowering to early fruit or seed stage.

- Determine whether federal/provincial/territorial protected species are likely to be present. If so, sampling program should be designed to avoid these species.

- Review the study area features and habitat types and devise a sampling design that provides representative coverage of habitat types, harvesting practices of human receptors, and/or foraging behaviours of VECs.

- Consult with analytical laboratory to determine the minimum mass per sample or composite sample required for chemical analysis.

- Consult the analytical laboratory to determine preferred storage for plant materials (some may suggest using paper bags to prevent decomposition of the sample). Review the SAP and coordinate with the laboratory to determine the appropriate steps for pooling multiple plants per sample for a composite or whether individual plants should be collected and shipped for combination in a composite sample later. If more than one plant or tissue type is necessary to meet minimum weight requirements, clarify the implications of collecting a composite sample for chemical analyses. Finally, clarify the desired procedure for removal of soil particles from plant tissues, especially roots, and determine whether or not the sample should be rinsed prior to shipment to the analytical laboratory.

- List equipment, supplies and procedures related to sample labelling, preservation, decontamination, handling, and shipping.

IMPORTANT: Consult with the analytical laboratory concerning requirements related to sample volume, holding times, and preservation. Develop a SAP that specifies practices that will prevent exceeding established holding times. The SAP should specify holding times for individual COPCs, especially for COPCs with short holding times that may dictate some aspects of field logistics. Coordinate with laboratory regarding shipping methods and processing.

☐ Prepare HASP (including an emergency plan)

- Determine and plan safety requirements specifically associated with sampling requirements based on study area features, COPCs, and personal health and safety precautions (e.g., chemical resistant clothing and gloves, respiratory protection, ear and eye protection, *etc.*).

- Check proper functioning and integrity of PPE, including respirators and filters.

- If formaldehyde solution will be used, it must be shipped as Dangerous Goods, and treated in a manner consistent with Workplace Hazardous

Materials Information System (WHMIS) requirements (e.g., stored in the hazardous chemical storage locker and used under a fume hood, with material safety data sheets provided).

- Hard hats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet.
- File a field plan and a boat plan (if appropriate) with a land-based supervisor.
- Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should carry an adequate supply of water or other liquids for protection against dehydration in hot weather.

☐ Reserve, order, and pack all required equipment

- Obtain and prepare the necessary sampling and storage equipment, sample containers, materials, and documents.
- Check proper functioning and integrity of field equipment.
- All sampling equipment should be cleaned and decontaminated at the laboratory or field office before going to the sampling site. If possible, a separate set of cleaned and decontaminated equipment should be available for each sampling site.
- Prepare a field notebook for documenting all activities related to field sampling. Information to be recorded in the field notebook includes, but is not limited to, weather conditions, progress toward accomplishing sampling objectives, deviations from the field sampling plan, sample identification numbers consistent with what is recorded on the laboratory chain-of-custody forms, sample observations, preservatives used and shipping protocol.

☐ Determine whether permits are needed for plant collection, and obtain any required permits from the appropriate territorial, provincial, and/or federal regulatory agencies

☐ If access to private property will be required, obtain written access permission from landowners

☐ Obtain First Aid/CPR certification and appropriate safety training for all field personnel

Sample Collection:

IMPORTANT: **Never compromise your personal safety or that of a field partner to collect a sample.** Always plan ahead to avoid falling and drowning hazards. Always wear appropriate safety gear such as personal floatation devices. When working with winches, cables, and similar machinery, gloves, hard hats, safety glasses, and steel-toed boots are important safety items. A qualified boat operator is required for all transport or sampling from a boat. Boat operations must conform to all requirements in federal and provincial laws, and a qualified boat operator is required for all sampling conducted from a vessel.

1) Based on the SAP, identify specific parts of the plant targeted for sampling for each species. Subsections below include instructions for sample collection for leafy vegetables, root vegetables, fruit, and aquatic plants.

2) Obtain samples in a random fashion from all parts of the field and collect a sufficient mass of sample.

3) Each sample will be labelled by affixing a Sample Identification Label to the sample container/package. All sample label entries should be made with black indelible ink.

4) Each sample label should include the following information:

   - ☐ project name
   - ☐ site identification
   - ☐ sample number
   - ☐ date of sample
   - ☐ time of collection
   - ☐ preservative used
   - ☐ collector's name
   - ☐ type of analysis required

5) Samples may be taken back to a laboratory whole for processing (e.g., cutting). If this is not practical, process in the field using stainless steel scalpels, or garden sheers or clippers if material is too thick.

6) Wrap plant tissue in aluminum foil and seal in a labelled zip-top bag.

7) Sample should be immediately stored at 4°C to minimize sample decomposition during storage and shipment. It is strongly recommended that an overnight courier service is used for shipping samples.

8)

*Leafy Vegetables (*e.g.*, lettuce, spinach, cilantro; also applies to other herbaceous plants sampled for ecological purposes)*

1) Document the sample location with a GPS unit and/or manual measurements to known locations. Also, photograph each sample location and record the photograph number in the field notebook.

2) While wearing non-powdered nitrile-type gloves, select a single plant and record the location of the plant on the sample study area map and harvest the plant in a manner consistent with how the plant would be harvested by human or ecological receptors.

3) If samples will be rinsed prior to analysis, clean the plant tissue in a clean 17-litre bucket filled with deionized water and gently agitate or wash soil particles from the plant. Samples are not typically rinsed if collected for ecological risk assessment purposes.

4) Remove the plant tissue and gently shake off to drain the water. Photograph sample and record photograph number in field notebook. Wrap in aluminum foil and seal in a labelled zip-top bag.

5) Freeze or store at 4°C before shipping to the analytical laboratory for chemical analysis.

6) After sampling is complete, rinse all sampling tools with tap water and a mild, non-phosphate detergent, followed by triple rinsing with deionized water. Discard all disposable sampling supplies in the garbage bag and dispose according to the project health and safety plan.

*Root Vegetables (*e.g.*, potatoes, carrots, onions; also applies to other roots, such as cattail tubers, that may be sampled for ecological purposes)*

2) Follow the steps described above for leafy vegetables, with this exception, remove the whole plant, cut off the top of the plant and shake off as much of the soil as possible if this tissue is not being sampled.

2) If samples will be rinsed prior to analysis, clean the plant in a bucket of deionized water using a vegetable brush to remove any soil particles. Rinse the scrubbed root tissue with deionized water and then wrap in aluminum foil and seal in a labelled zip-top bag. Samples are not typically rinsed if being collected for ecological risk assessment purposes.

3) Follow the steps outlined above for storage, shipping, and decontamination.

*Fruit (*e.g.*, squash, beans, tomatoes; also applies to berries and other fruits that may be sampled for ecological purposes)*

1) Follow the steps described above for leafy and root vegetables, except manually remove the fruit from a vine or stalk or cut it off using decontaminated scalpel, snippers or shears.

2) Immerse in a bucket with deionized water and wipe off any soil residue (if being used for human health risk assessment purposes). For fruits such as corn, use a stainless steel knife to remove kernels and allow them to directly fall into the zip-top bag.

3) Follow the steps outlined above for storage, shipping, and decontamination.

Aquatic Vegetation

3) While wearing non-powdered nitrile-type gloves, use a rake or garden tool to remove the entire plant from aquatic media (applies to both emergent and submergent vegetation). Shake the plant to remove extra water.

2) Examine plants for presence of invertebrates (e.g., snails, small minnows, other biota), which can be removed by hand.

3) Follow the steps outlined above for storage, shipping, and decontamination.

REFERENCES

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SUGGESTED OPERATING PROCEDURE NUMBER 14: TERRESTRIAL INVERTEBRATE SAMPLING

SCOPE This suggested operating procedure (SOP) provides general direction and guidance for collecting representative terrestrial invertebrate tissue samples for chemical analysis. Terrestrial invertebrates encompass organisms from a wide range of taxa, from the most primitive (worms) to highly evolved (insects).

There are several options for collecting invertebrate tissue samples and specific procedures may apply to each method in the acquisition of the tissue samples. However, detailed descriptions of procedures that are infrequently used are beyond the scope of this SOP. Additional information on terrestrial invertebrate tissue sampling is provided in Chapter 11 of Volume 1 of the *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as "*Guidance Manual"*).

APPLICATION This SOP describes the methods for collecting terrestrial invertebrate tissue samples for chemical analysis in support of ecological risk assessments.

WHEN? Terrestrial invertebrate tissue samples are usually collected to characterize concentrations of chemicals of potential concern (COPCs) in terrestrial invertebrates, in order to allow estimation of dietary exposures for valued ecosystem components (VECs) in ecological risk assessments.

WHY? Terrestrial invertebrate tissue sampling is typically conducted to evaluate bioavailability of COPCs in soil and to obtain direct measures of the dietary exposure of invertivorous VECs for ecological risk assessment.

HOW? The choice of sampling method for terrestrial invertebrates primarily depends on: target organism (e.g., worms, spiders, insects); COPCs; habitat type to be characterized for exposure (e.g., forests *vs*. grasslands); and sensitivity of the target organism to the sampling method (i.e., pitfall traps *vs.* soil excavation).

TYPES ***Pitfall traps***: Pitfall trapping is the preferred method of collecting terrestrial invertebrates that live above ground, rather than in the soil.

Soil excavation: Soil excavation is the preferred method of collecting terrestrial invertebrates that live in the soil or below ground, such as worms.

Additional information on these and other trapping methods is provided in *Ecological Methods with Particular Reference to the Study of Insect Populations* (Southwood, 1978) and *Inventory Methods for Terrestrial Arthropods, Standards for Components of British Columbia's Biodiversity No. 40* (BCMOE, 1998).

COLLECTION An important consideration in terrestrial invertebrate tissue sampling is the method by which samples will be collected and shipped. Prior to initiation sampling, coordinate with the analytical laboratory to verify that sampling equipment will not cross-contaminate samples.

IMPORTANT: Terrestrial invertebrates all have toxins for defensive purposes and exposure to such toxins may cause an allergic reaction in people.

If bitten, stung, or exposed to urticating hairs or millipede toxin:

- Immediately wash the exposed area with hot water and antibacterial soap. Try to use the hottest water temperature without burning your skin. Hot water works best to help neutralize the venom.
- If you are exposed to urticating hairs then wash the area with hot water and antibacterial soap. **Do not touch** your face or eyes until you have sought medical help to remove all of the hairs.
- Seek medical attention if you believe you are having an allergic reaction or if you have been exposed to urticating hairs.
- If seeking medical attention, be sure to write down the species of terrestrial invertebrate you were exposed to in order to ensure proper treatment.

Note: The information is not a substitute for medical diagnosis and treatment.

TERRESTRIAL INVERTEBRATE TISSUE SAMPLING CHECKLIST

Equipment Checklist:

☐ Stainless steel shovel, hand auger, or garden tool for soil excavation (preferable stainless steel unless equipment will not contact organisms being sampled, e.g., shovel can be used to bring up shovelful of soil and then earthworms can be picked by hand from the soil).

☐ Non-powdered nitrile-type gloves for handling invertebrates

☐ Tweezers or forceps to handle invertebrates

☐ Sieve or mesh covered sorting tray or stainless steel bowl

☐ Field notebook and permanent markers

☐ Invertebrate identification guide

☐ Plastic containers with lids or cover boards for pitfall traps

☐ Global Positioning System (GPS) unit and flagging tape to mark pitfall trap locations

- ☐ Personal protective equipment (PPE)
- ☐ Decontamination bucket (17-litre) with scrubber
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Deionized water
- ☐ Aluminum foil
- ☐ Large zip-top plastic bags
- ☐ Scale and ruler
- ☐ Garbage bags
- ☐ Cooler, packing material and ice packs (for maintaining samples at 4°C)
- ☐ First aid kit
- ☐ Camera
- ☐ Quality Assurance Program Plan (QAPP)
- ☐ Sampling and Safety Plan (SAP)
- ☐ Health and Safety Plan (HASP)

IMPORTANT: Prior to initializing sampling, coordinate with the analytical laboratory to determine whether using foil in the plastic bags will cross-contaminate samples analyzed for metals or other target analytes. If so, avoid using foil and simply place invertebrates in the plastic bag and freeze.

Planning and Preparation:

- ☐ Review site-specific information, such as regional land use patterns; site layout and topography; general information on the watershed; water bodies present; water depth; distribution, thickness, and type of sediment; nature of the shoreline; potential onshore and offshore sources of chemicals; ecological habitats and VECs; use of the study area for fishing, shellfish harvest, and boating or recreation; and current and anticipated future use of the water.
- ☐ Conduct site reconnaissance visit to aid in the preparation of the QAPP, SAP, and HASP

☐ Use the reconnaissance survey to generally characterize relative invertebrate densities and the approximate number of sampling locations and sample mass required for the COPCs

☐ Prepare QAPP

- Define and document the data quality objectives, including specific types of Quality Assurance/Quality Control (QA/QC) samples required to meet those data quality objectives. Duplicate samples generally should be collected at a rate of 10%, in order to assess sample location variability.

☐ Prepare SAP

Essential Information

☐ The Sampling and Analysis Plan (SAP) should specify targeted types of invertebrates and whether surface-dwelling or sub-surface-dwelling invertebrates are targeted. The plan also should specify the degree of compositing to be done (i.e., whether or not to use only earthworms, or whether to use all invertebrates encountered).

☐ An invertebrate field guide is useful for keying and sorting invertebrates to an appropriate taxonomic level.

- Determine the life histories of the target organisms to verify whether they are active and available for sampling during the target time of year and weather conditions. Consult web resources such as the *Biological Survey of Canada for Terrestrial Invertebrates* for keys and life history information (http://biologicalsurvey.ca/).

- Determine whether federal/provincial/territorial protected species are likely to be present. If so, select a sampling method that can minimize harm to these species.

- Review the study area features and habitat types and devise a sampling location map that provides representative coverage of habitat types with exposure.

- Consult with the analytical laboratory to determine the minimum mass per sample or composite sample required for chemical analysis.

- Determine whether tissue samples from soil excavation will be purged of ingested soil (i.e., depurated). If depuration is desired, plan for an additional 24 hour period to allow passage of the soil from the live target organisms before submitting for chemical analyses. Coordinate with the laboratory to determine appropriate holding times and to verify that a time allowance for depuration will not compromise chemical analyses.

- Determine whether invertebrates will be washed to remove excess soil and, if so, specify method that minimizes handling and uses deionized water.

- Coordinate with the analytical laboratory regarding methods for sample collection, processing, and shipping. Discuss the desired preparation method (e.g., depuration) for chemical analysis.

- List equipment, supplies and procedures related to sample labelling, preservation, decontamination, handling, and shipping.

IMPORTANT: Consult with the analytical laboratory concerning requirements related to sample volume, holding times, and preservation. Develop an SAP that specifies practices that will prevent exceeding established holding times. The SAP should specify holding times for individual COPCs, especially for COPCs with short holding times that may dictate some aspects of field logistics. Coordinate with laboratory regarding shipping methods and processing.

□ Prepare HASP (including an emergency plan)

- Determine and plan safety requirements specifically associated with sampling requirements based on study area features, COPCs, and personal health and safety precautions (e.g., personal floatation devices and other boat safety requirements, if applicable, chemically resistant clothing and gloves, respiratory protection, ear and eye protection , *etc.*).

- Check proper functioning and integrity of PPE, including respirators and filters.

- If formaldehyde solution will be used, it must be shipped as Dangerous Goods, and treated in a manner consistent with Workplace Hazardous Materials Information System (WHMIS) requirements (e.g., stored in the hazardous chemical storage locker and used under a fume hood, with material safety data sheets provided).

- Hard hats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet.

- File a field plan with a land-based supervisor.

- Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should carry an adequate supply of water or other liquids for protection against dehydration in hot weather.

☐ Reserve, order, and pack all required equipment

- Obtain and prepare the necessary sampling and storage equipment, sample containers, materials, and documents.

- For efficiency and to reduce field decontamination activities, all sampling equipment should be cleaned and decontaminated at the laboratory or field office before going to the sampling site. If possible, a separate set of cleaned and decontaminated equipment should be available for each sampling site.

☐ Determine whether permits are needed for trapping or insect handling and obtain any required permits from the appropriate territorial, provincial and/or federal regulatory agencies

☐ If access to private property will be required, obtain written access permission from landowners

☐ Obtain First Aid/CPR certification and appropriate safety training for all field personnel

IMPORTANT: Biological samples collected for chemical analysis *should not* be chemically preserved, while biological samples collected for taxonomy *should* be chemically preserved. Consult with the taxonomy laboratory in advance to determine the appropriate preservative and container for use with that preservative. If both types of samples are being collected, store the two types of biological samples in separate, clearly marked containers.

Sample Collection:

Pitfall Trapping (used to collect invertebrates living at the soil surface)

4) Establish a trapping grid or other appropriate sampling design for pitfall trapping locations. Use a GPS unit and/or flagging to mark trap location.

2) Using a shovel or handheld auger, dig a hole slightly deeper and wider than the plastic container that will be used. Place container in the hole, so that lid is flush with (or slightly below) the ground surface. Firmly compact the soil around the bucket.

3) Suspend a wooden or plastic plate or lid approximately 2.5 centimetres (cm) above the trap to protect the trap from rain. These covers should be weighted with a stone and supported by 7.5 cm to 15 cm square wooden or hollow aluminum rods, set at right angles around the trap mouth to form an X. These support rods should touch the mouth of the trap for efficient capture of specimens. Support rods arranged in this fashion not only support the cover firmly, but also intercept animals moving near the trap and funnel them into the trap. The covers

serve to keep out debris and prevent the entry of animals and precipitation from above.

4) Record the trap location using GPS unit and/or manual measurements to known locations. Use flagging tape or pin-flags to provide a visible marker for the field technician when checking traps. Photograph each trap location and record the photograph number in the field notebook.

5) Check the traps each morning and evening. If invertebrates are captured, remove and sort them to the appropriate taxonomic level, photograph the sample, place organisms in a labelled zip-top bag lined with aluminum foil, and freeze or store at 4°C for shipping to the laboratory for chemical analysis. It is recommended to handle them as little as possible. Biota for taxonomy should be chemically preserved because freezing may damage tissues and hinder identification.

6) Always wear dry non-powdered nitrile-type gloves.

7) Always wash hands thoroughly – before and after handling invertebrates – with antibacterial soap and water.

8) Decontaminate all sampling equipment using tap water to remove soil particles followed by a surfactant and additional rinsing with deionized water, as described in the SAP.

9) All information regarding the sampling event should be accurately recorded in field notes, in order to support the subsequent preparation of a sampling event report.

Soil Excavation (used to collect soil-dwelling invertebrates)

1) Document the sample locations using GPS unit and/or manual measurements to known locations. Photograph each sample location and record the photograph number in the field notebook.

2) Determine the appropriate depth for soil excavation samples and use the shovel or hand auger to excavate to this depth. Adjust the sampling area at each location to account for the minimum mass required for chemical analysis.

3) Always wear dry non-powdered nitrile-type gloves.

4) After excavating each sampling location using a shovel, place soil material in a sieve or tray or bowl, and gently break apart soil, removing invertebrates as they are encountered and placing them in a sample container.

5) If depuration is desired, place living organisms in a sorting pan with moistened filter paper (use deionized water) and allow 24 hours for ingested soil to be excreted prior to chemical analyses.

6) Combine invertebrate tissue, if necessary, into composite samples to achieve the minimum mass necessary for chemical analysis. Decisions regarding which organisms are grouped together in a composite sample depend on the study objectives. For example, if data may be used to develop site-specific bioaccumulation factors, the proximity of each organism to the associated soil sample will influence compositing decisions. Alternatively, if the invertebrate samples will be used to characterize exposure to birds and mammals, it may be appropriate to group all types of invertebrates likely to be consumed across the bird's or mammal's foraging area. To the extent possible, identify invertebrate taxa in the field using field guides. Note the approximate numbers of different types of organisms included in each composite sample.

7) If specified in the SAP, wash invertebrates with deionized water to remove excess soil. However, organisms should be handled as little as possible.

8) Always wash hands thoroughly – before and after handling invertebrates – with antibacterial soap and water.

9) Photodocument the sample, and place in a labelled zip-top bag, lined with aluminum foil. Freeze or store at 4°C for shipping. Avoid crushing or compacting samples. Biota for taxonomy should be chemically preserved because freezing may damage tissues and hinder identification.

10) Decontaminate all sampling equipment using tap water to remove soil particles followed by a surfactant and additional rinsing with deionized water, as described in the SAP.

11) All information regarding the sampling event should be accurately recorded in field notes, in order to support the subsequent preparation of a sampling event report.

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SUGGESTED OPERATING PROCEDURE NUMBER 15: BENTHIC INVERTEBRATE COLLECTION AND PROCESSING

SCOPE This suggested operating procedure (SOP) recommends methods for collecting and processing benthic invertebrates in freshwater, estuarine, and marine environments to ensure quality control in field operations and uniformity among technicians. Additional information on benthic invertebrate collection and processing is provided in Chapter 11 of Volume 1 of the *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as *"Guidance Manual"*). For more information refer to Section 9 of *Protocols Manual for Water Quality Sampling in Canada* (http://www.ccme.ca/files/Resources/water/protocols_document_e_final_101.pdf).

APPLICATION This SOP describes the methods for collecting and processing benthic invertebrates in the field for both tissue analysis and taxonomy (in support of benthic community assessment) for use in human health and ecological risk assessments.

WHEN? Benthic invertebrate sampling is frequently used in ecological risk assessments to evaluate benthic invertebrate community structure, as well as to estimate exposure valued ecosystem components (VECs) that consume invertebrates. Benthic community assessment is commonly used in ecological risk assessments to evaluate environmental condition as part of the sediment quality triad approach (for example, see Environment Canada and Ontario Ministry of the Environment, 2007, for guidance on using this approach to assess contaminated sediments). Tissue sampling and analysis of some aquatic invertebrates (e.g., mussels, clams, and crabs) may also be used in human health risk assessments to estimate exposure to people who consume those organisms.

WHY? The assessment of benthic invertebrates is considered integral to the evaluation of contaminated sediments in Canada. Benthic community structure analysis is often used in ecological risk assessments to evaluate the biological condition of the study area, relative to that of reference areas. Benthic invertebrate sampling and processing can be conducted to characterize exposure of benthic invertebrates and VECs that consume them to contaminants of potential concern (COPCs). Benthic invertebrate tissue sampling and chemical analysis may also be conducted to estimate bioaccumulation and predict potential concerns due to biomagnification.

HOW? Benthic invertebrate samples can be collected using a variety of methods. The choice of sampling method for freshwater, estuarine, and marine environments depends primarily on: target organisms; habitat present at the study area; and water depth.

TYPES **Organism Traps:** Commercially available minnow, crab, or lobster traps are constructed of wire or mesh. The traps have built in funnel entrances to allow

large macroinvertebrates to enter, but not exit. Many such traps (e.g., minnow traps) are easily constructed. Traps are deployed on the sediment surface and retrieved at least once daily. Minnow traps are most effective for collecting mobile macroinvertebrates. Commercial crab traps or lobster traps may be necessary for the collection of larger macroinvertebrates (e.g., dungeness or rock crab, lobster). It is important to bear in mind that traps in visible locations may be disturbed or vandalized, and therefore precautions should be taken to protect the equipment and samples.

Sediment Sampler: Upon retrieval of the sediment sampling device, the collected sediment is either placed directly into a sample container and preserved, or it may be released into a sieve bucket, box sieve, or dip net to allow fine sediment to be rinsed from the sample and reduce sample volume (particularly useful for samples being shipped). U.S. 30 mesh screen is typical for freshwater, while smaller sized mesh – e.g., 0.5 to 0.1 millimetre (mm) – may be more useful for marine sampling. The invertebrates and remaining sediment that accumulate in the sieve are rinsed into a pre-cleaned and labelled sample container for storage until subsequent processing in the field or laboratory and analysis. The sediment sampling SOPs included with this guidance manual provide additional information on sediment sampling techniques.

Hand Digging: In tidal flats or shallow inlets, the most efficient way to collect molluscs, such as geoducks, clams, and mussels, is often by hand picking and/or using a clam rake. Some molluscs (e.g., mussels) can also be collected in the dry. Identification of candidate areas for digging is done by visual inspection. Water must be shallow enough to see the bottom, preferably on an outgoing tide. Target locations are identified by looking for holes in the mud or sand that indicate molluscs have been filtering water. Dark, muddy spots can indicate where the clam filtered seawater and left a “stain” of mud on the surface. Mussels may colonize fixed features with the intertidal zone, such as boulders, piers, and docks. For collecting clams in Pacific Region (e.g., geoducks), a full-size shovel may be required.

Dip Net: In streams and other shallow water, dip nets are often used to obtain invertebrate samples representing different habitats, such as riffles, runs, undercut banks, and leaf packs. Dip nets are available in several sizes with varying handle lengths to accommodate different water body depths and substrates.

Kick Net: Benthic invertebrate samples in flowing streams are sometimes collected using kick nets by placing the bottom of the net on the stream bed, perpendicular to water flow and using the foot or hand to dislodge organisms from substrate upstream of the net.

Surber Samplers and Hess Samplers: These two sampling devices are used to sample epibenthos in streams. The base of sampler is embedded in substrate, with the sampler body mesh facing upstream and the sample-collection net facing downstream. Large substrate elements upstream from the sampler are picked up

and held in the current in front of the collection net, while macroinvertebrates are scrubbed from the surface using a wire brush.

Additional information on benthic invertebrate sampling devices and methods is provided in the references at the end of this SOP (e.g., Canadian Aquatic Biomonitoring Network [CABIN], Florida Department of Environmental Protection, 2009; Jones et al., 2004; United States Environmental Protection Agency [USEPA], 2000; USEPA, 2013).

Essential Information

Determine in advance the desired level of taxonomic sorting required in the field. Coordinate with the analytical laboratory to determine appropriate storage and preservation techniques for all major taxa.

COLLECTION Field sampling and sorting are important considerations in benthic invertebrate tissue collecting and processing. The desired level of taxonomic sorting in the field should be determined prior to beginning the sampling program. The level of sorting required is sometimes dictated by the planned data analyses. The Canadian Aquatic Biomonitoring Network (CABIN) protocol specifies that the family level of taxonomic organization is sufficient for benthos, while other protocols may require genus or genus and species. Because CABIN was developed for freshwater, if it is to be applied to marine or estuarine environments, the Sampling and Analysis Plan (SAP) should explicitly describe the required modifications to the protocol. If samples are being submitted to a laboratory for additional taxonomic sorting, sample handling and storage methods should be discussed in advance with the laboratory. In addition, prior to initiating the sampling program, if tissue sampling and analysis will be performed (e.g., COPC and/or lipid content analysis) determine the minimum sample mass and methods for processing and shipping.

SAMPLING CHECKLIST

Equipment Checklist:

Organism Traps

- ☐ Minnow traps, crab traps, lobster traps or other types of organism traps
- ☐ Rope and/or twine
- ☐ Buoy for surface marker
- ☐ Stakes, flagging, and/or Global Positioning System (GPS) unit
- ☐ Stainless steel sorting bowls or trays

- ☐ Forceps
- ☐ Scale and ruler
- ☐ Deionized water
- ☐ Pre-cleaned and labelled sample bags or containers
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Aluminum foil
- ☐ Fixing solution (e.g., buffered formalin) if needed for taxonomic identification
- ☐ Cooler, packing material and ice or dry ice (for tissue chemistry samples)
- ☐ Non-powdered, nitrile-type gloves
- ☐ Field notebook and permanent pens/markers
- ☐ Waders
- ☐ Personal protection equipment (PPE)
- ☐ First aid kit
- ☐ Camera
- ☐ Quality Assurance Program Plan (QAPP)
- ☐ Sampling and Analysis Plan (SAP)
- ☐ Health and Safety Plan (HASP)

Sediment Samplers

- ☐ Sediment sampler, such as a Ponar, petite Ponar, or van Veen grab
- ☐ Sieve bucket, or D-frame dip net (mesh size may vary depending on the system). U.S. 30 mesh screen is typical for freshwater, while smaller sized mesh – e.g., 0.5 to 0.1 millimetre (mm) – may be more useful for marine sampling.
- ☐ Fixing solution (e.g., buffered formalin) for benthic community survey
- ☐ Cooler, packing material and ice or dry ice (for tissue chemistry samples)

- ☐ Stainless steel sorting bowls or trays
- ☐ Forceps
- ☐ Scale and ruler
- ☐ Deionized water
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Pre-cleaned and labelled sample bags or containers
- ☐ Aluminum foil
- ☐ Non-powdered, nitrile-type gloves
- ☐ Field notebook and permanent pens/markers
- ☐ Waders
- ☐ PPE
- ☐ Boat, if needed, and personal floatation devices for all field personnel
- ☐ Bathymetric map (detailed description of submerged terrain) or nautical charts (primarily for safe navigation) if applicable
- ☐ First aid kit
- ☐ Camera
- ☐ QAPP
- ☐ SAP
- ☐ HASP

Hand Picking

- ☐ Clean rake, garden trowel, or shovel for clams and mussels, oyster knife or pry tool for encrusting organisms
- ☐ Pre-cleaned and labelled sample buckets or collecting containers
- ☐ Deionized water

- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Pre-cleaned and labelled sample bags or containers
- ☐ Aluminum foil
- ☐ Cooler, packing material and ice or dry ice (for tissue chemistry samples)
- ☐ Scale and ruler
- ☐ Non-powdered, nitrile-type gloves
- ☐ Field notebook and permanent pens/markers
- ☐ Waders
- ☐ PPE
- ☐ First aid kit
- ☐ Camera
- ☐ QAPP
- ☐ SAP
- ☐ HASP

Dip Net

- ☐ D-frame dip net with No. 30 mesh and handle marked in 0.1-metre (m) increments
- ☐ Deionized water
- ☐ Forceps
- ☐ Scale and ruler
- ☐ Pre-cleaned and labelled sample bags or containers
- ☐ Aluminum foil
- ☐ Stainless steel sorting bowls or trays

- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Cooler, packing material and ice or dry ice (for tissue chemistry samples)
- ☐ Fixing solution (e.g., buffered formalin) for benthic community survey
- ☐ Non-powdered, nitrile-type gloves
- ☐ Field notebook and permanent pens/markers
- ☐ Waders
- ☐ PPE
- ☐ First aid kit
- ☐ Camera
- ☐ QAPP
- ☐ SAP
- ☐ HASP

Kick Net

- ☐ Forceps
- ☐ Square aquatic kick-net (900 micron mesh size)
- ☐ Stainless steel sorting bowls or trays
- ☐ Cooler, packing material and ice or dry ice (for tissue chemistry samples)
- ☐ Scale and ruler
- ☐ Waders
- ☐ Personal protection equipment
- ☐ Field notebook and permanent pens/markers
- ☐ Fixing solution (e.g., buffered formalin) for benthic community survey
- ☐ Non-powdered, nitrile-type gloves
- ☐ Deionized water

- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Pre-cleaned and labelled sample bags or containers
- ☐ Aluminum foil
- ☐ First aid kit
- ☐ Camera
- ☐ QAPP
- ☐ SAP
- ☐ HASP

IMPORTANT: Coordinate with the analytical laboratory in advance to determine whether using foil in the plastic bags will cross-contaminate samples analyzed for metals or other target analytes. If so, avoid using foil and simply place invertebrates in the plastic bag and freeze.

Planning and Preparation:

- ☐ Review site-specific information, such as regional land use patterns; site layout and topography; general information on the watershed; water bodies present; water depth; distribution, thickness, and type of sediment; nature of the shoreline; potential onshore and offshore sources of contaminants; ecological habitats and VECs; use of the study area for fishing, shellfish harvest, and boating or recreation; and current and anticipated future use of the water.
- ☐ Conduct site reconnaissance to aid in the preparation of QAPP, SAP, and HASP

IMPORTANT: Prior to initiation of sampling, consider field logistics – identify activities that must be done on the boat or immediately in the field, as opposed to those that could be performed later, on land or at a laboratory or in the office. It is often safest and most efficient to preserve samples in the field and to sort samples in a field trailer or in the laboratory.

- ☐ Prepare QAPP
  - Define and document the data quality objectives, including specific types of Quality Assurance/Quality Control (QA/QC) samples required to meet those data quality objectives.

- Quality assurance procedures, such as those outlined by ICES (2004) should be specified in the QAPP.

- Determine and plan the field quality control samples that will be obtained for the project, including field duplicate samples, field blanks, equipment blanks, and trip blanks.

- Duplicate samples generally should be collected at a rate of 10%, in order to assess sample location variability.

☐ Prepare SAP

- Determine the life histories of the target organisms to verify whether they are active and available for sampling during the target time of year and weather conditions.

- Determine whether federal/provincial/territorial protected species are likely to be present. If so, select a sampling method that can minimize harm to these species.

- For tissue analysis for contaminant analysis, consult with the analytical laboratory regarding minimum mass required for analysis, and if any potential contamination concerns with use of preservatives.

IMPORTANT: Consult with the analytical laboratory concerning requirements related to sample volume, holding times, and preservation. Develop a SAP that specifies practices that will prevent exceeding established holding times. The SAP should specify holding times for individual COPCs, especially for COPCs with short holding times that may dictate some aspects of field logistics. Coordinate with laboratory regarding shipping methods and processing.

- Review the study area features and habitat types and devise a sampling location map that provides representative coverage of habitat types with exposure. Determine the appropriate habitat types to target and the numbers of samples or sweeps per habitat type necessary to meet minimum mass requirements for intended analysis.

- Regardless of method used to collect organisms (e.g., clams, mussels, lobsters, crabs), determine whether or not organisms will be depurated prior to submittal for analysis. If the primary purpose for collecting these organisms is to address risks associated with human consumption, depuration is appropriate, given that most people repeatedly rinse their clams, mussels, lobsters, and crabs prior to cooking them. If the primary purpose is to evaluate ecological risks based on comparisons to literature-derived tissue-based toxicity reference values (TRV), depuration practices should be consistent with the underlying study that provides the basis for the TRV. If the primary purpose is to address risks associated with

wildlife consumption, then samples should not be depurated, given that wildlife typically consume invertebrates immediately upon capture. If depuration is desired, plan for an additional 24 hour period to allow passage of the sediment from the live target organisms before submitting for chemical analyses.

- Coordinate with the analytical laboratory regarding methods for sample collection, processing, and shipping. Discuss the desired preparation method (e.g., depuration) for chemical analysis.

- For taxonomic samples, consult with the taxonomic laboratory regarding the appropriate preservative (e.g., 90% ethanol, 10% formalin, other) for the types of samples to be collected.

IMPORTANT: Biological samples collected for chemical analysis *should not* be chemically preserved, while biological samples collected for taxonomy *should* be chemically preserved. Consult with the taxonomy laboratory in advance to determine the appropriate preservative and container for use with that preservative. Store the two types of biological samples in separate clearly marked containers.

- List equipment, supplies and procedures related to sample labelling, preservation, decontamination, handling, and shipping.

☐ Prepare HASP (including an emergency plan)

- Determine and plan safety requirements specifically associated with sampling requirements based on study area features, COPCs, and personal health and safety precautions (e.g., personal floatation devices and other boat safety requirements, if applicable, chemical resistant clothing and gloves, respiratory protection, ear and eye protection , *etc.*).

- Check proper functioning and integrity of PPE, including respirators and filters.

- If formaldehyde solution will be used, it must be shipped as Dangerous Goods, and treated in a manner consistent with Workplace Hazardous Materials Information System (WHMIS) requirements (e.g., stored in the hazardous chemical storage locker and used under a fume hood, with material safety data sheets provided).

- Hard hats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet.

- File a field plan and boat plan (if appropriate) with a land-based supervisor.

- Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should carry an adequate supply of water or other liquids for protection against dehydration in hot weather.

☐ Reserve, order, and pack all required equipment

- Obtain and prepare the necessary sampling and storage equipment, sample containers, materials, and documents.

- For efficiency and to reduce field decontamination activities, all sampling equipment should be cleaned and decontaminated at the laboratory or field office before going to the sampling site. If possible, a separate set of cleaned and decontaminated equipment should be available for each sampling site.

- If necessary, reserve a boat and make arrangements for qualified boat operator.

- Prepare a field notebook for documenting all activities related to field sampling. Information to be recorded in the field notebook includes, but is not limited to, weather conditions, progress toward accomplishing sampling objectives, deviations from the field sampling plan, sample identification numbers consistent with what is recorded on the laboratory chain-of-custody forms, sample observations, preservatives used and shipping protocol.

☐ Determine whether permits are needed for collecting organisms and obtain any required permits from the appropriate territorial, provincial, and/or federal regulatory agencies

☐ If access to private property will be required, obtain written access permission from landowners

☐ Obtain First Aid/CPR certification and appropriate safety training for all field personnel

Sample Collection:

General Considerations

1) Collecting samples in cold weather, especially around cold water bodies, carries the risk of hypothermia and frostbite. Sampling team members should wear adequate clothing for protection in cold weather.

IMPORTANT: Do not sample for benthic invertebrate tissue during flood stage or drought conditions, in order to avoid atypical hydrological influences on the samples and in order to obtain representative samples for the targeted sampled community types.

2) Hard hats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet.

3) All sample containers should be labelled with the site name as it appears on the laboratory submission form, the date and time of the sample collection and the name of the sample collector or other information specified by the laboratory. Each sample label should include the following information:

    - project name
    - site identification
    - sample number
    - date of sample
    - time of collection
    - preservative used
    - collector's name
    - type of analysis required

4) All biota samples collected for chemical or bioassay analysis should be immediately chilled and stored at 4°C.

5) Sample containers should be placed in clear plastic bags to minimize soiling of the shipping container and to protect laboratory personnel.

6) Glass containers should be protected from breakage. All sediment samples should be chilled and stored in coolers or similar containers at $\leq$ 10°C in transit (but not frozen). Note: sediment and biota samples have different preferred temperatures for transport to laboratory.

7) A description of how the samples were packed in the field, what preservatives were used and how they were shipped to the laboratory should be recorded.

8) All information regarding the sampling event should be accurately recorded in field notes, in order to support the subsequent preparation of a sampling event report. It is important not to discard the sample back in to the water until all sampling is finished, to minimise contaminating subsequent samples.

Organism Traps (may be used for collecting benthic macroinvertebrates for tissue analysis)

1) Based on study area reconnaissance, select locations for trap placement prior to initiating sampling program. Consider habitat requirements for target organisms and place traps accordingly. Traps may be used successfully in both lentic and lotic habitats. In shallow water, areas with surrounding submerged debris (e.g., logs, rocks) or emergent vegetation may be most productive.

2) Deploy traps by attaching rope or twine to the traps and then placing the traps on the sediment surface. To facilitate surface detection, it can be helpful to attach a buoy to each trap using rope or twine, particularly in large bodies of water. Because bait can be a source of chemical exposure, its use is not recommended. If bait is used, a sample of the bait should be analyzed for the same target analytes applied to the tissue samples.

3) Mark the trap locations using stakes, flagging, and/or a GPS unit. Photograph each trap location and record the photograph number in the field notebook.

4) Check traps at least daily. Wear non-powdered nitrile-type gloves and retrieve any trapped crayfish or other invertebrates. Lobster or crab traps may be checked less frequently (i.e., every one to three days).

5) Weigh and sort the organisms to the appropriate taxonomic level in the field and place in pre-cleaned and labelled sample bags or containers. Photograph sample and record the photograph number in the field notebook.

6) Freeze or store biota at 4°C until shipping. Coordinate with the analytical laboratory in advance to determine appropriate holding time limits. Biota for taxonomy should be preserved because freezing may damage tissues and hinder identification.

7) Follow decontamination procedures specific to the equipment and target analytes in both surface water and sediment.

IMPORTANT: Never compromise your personal safety or that of a field partner to collect a sample. Always plan ahead to avoid falling and drowning hazards. Always wear appropriate safety gear, such as personal floatation devices. When working with winches, cables, and similar machinery, gloves, hard hats, safety glasses, and steel-toe boots are also important safety items. A qualified boat operator is required for all travel or sampling from a boat. Boat operations must conform to all requirements in federal and provincial laws.

Sediment Sampler (may be used for collecting benthic invertebrates for either tissue analysis or benthic taxonomy in any habitat, but particularly useful in deeper freshwater or marine habitats where other methods are not feasible. Sediment samplers appropriate for various habitats and conditions are described in Chapter 10 and its associated SOPs)

1) If sediment sampling devices are being used repeatedly, care should be taken to avoid cross-contamination. Sample collection should proceed from areas with lowest COPC concentrations (i.e., reference areas or portions of the study area more distant to source areas) and proceeding to areas with highest COPC concentrations.

2) In order to minimize the potential for suspended sediment in flowing systems, begin sampling at the farthest downstream/downcurrent location and proceed successively upstream/upcurrent.

3) Mark sample locations using stakes, flagging, and/or a GPS unit. Photograph each sample location and record the photograph number in the field notebook.

4) Take precautions to avoid disturbing sediment prior to sampling.

5) A clean pair of powder-free nitrile-type gloves should be worn at each sampling location. The gloves should not contact the sediment sample and should be changed when their cleanliness is compromised.

6) Prepare the sampler for deployment, including for proper decontamination and operation of the sampling device.

7) Lower the sampler to the substrate, ensuring that it settles flat while avoiding excessive sediment disturbance.

8) Activate the device following the manufacture's operating instructions and carefully retrieve it from the water. Verify proper operation of the device as the sampler reaches the surface (keep the sampler as horizontal as possible). If it is determined that the device did not activate properly, the acceptability of the sample must be determined upon sample inspection. If unacceptable, discard the sample, decontaminate the sampling device, and re-deploy the sampling device.

9) Pour or decant excess water out of the sampler. Care should be taken to minimize the loss of fine-grained material.

10) Carefully open the sampling device and transfer the collected sediment with a stainless steel spoon into a sieve bucket. Upon retrieval of the sediment sampling device, the collected sediment may be transferred directly into a sample container and preserved, or it may be released into a sieve bucket, box sieve, or dip net to allow fine sediment to be rinsed from the sample and reduce sample volume (particularly useful for samples being shipped) prior to placement in sample container.

11) If sample is placed in sieve bucket, excess sediment may be removed by rinsing and then spinning the sieve bucket vigorously, until only large debris and organisms remain. If sample is placed in a sieve tray, excess sediment may be removed by rinsing until only large debris and organisms remain. Smaller quantities of sediment may be placed in a dip net and rinsed to allow fine sediment to be removed.

NOTE: In order to collect representative benthic community samples, it is important that experienced field personnel complete the sieving process.

12) If sieve bucket/tray or dip net is used, a combination of gloved hands, clean forceps, and additional rinsing may be used to transfer organisms to pre-cleaned and labelled sample containers.

13) To obtain additional sample volume, continue collecting additional sediment samples from the same location and repeat Steps 6 through 12.

14) Biota for taxonomy should be preserved because freezing may damage tissues and hinder subsequent identification. For marine benthic community sampling, sediment samples may be sieved onboard, transferred to containers and immediately fixed by buffered formalin. Sorting is normally carried out in the lab after the samples are washed and transferred to 70% alcohol for preservation.

15) All other types of samples may be frozen or store at 4^{o}C until shipping.

16) Follow proper procedures related to holding conditions (i.e., store at 4°C), chain-of-custody, and shipping.

17) Following all sampling events, clean and decontaminate sampling devices. In general, a mild, non-phosphate detergent cleaning followed by a deionized water rinse is sufficient. Collect and contain all decontamination washwater for appropriate disposal.

Hand Picking (primarily used for collection of molluscs in tidal flats or shallow water for tissue analysis)

1) Determine sampling locations and times prior to initiating sampling, based on tidal fluctuations and exposure of clam or mussel zones along the shoreline.

2) Using a rake, garden trowel, or shovel, excavate clams or mussels and place in clean bucket. Retrieval of mussels that colonize fixed objects (e.g., boulders, piers, docks) may be collected by hand (or using a stainless steel knife if needed).

3) Document sample retrieval locations on a map, ideally using GPS. Photograph each sample location and record the photograph number in the field notebook.

4) Sort and measure length and weight of individual clams or mussels. Place in pre-cleaned and labelled sample bags or containers. Photograph sample and record the photograph number in the field notebook. Store on ice until shipping.

5) Follow proper procedures related to holding conditions (i.e., at 4°C), chain-of-custody, and shipping.

6) Following all sampling events, clean and decontaminate sampling devices. In general, a mild, non-phosphate detergent cleaning followed by a deionized water rinse is sufficient. Collect and contain all decontamination washwater for appropriate disposal.

Dip Net/Kick Net (may be used for collection of epibenthic organisms for benthic community survey in freshwater areas; may also be used to collect these organisms for tissue analysis; dip nets may also be used for sampling in some shallow estuarine or marine habitats)

1) Use a GPS unit and/or flagging to mark the centre of the sample location. Photograph each sample location and record the photograph number in the field notebook. Include a description of the site, habitat, substrate, embeddedness, percent cover, other habitat factors to be used in community analysis.

2) Use of dip nets or kick nets to collect aquatic macroinvertebrates for tissue analysis is most efficient in productive habitat types, such as leaf packs, snags, aquatic vegetation, roots, and rocky outcrops. Use of Surber samplers and Hess samplers is most effective in riffle areas of freshwater streams. Habitats in areas of water velocity greater than 0.2 metres per second should be perferred over those with slower water velocities, as macroinvertebrates are easier to capture in the net because they drift downstream when dislodged.

3) In order to minimize the potential for suspended sediment in flowing systems, begin sampling at the farthest downstream location and proceed successively upstream. Stand facing the direction of flow and approach the location from the downstream direction, while pulling the net behind you (i.e., the net must be downstream of the kicking, so that organisms dislodged by the kicking are caught in the net).

4) If using a dip net, conduct several (three or more) passes in each 0.5-m sweep location with the D-frame dip net in the most productive habitats. Dislodge material by kicking, probing, or sweeping the sediment and catch organisms by allowing them to flow into the net and also by sweeping the net towards disturbed material. If using a kick net, dislodge material using feet while

holding the net directly behind to catch the drift material. Begin downstream and walk or kick the net forward in productive habitat types to capture invertebrates. The CABIN protocol for benthic community analysis specifies 3-minute kick samples in a zig zag pattern; regardless of whether CABIN or another protocol is used, it is imperative that the level of effort is consistent at each locations, so that community results can be compared across sample locations.

5) A clean pair of powder-free nitrile-type gloves should be worn before handling macroinvertebrate samples from each location. The gloves should be changed when their cleanliness is compromised.

6) Remove organisms from the net between each sweep location. Place samples in pre-cleaned and labelled sample containers and place on wet ice for return to the processing area.

7) Label each sample container with a project identification or project code, the date, time of sampling, sample identification number, target analytes, and initials of the samplers.

8) At the processing area, drain and weigh samples. Photograph sample and record the photograph number in the field notebook. Freeze or store sample at 4°C. Biota for taxonomy should be preserved because freezing may damage tissues and hinder identification.

9) Samples from areas with highest COPC concentrations should be stored separately from those likely to contain trace concentrations. Reference area samples should be stored in separate coolers and shipping containers.

10) Follow proper procedures related to holding conditions (i.e., at 4°C), chain-of-custody, and shipping.

11) Following all sampling events, clean and decontaminate sampling devices. In general, a mild, non-phosphate detergent cleaning followed by a deionized water rinse is sufficient. Collect and contain all decontamination washwater for appropriate disposal.

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SUGGESTED OPERATING PROCEDURE NUMBER 16: FISH SAMPLING

SCOPE This suggested operating procedure (SOP) describes various recommended methods for collecting fish samples from various types of waterbodies (e.g., freshwater, marine, lentic, lotic, wadeable, deep). Additional information on fish tissue collection is provided in Chapter 11 of Volume 1of the *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as "*Guidance Manual"*). For more information refer to Section 8 of *Protocols Manual for Water Quality Sampling in Canada* (http://www.ccme.ca/files/Resources/water/protocols_document_e_final_101.pdf).

APPLICATION This SOP describes the handling procedures, methodology, and equipment to effectively sample fish to support human health and ecological risk assessments.

WHEN? Fish samples are typically collected for the following purposes:

- □ To characterize bioaccumulation of chemicals of potential concern (COPCs) in aquatic media for human health and ecological risk assessment;
- □ To model bioaccumulation in those valued ecological components (VECs) that eat fish (i.e., piscivorous fish, birds, and mammals);
- □ To characterize potential human health risks from consumption of recreationally-caught, commercially-caught, or subsistence-caught fish; and/or
- □ To complete taxonomic analysis of fish and characterize overall condition of the fish community in the study area.

WHY? Accurate characterization of fish tissue concentrations—representing the species, size, and preparation methods targeted by human and ecological receptors—is critical to accurate estimation of risks from consumption of fish. Risks to fish themselves, also can be evaluated by comparing fish tissue concentrations to literature-derived thresholds that are protective of fish and by evaluating the community composition, sex ratios, and condition of fish.

HOW? Fish samples can be collected using a variety of sampling methods. The choice of sampling method primarily depends on: study objectives; size of the targeted fish; habitat use preferences of target fish species (e.g., bottom feeders *vs*. water column feeders); and habitat characteristics (e.g., lakes, fast flowing rivers, small ponds, estuaries, open ocean).

TYPES ***Fyke Net Sampling***: A preferred technique in shallow water (i.e., less than 1 metre (m) deep, fyke nets create a funnel in shallow water so that fish swim into the net, but are unable to escape.

Seining: This technique is preferred for fish tissue collection for chemical analysis where the water body lacks physical barriers or substrates that would interfere with the use of seine. This technique is also preferred when targeting small or young fish that could be injured with electrofishing.

Electrofishing: This method is preferred for fish community analysis (e.g., non-retention inventory) and chemical analysis of tissue, particularly where safety concerns or physical barriers prevent seining. Electrofishing can pose an electrical safety hazard if water conductivity is high (e.g., marine waters), and some internal damage and/or mortality to target and non-target fish species may occur.

Rod and Reel: This method is preferred for human health risk assessments where the objective is to target fish that reflect exposures to people from consumption of recreationally-caught sportfish. Rod and reel sampling can be particularly valuable in fast-flowing rivers, where other methods may be problematic. It is also a good method when targeting a particular species or size range, since catch-and-release survival is high. However, this method is very labour intensive and may not result in adequate numbers of samples.

Minnow Traps: Commercially available minnow trap models are cone or cylinder shapes constructed of wire or mesh. The traps have built in funnel entrances to allow small fish to enter, but not exit. Minnow traps are also easily constructed using plans available from a variety of sources and materials that are generally available at any hardware store. Traps are deployed on the sediment surface and retrieved at least once daily. Because bait can be a source of chemical exposure, its use is not recommended. If bait is used, a sample of the bait should be analyzed for the same COPCs applied to the tissue samples.

Gill Netting: Gill nets are commonly used in lakes, slow-moving portions of rivers, and in marine environments. These have monofilament mesh panels, which may have various opening dimensions that entrap fish by their opercula. This is a highly effective method for fish collection, but is relatively non-specific and results in a high mortality of trapped fish. Nets can be set as surface, midwater, or bottom sets by varying the weights of float-line to lead-line (top and bottom of panels).

Additional information on fish tissue sampling is provided in U.S. Environmental Protection Agency's *Operating Procedure for Field Fish Sampling* (USEPA 2011), as well as the American Fisheries Society's *Fisheries Techniques* (AFS, 1996), and British Columbia Resources Information Standards Committee's *Fish Collection Methods and Standards* (RISC, 1997). For information on euthanizing fish, consult the *Canadian Council on Animal Care guidelines on: the care and use of fish in research, teaching and testing* (CCAC, 2005) and Department of Fisheries and Oceans training material.
http://www.ccac.ca/Documents/Education/DFO/3_Euthanasia_of_Finfish.pdf.

Essential Information

- ☐ During site reconnaissance, determine water depth and substrate type, so that the most appropriate fish sampling method can be selected.
- ☐ If electrofishing, be familiar with the specific safety requirements to avoid unnecessary harm to fish and to protect sampling personnel. Discuss electrofishing hazards and precautions in the Health and Safety Plan (HASP).
- ☐ Upon collecting individual fish, record the length, weight, and any other pertinent observations.

COLLECTION Prior to initiating the sampling program, it is important to document all methods to be used in a Sampling and Analysis Plan (SAP). For example, the minimum sample mass and methods for processing and shipping fish samples (based on the COPCs) are defined in the SAP. If fillet samples are desired (e.g., for use in human health risk assessments), it is important to coordinate with the analytical laboratory prior to sampling to determine whether fish will be filleted in the field or laboratory. In addition, lipid content analysis may be needed to normalize tissue concentrations of lipophilic chemicals. Therefore, arrangements should be made in advance with the laboratory if this analysis is required.

SAMPLING CHECKLIST

Equipment Checklist:

Fyke Nets

- ☐ Collection permit
- ☐ Field logbooks
- ☐ Fyke net
- ☐ Polyvinyl chloride (PVC) poles or crowbars (minimum 1 m height, 4 for each net)
- ☐ Buoys
- ☐ Rope
- ☐ Chest waders
- ☐ Personal floatation devices for all personnel
- ☐ Buckets/live wells

- ☐ Chemical preservative specified by the laboratory (for preservation of voucher samples requiring later taxonomic analysis)
- ☐ Pre-cleaned and labelled sample bags or containers
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Aluminum foil
- ☐ Cooler, packing material and ice or dry ice (for tissue chemistry samples)
- ☐ Non-powdered, nitrile-type gloves
- ☐ Scale, fish board, rulers
- ☐ Fish identification guide or field key
- ☐ Global Positioning System (GPS)
- ☐ Bathymetric map (detailed description of submerged terrain) or nautical charts (primarily for safe navigation) if applicable
- ☐ Clipboards, pens, sharpies, *etc*.
- ☐ First aid kit
- ☐ Personal protection equipment (PPE)
- ☐ Camera
- ☐ Quality Assurance Program Plan (QAPP)
- ☐ Sampling and Analysis Plan (SAP)
- ☐ Health and Safety Plan (HASP)

Seining

- ☐ Collection permit
- ☐ Field logbooks
- ☐ Seine with lead line and float line
- ☐ Poles (two for each seine) for pole seining
- ☐ Boat, if needed for beach or deepwater seining, and qualified boat operator

- ☐ Chest waders
- ☐ Personal floatation devices
- ☐ Buckets/live wells
- ☐ Fish identification guide or field key
- ☐ Chemical preservative specified by the laboratory (for preservation of voucher samples requiring later taxonomic analysis)
- ☐ Pre-cleaned and labelled sample bags or containers
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Aluminum foil
- ☐ Cooler, packing material and ice or dry ice (for tissue chemistry samples)
- ☐ Non-powdered, nitrile-type gloves
- ☐ Scale, fish board, rulers
- ☐ GPS
- ☐ Bathymetric map (detailed description of submerged terrain) or nautical charts (primarily for safe navigation) if applicable
- ☐ Clipboards, pens, sharpies, *etc*.
- ☐ First aid kit
- ☐ PPE
- ☐ Camera
- ☐ QAPP
- ☐ SAP
- ☐ HASP

Electrofishing

- ☐ Collection permit
- ☐ Field logbooks

- ☐ Backpack shocker or boat-mounted electroshocker
- ☐ Boat, if needed, and qualified boat operator
- ☐ Dip nets, block nets/seine
- ☐ Insulated rubber gloves
- ☐ Chest waders
- ☐ Personal floatation devices
- ☐ Buckets/live wells
- ☐ Fish identification guide or field key
- ☐ Chemical preservative specified by the laboratory (for preservation of voucher samples requiring later taxonomic analysis)
- ☐ Pre-cleaned and labelled sample bags or containers
- ☐ Aluminum foil
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Cooler, packing material and ice or dry ice (for tissue chemistry samples)
- ☐ Non-powdered, nitrile-type gloves
- ☐ Scale, fish board, rulers
- ☐ GPS
- ☐ Bathymetric map (detailed description of submerged terrain) or nautical charts (primarily for safe navigation) if applicable
- ☐ Clipboards, pens, sharpies, *etc*.
- ☐ First aid kit
- ☐ PPE
- ☐ Camera
- ☐ QAPP
- ☐ SAP

- ☐ HASP

Rod and Reel Collection

- ☐ Boat, if needed, and qualified boat operator
- ☐ Bathymetric map (detailed description of submerged terrain) or nautical charts (primarily for safe navigation) if applicable
- ☐ Fishing pole(s)
- ☐ Lures or bait for target species
- ☐ Fishing line that is of the appropriate strength/size
- ☐ Pliers
- ☐ Scale, fish board, rulers
- ☐ Buckets/live wells
- ☐ Chemical preservative specified by the laboratory (for preservation of voucher samples requiring later taxonomic analysis)
- ☐ Pre-cleaned and labelled sample bags or containers
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Aluminum foil
- ☐ Cooler, packing material and ice or dry ice (for tissue chemistry samples)
- ☐ Non-powdered, nitrile-type gloves
- ☐ Fish identification guide or field key
- ☐ First aid kit
- ☐ PPE
- ☐ Camera
- ☐ QAPP
- ☐ SAP
- ☐ HASP

Minnow Traps

- ☐ Minnow traps
- ☐ Buoy and rope for surface marker
- ☐ Stakes, flagging, or GPS unit
- ☐ Bathymetric map (detailed description of submerged terrain) or nautical charts (primarily for safe navigation) if applicable
- ☐ Buckets/live wells
- ☐ Bait if being used
- ☐ Boat, if needed, and qualified boat operator
- ☐ Chemical preservative specified by the laboratory (for preservation of voucher specimen requiring later taxonomic analysis)
- ☐ Scale and ruler
- ☐ Deionized water
- ☐ Pre-cleaned and labelled sample bags or containers
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Aluminum foil
- ☐ Cooler, packing material and ice or dry ice (for tissue chemistry samples)
- ☐ Non-powdered, nitrile-type gloves
- ☐ Field notebook and permanent pens/markers
- ☐ Fish identification guide or field key
- ☐ Waders
- ☐ First aid kit
- ☐ PPE
- ☐ Camera
- ☐ QAPP

- ☐ SAP
- ☐ HASP

IMPORTANT: Coordinate with the analytical laboratory in advance to determine whether use of foil inside the plastic bags risks cross-contaminating samples that will be analyzed for metals or other target analytes. If so, avoid using foil and place fish in the plastic bag and freeze.

IMPORTANT: Consult with the analytical laboratory concerning requirements related to sample volume, holding times, and preservation. Develop an SAP that specifies practices that will prevent exceeding established holding times. The SAP should specify holding times for individual COPCs, especially for COPCs with short holding times that may dictate some aspects of field logistics. Coordinate with laboratory regarding shipping methods and processing.

Planning and Preparation:

- ☐ Review site-specific information, such as regional land use patterns; site layout and topography; general information on the watershed; water bodies present; water depth; distribution, thickness, and type of sediment; nature of the shoreline; potential onshore and offshore sources of contaminants; ecological habitats and VECs; use of the study area for fishing, shellfish harvest, and boating or recreation; and current and anticipated future use of the water.
- ☐ If considering use of electrofishing, determine the water's conductivity as part of the study area reconnaissance, in order to verify that it is within the range of recommended conditions by the equipment manufacturer.
- ☐ As part of the reconnaissance, visit multiple candidate reference areas, in order to select those that most closely match conditions at the study area (but for the release of COPCs), and are safe and accessible. *In situ* water quality characterization (pH, dissolved oxygen [DO], conductivity, temperature) can also aid in identifying comparable reference areas.
- ☐ Prepare QAPP
  - Define and document the data quality objectives, including specific types of Quality Assurance/Quality Control (QA/QC) samples required to meet those data quality objectives. Duplicate samples generally should be collected at a rate of 10%, in order to assess sample location variability.
- ☐ Prepare SAP

- Review existing study area information to determine the species of fish likely to be present and those that are consistent with the project data quality objectives.

- Determine whether federal/provincial/territorial protected species are likely to be present. If so, select a sampling method that can minimize harm to these species.

- Coordinate with the analytical laboratory regarding methods for sample collection, processing, and shipping. Discuss the desired preparation method (i.e., whole body or fillet) for chemical analysis and/or aging. Include this information within the SAP.

- When preparing the SAP, note that while more fish are caught the longer nets/traps are deployed, longer deployment times will also increase the risk of mortality to target and non-target species, predation and possible escape.

- The SAP should specify water quality sampling and measurements that are required as part of the sampling program. [26]

- List equipment, supplies, and procedures related to sample labelling, preservation, decontamination, handling, and shipping.

☐ Prepare HASP (including an emergency plan)

- Determine and plan safety requirements specifically associated with sampling requirements based on study area features, COPCs, and personal health and safety precautions (e.g., personal floatation devices and other boat safety requirements, if applicable, chemical resistant clothing and gloves, respiratory protection, ear and eye protection , *etc.*).

- Check proper functioning and integrity of PPE, including respirators and filters.

- If a boat will be used for sampling, the HASP should include boating-specific safety requirements.

- If formaldehyde solution will be used, it must be shipped as Dangerous Goods, and treated in a manner consistent with Workplace Hazardous Materials Information System (WHMIS) requirements (e.g., stored in the hazardous chemical storage locker and used under a fume hood, with material safety data sheets provided).

[26] pH, conductivity, dissolved oxygen, temperature, turbidity, flow measurements, etc.

- Hard hats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet.

- File a field plan and a boat plan (if appropriate) with a land-based supervisor.

- Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should carry an adequate supply of water or other liquids for protection against dehydration in hot weather.

☐ Reserve, order, and pack all required equipment

- Reserve a boat and make arrangements for qualified boat operator.

- Obtain and prepare the necessary sampling and storage equipment, sample containers, materials, and documents.

- For efficiency and to reduce field decontamination activities, all sampling equipment should be cleaned and decontaminated at the laboratory or field office before going to the sampling site. If possible, a separate set of cleaned and decontaminated equipment should be available for each sampling site.

- Prepare a field notebook for documenting all activities related to field sampling. Information to be recorded in the field notebook includes, but is not limited to, weather conditions, progress toward accomplishing sampling objectives, deviations from the field sampling plan, sample identification numbers consistent with what is recorded on the laboratory chain-of-custody forms, sample observations, preservatives used and shipping protocol.

☐ Determine whether permits are needed and obtain any required permits from the appropriate provincial, territorial and/or federal regulatory agencies

☐ If access to private property will be required, obtain written access permission from landowners

☐ Obtain First Aid/CPR certification and appropriate safety training for all field personnel

Sample Collection:

1) Proper safety precautions must be observed when collecting samples. All field staff must be familiar with the project HASP. Crew members should have current First Aid/CPR certification.

2) If sampling involves the use of a boat, the weather forecast or marine conditions should be obtained prior to departure. If weather conditions are poor, the sampling program should be postponed.

3) Complete, accurate, and legible record-keeping in the field is a critical component of field sampling activities. Throughout the sampling event, document all activities, conditions, deviations from the SAP in field notes and through photographs. Field notes should also document how samples were packed in the field, what preservatives were used and how samples were shipped to the laboratory.

4) A clean pair of new, non-powdered, disposable gloves will be worn each time different location is sampled.

5) Water quality measurements should be collected and recorded at each fish collection station prior to sampling.

6) Sample containers should be labelled with the site name as it appears on the laboratory submission form, the date and time of the sample collection and the name of the sample collector or other information specified by the laboratory. Each sample label should include the following information:

    - project name
    - site identification
    - sample number
    - date of sample
    - time of collection
    - preservative used (chemical, dry ice or frozen)
    - collector's name
    - type of analysis required

7) Fish samples collected for chemical or bioassay analysis should be immediately chilled and stored in coolers or similar containers at 4°C.

8) Sample containers should be placed in clear plastic bags to minimize soiling of the shipping container and to protect laboratory personnel.

9) Glass containers should be protected from breakage.

10) All information regarding the sampling event should be accurately recorded in field notes, in order to support the subsequent preparation of a sampling event report.

Methods of Fish Collection

Fyke Nets

1) Unless a specific component of the fish community is targeted, select sampling locations with a variety of habitats, in order to assess the broadest possible extent of the fish community.

2) Establish sampling locations with comparable habitats (e.g., riffle/run/pool prevalence, in-stream cover, substrate, vegetative cover, depth, *etc*.) to ensure comparability of data.

3) Use a GPS unit and/or flagging to mark the centre of the sample location. Photograph each sample location and record the photograph number in the field notebook.

4) Depending on water depth, select the appropriate net size. Large nets (~1.0 m x 1.5 m opening) should be used in water greater than 0.75 m deep. Small nets (~0.5 m x 1.0 m opening) should be used in water less than 0.75 m deep.

5) Place nets with the opening facing the shore/vegetation and the funnel perpendicular to the current/flow or shore/vegetation.

6) Set wings at a 45^{o} angle to the net opening.

7) Once the net is set, confirm that funnels are under water.

8) Use rope to attach the buoy to make the net more visible to boaters.

9) Leave net in place for 24 to 48 hours before collection. If the net is set in a tidal area, it must be checked before it is exposed by a receding tide. Record the start and retrieval time of the net, noting flow/tides during the set and note general condition of the net on retrieval, i.e., debris in net, tears, *etc.*

10) Collect fish by starting at the open end and simultaneously holding the net up while shaking fish down. Successive hoops should then be lifted while keeping the opening of the net out of the water. This action will move fish down to the end of net and keep fish from escaping.

11) Hold all fish in buckets or tubs of study area water, using aeration if extended holding is necessary or if large numbers of fish are collected.

12) All fish, excluding larvae, should be collected, enumerated, and identified (to the species level if possible) using standard taxonomic keys specific to the region sampled (if available). Record total length and wet weight, and enumerate and photograph any external lesions, anomalies, and parasites.

13) If the study requires all fish to be identified to species level, specimens that cannot be so identified in the field may be preserved in a chemical preservative specified by the laboratory and stored in labelled containers for subsequent laboratory identification.

14) Determine individual fish to be incorporated into each sample. Photograph sample and record the photograph number in the field notebook. Wrap fish in aluminum foil and place in pre-cleaned and labelled sample container.

15) Label all sample containers with respect to sampling location, date, time collectors' initials, and sample identification code and/or station numbers.

16) Return any remaining live fish (i.e., any fish not being submitted for chemical analysis or taxonomic identification) to the water from which they were collected.

17) If necessitated by project goals, maintain a representative voucher collection (i.e., one specimen of each species collected), excluding threatened and endangered species or other species of special concern. Preserve in a chemical preservative specified by the laboratory and store in labelled containers.

18) Freeze or store samples at 4°C until shipping. Coordinate with the analytical laboratory in advance to determine appropriate holding time limits. Biota for taxonomy should be preserved because freezing may damage tissues and hinder identification.

Pole Seining

1) Sampling should be conducted at normal seasonal flows with high water clarity.

2) Unless a specific component of the fish community is targeted, select sampling locations with a variety of habitats, in order to assess the broadest possible extent of the fish community.

3) Establish sampling stations with comparable habitats (e.g., riffle/run/pool prevalence, in-stream cover, substrate, depth, *etc*.) to ensure comparability of fish community data.

4) Use a GPS unit and/or flagging to mark the centre of the sample location. Photograph each sample location and record the photograph number in the field notebook.

5) Place the net in the water, perpendicular to the flow or current (if any), with the float line at the surface, the lead line at the stream substrate, and a pole at each end. The pole lengths should be at least equal to the heights of the net

and should be at a 45° angle away from the direction of movement when pulling the seine.

6) One person holds each pole on either side of the net. Pull the seine against the current, keeping the poles directly along the bank, and under it, if the bank is undercut. The lead line must remain in contact with the bottom to prevent fish from escaping under the net, and the float line must stay on or above the water surface.

7) Following collection, both seiners walk onshore and immediately pull up the lead line. If there is no convenient place to beach the seine, the lead line can be lifted above water by both collectors at the same time. After the net is out of the water, captured fish should be immediately transferred to water-filled containers.

8) Record the level of effort at each sampling station, striving to implement similar effort at all sampling stations.

9) Hold all fish in buckets or tubs of study area water, using aeration if extended holding is necessary or large numbers of fish are collected.

10) All fish, excluding larvae, should be collected, enumerated, and identified (to the species level if possible) using standard taxonomic keys specific to the region sampled (if available). Record total length and wet weight, and photograph and enumerate any external lesions, anomalies, and parasites.

11) If the study requires all fish to be identified to species level, specimens that cannot be so identified in the field may be preserved in a chemical preservative specified by the laboratory and stored in labelled containers for subsequent laboratory identification.

12) Determine individual fish to be incorporated into each sample. Photograph sample and record photograph number in the field notebook. Wrap fish in aluminum foil and place in pre-cleaned and labelled sample container.

13) Label all sample containers with respect to sampling location, date, time collectors' initials, and sample identification code and/or station numbers.

14) Return any remaining live fish (i.e., any fish not being submitted for chemical analysis or taxonomic identification) to the water from which they were collected.

15) If necessitated by project goals, maintain a representative voucher collection (i.e., one specimen of each species collected), excluding threatened and endangered species or other species of special concern. Preserve in a chemical preservative specified by the laboratory and store in labelled containers.

16) Freeze or store samples at 4°C until shipping. Coordinate with the analytical laboratory in advance to determine appropriate holding time limits. Biota for taxonomy should be preserved because freezing may damage tissues and hinder identification.

Electrofishing

1) Sampling should be conducted at normal seasonal flows at times of high water clarity.

2) Unless a specific component of the fish community is targeted, select sampling locations with a variety of habitats, in order to assess the broadest possible extent of the fish community.

3) Establish sample sites with comparable habitats (e.g., riffle/run/pool prevalence, in-stream cover, substrate, depth, *etc*.) to ensure comparability of the fish community data.

4) Document sampling locations with GPS and topographic maps. Photograph setting and record photograph number(s) in field notebook.

5) Place block-nets in wadeable streams, and collect fish with a direct-current backpack electroshocker beginning at a shallow riffle. Progress from downstream to upstream, sampling all major habitat types.

IMPORTANT: Do not use electrofishing if the conductivity of the water body is outside of the tolerable range of conditions specified by the manufacturer.

6) Record the level of effort at each sampling station, striving to implement similar effort at all sampling stations.

7) Hold all fish in buckets or tubs of study area water, using aeration if extended holding is necessary or large numbers of fish are collected.

8) All fish, excluding larvae, should be collected, enumerated, and identified to the species level (if possible) using standard taxonomic keys specific to the region sampled (if available). Record total length and wet weight, and photograph and enumerate any external lesions, anomalies, and parasites.

9) If the study requires all fish to be identified to species level, specimens that cannot be so identified in the field may be preserved in a chemical preservative specified by the laboratory and stored in labelled containers for subsequent laboratory identification.

10) Determine individual fish to be incorporated into each sample. Photograph sample and record photograph number in field notebook. Wrap fish in aluminum foil and place in pre-cleaned and labelled sample container.

11) Label all sample containers with respect to sampling location, date, time collectors' initials, and sample identification code and/or station numbers.

12) Return any remaining live fish (i.e., any fish not being submitted for chemical analysis or taxonomic identification) to the water from which they were collected.

13) If necessitated by project goals, maintain a representative voucher collection (i.e., one specimen of each species collected), excluding threatened and endangered species or other species of special concern. Preserve in a chemical preservative specified by the laboratory and store in labelled containers.

14) Freeze or store samples at 4°C until shipping. Coordinate with the analytical laboratory in advance to determine appropriate holding time limits. Biota for taxonomy should be preserved because freezing may damage tissues and hinder identification.

Rod and Reel

1) Select appropriate habitats and focus angling efforts on a representative sample of habitats, moving from upstream to downstream in flowing habitats.

2) Document sampling locations with GPS and topographic maps. Photograph setting and record photograph number(s) in field notebook.

3) Unless every fish collected is to be submitted for analysis, hold all fish in buckets or tubs of study area water, using aeration if extended holding is necessary or large numbers of fish are collected.

4) All fish should be collected, enumerated, and identified to the species level (if possible) using standard taxonomic keys specific to the region sampled (if available). Record total length and wet weight, and photograph and enumerate any external lesions, anomalies, and parasites.

5) If the study requires all fish to be identified to species level, specimens that cannot be so identified in the field may be preserved in a chemical preservative specified by the laboratory and stored in labelled containers for subsequent laboratory identification.

6) Determine individual fish to be incorporated into each sample. Photograph sample and record photograph number in field notebook. Wrap fish in aluminum foil and place in pre-cleaned and labelled sample container.

7) Label all sample containers with respect to sampling location, date, time collectors' initials, and sample identification code and/or station numbers.

8) Return any remaining live fish (i.e., any fish not being submitted for chemical analysis or taxonomic identification) to the water from which they were collected.

9) If necessitated by project goals, maintain a representative voucher collection (i.e., one specimen of each species collected), excluding threatened and endangered species or other species of special concern. Preserve in a chemical preservative specified by the laboratory and store in labelled containers.

10) Freeze or store samples at 4°C until shipping. Coordinate with the analytical laboratory in advance to determine appropriate holding time limits. Biota for taxonomy should be preserved because freezing may damage tissues and hinder identification.

Minnow Traps

1) Based on site reconnaissance, select locations for minnow trap placement prior to initiating sampling program.

2) Deploy minnow traps by attaching rope or twine to the traps and then placing the traps on the sediment surface in wadeable streams and other shallow water bodies. To facilitate surface detection, it can be helpful to attach a buoy to each trap using rope or twine. Because bait can be a source of chemical exposure, its use is not recommended. If bait is used, a sample of the bait should be analyzed for the same target analytes applied to the tissue samples.

3) Mark traps locations using stakes, flagging, and/or a GPS unit. Photograph setting and record photograph number(s) in field notebook.

4) Check traps at least daily. Record set times to determine catch per unit effort.

5) Hold all fish in buckets or tubs of study area water, using aeration if extended holding is necessary or large numbers of fish are collected.

6) All fish, excluding larvae, should be collected, enumerated, and identified to the species level (if possible) using standard taxonomic keys specific to the region sampled (if available). Record total length and wet weight, and photograph and enumerate any external lesions, anomalies, and parasites.

7) If the study requires all fish to be identified to species level, specimens that cannot be so identified in the field may be preserved in a chemical preservative specified by the laboratory and stored in labelled containers for subsequent laboratory identification.

8) Determine individual fish to be incorporated into each sample. Photograph sample and record photograph number in field notebook. Wrap fish in aluminum foil and place in pre-cleaned and labelled sample container.

9) Label all sample containers with respect to sampling location, date, time collectors' initials, and sample identification code and/or station numbers.

10) Return any remaining live fish (i.e., any fish not being submitted for chemical analysis or taxonomic identification) to the water from which they were collected.

11) If necessitated by project goals, maintain a representative voucher collection (i.e., one specimen of each species collected), excluding threatened and endangered species or other species of special concern. Preserve in a chemical preservative specified by the laboratory and store in labelled containers.

12) Freeze or store samples at 4°C until shipping. Coordinate with the analytical laboratory in advance to determine appropriate holding time limits. Biota for taxonomy should be preserved because freezing may damage tissues and hinder identification.

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SUGGESTED OPERATING PROCEDURE NUMBER 17: SMALL MAMMAL SAMPLING

SCOPE This suggested operating procedure (SOP) recommends methods for collecting and processing small mammals in the field to ensure safety and quality control in field operations and uniformity among technicians. Additional information on biological tissue sampling is provided in Chapter 11 of Volume 1 of the *Guidance Manual for Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment* (referred to as "*Guidance Manual"*). Additional information on sampling procedures for the collection of small mammals is available from the United States Department of Health and Human Services (USDHHS, 1995).

APPLICATION This SOP describes methods for collecting and processing small mammals in the field, in support of ecological risk assessments.

WHEN? Small mammal samples are usually collected to characterize chemical concentrations in herbivorous and carnivorous feeding guilds for ecological risk assessment when bioaccumulative chemicals of potential concern (COPCs) are present at a study area.

WHY? Small mammal sampling supports development of tissue chemistry data that can be used to characterize ecological risks to small mammals, as well as to hawks, owls, fox, weasels, or other valued ecosystem components (VECs) that consume small mammals. Small mammal sampling can also be conducted to evaluate age structure, sex ratio, and/or composition of the small mammal community.

HOW? Small mammal samples can be collected using a variety of sampling methods. The choice of sampling method depends primarily on: the exposure pathways, diets, feeding guilds, and trophic levels of the VECs; the habitats present in the study area; and the target analytes.

TYPES ***Snap Traps***: Snap traps are often used when destructive (i.e., lethal) sampling is planned. Snap traps are common mouse or rat traps. Because snap traps are lethal, their use is not recommended when there are provincial or federally protected species known to occur in the study area, unless specific permit conditions allow their use.

Live Traps: Live traps are preferred for non-destructive sampling, such as when federal/provincial/territorial protected species are known to occur in the study area or when conducting a mark-recapture community survey. The two most commonly used live traps, Sherman traps and Havahart traps, have pressure-sensitive treble devices that close when the small mammal enters the trap. Other live traps may contain a one-way entrance that allows the animal to enter the trap but not escape. There is potential for animal injury or death if the small mammal gets caught in the door as the snap mechanism is deployed. Therefore, live traps should only be used by individuals trained in handling live small mammals,

including humane methods of euthanasia, which may be required in the event of injury to the trapped specimen. In general, cervical dislocation is considered an acceptable means of euthanasia, provided it is conducted by trained individuals.

COLLECTION An important consideration in small mammal collecting and processing is the manner in which the small mammals will be handled, with the goal of providing appropriate safety protections for field sampling personnel. Personnel performing rodent trapping and specimen collection should be made aware of the risks associated with these tasks and the precautions to minimize these risks.

Essential Information: Minimizing Your Risk

Some small mammals may carry Hantavirus or other diseases that are transmittable to humans. Therefore, special precautions should be taken when handling small mammals. Infected rodents shed viruses such as Hantavirus through urine, droppings, and saliva. Viruses can be transmitted to humans through a process called aeroionisation, which occurs when dried materials contaminated by rodent excreta or saliva are disturbed. Humans can become infected by inhaling infectious aerosols or by touching infected rodent excreta or nesting materials and then touching their eyes, nose, or mouth. Some diseases may be transmitted from a mouse or rat bite. Additional details on symptoms and treatment of Hantavirus are presented in the text box below. Planning small mammal sampling programs should include consideration of health-protective measures, such as use of air-purifying respirators. Additional information on safety precautions for handling small mammals are described in the standard reference guide produced by The Wildlife Society titled *Techniques for Wildlife Investigations and Management* (The Wildlife Society, 2005).

SAMPLING CHECKLIST

Equipment Checklist:

- ☐ Traps (snap, live, or Sherman)
- ☐ Bait (e.g., peanut butter, rolled oats)
- ☐ Flagging tape
- ☐ Aluminum foil
- ☐ Zip-top plastic bags
- ☐ Mammal identification guide
- ☐ Scale and ruler
- ☐ Cooler, packing material and ice or dry ice (for tissue chemistry samples)

- ☐ Wet and/or dry ice
- ☐ Non-powdered nitrile-type gloves
- ☐ Heavy leather gloves
- ☐ Safety goggles
- ☐ Air-purifying respirator
- ☐ N-100 or P-100 filter cartridges for the appropriate respirator make and model
- ☐ Table cover
- ☐ Soap for washing hands
- ☐ 17-litre bucket
- ☐ Liquid disinfectant
- ☐ Scrub brush (for instruments)
- ☐ Biohazard bags
- ☐ Decontamination equipment, including buckets, brushes, surfactant detergent, and/or organic solvents and appropriate containment for decontamination washwater
- ☐ Heavy tape
- ☐ First aid kit
- ☐ Paper towels
- ☐ Camera
- ☐ Personal protection equipment (PPE)
- ☐ Quality Assurance Program Plan (QAPP)
- ☐ Sampling and Analysis Plan (SAP)
- ☐ Health and Safety Plan (HASP)

Traps and Target Species	
Snap trap – mouse	Mice, voles, shrews
Snap trap – rat	Rat, chipmunk, red squirrel, ground squirrel
Sherman/Havahart	Several sizes available based on target species; check with manufacturer

Planning and Preparation:

- ☐ Review site-specific information, such as regional land use patterns; site layout and topography; general information on the watershed; water bodies present; water depth; distribution, thickness, and type of sediment; nature of the shoreline; potential onshore and offshore sources of chemicals; ecological habitats and VECs; use of the study area for fishing, shellfish harvest, and boating or recreation; and current and anticipated future use of the water.

- ☐ Conduct site reconnaissance visit to aid in the preparation of the QAPP, SAP, and HASP

- ☐ Prepare QAPP

  - Define and document the data quality objectives, including specific types of Quality Assurance/Quality Control (QA/QC) samples required to meet those data quality objectives. Duplicate samples generally should be collected at a rate of 10%, in order to assess sample location variability.

- ☐ Prepare SAP

  - Consult Guidelines on the Care and Use of Wildlife (Canadian Council on Animal Care, 2003) for key information related to planning, permit requirements, sampling methods, handling, holding, and euthanasia.

  - Determine whether federal/provincial/territorial protected species are likely to be present. If so, select a sampling method that can minimize capture and harm to these species.

IMPORTANT: Prior to sampling, determine whether provincially or federally protected species may be present. If so, determine appropriate procedures to avoid these species or appropriate measures to release them unharmed and report incidental captures if required by provincial, territorial, or federal permits.

- Review the study area features and habitat types and devise a sampling design and trap placement plan that provides representative coverage of habitat types with exposure, in order to achieve sampling objectives, and develop strategy for trap placement.

- Consult with analytical laboratory to determine the minimum mass per sample or composite sample required for chemical analysis.

- Coordinate with the analytical laboratory regarding methods for sample collection, processing, and shipping. Discuss the desired preparation method (e.g., whole body, specific organs) for chemical analysis.

- List equipment, supplies and procedures related to sample labelling, preservation, decontamination, handling, and shipping.

- Include approved animal care and use protocols.

IMPORTANT: Consult with the analytical laboratory concerning requirements related to sample volume, holding times, and preservation. Develop a SAP that specifies practices that will prevent exceeding established holding times. The SAP should specify holding times for individual COPCs, especially for COPCs with short holding times that may dictate some aspects of field logistics. Coordinate with laboratory regarding shipping methods and processing.

☐ Prepare HASP (including an emergency plan)

- Determine and plan safety requirements specifically associated with sampling requirements based on study area features, COPCs, and personal health and safety precautions (e.g., chemically resistant clothing and gloves, respiratory protection, ear and eye protection , *etc.*).

- Determine appropriate level of respiratory protection required to protect against diseases or bacteria known to be carried by small mammals in the area.

- Check proper functioning and integrity of PPE, including respirators and filters.

- If formaldehyde solution will be used, it must be shipped as Dangerous Goods, and treated in a manner consistent with Workplace Hazardous Materials Information System (WHMIS) requirements (e.g., stored in the hazardous chemical storage locker and used under a fume hood, with material safety data sheets provided).

- Hard hats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet.

- File a field plan with a land-based supervisor.
- Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should carry an adequate supply of water or other liquids for protection against dehydration in hot weather.

IMPORTANT:

Hantaviruses are part of a group of viruses called the Bunyaviridae. Exposure to hantaviruses can cause a rare, but often fatal, disease called Hantavirus pulmonary syndrome (HPS).

The following flu-like symptoms can result from HPS:

- fever chills
- muscle aches
- headaches
- nausea
- Stomach problems.

Symptoms can appear within 3 to 60 days after exposure. However, the average time it takes for symptoms to appear is 14 to 30 days after exposure. HPS is extremely serious since approximately 30-40% of cases result in death

Personnel performing rodent trapping and specimen collection should be made aware of the risks associated with these tasks and precautions to minimize these risks. Baseline serum samples should be collected from each worker and stored at -20°C.

Personnel should be made aware of symptoms of Hantavirus infection or other diseases and advised to seek medical attention if these symptoms occur within 45 days of exposure.

If the physician suspects hantavirus infection, he or she should contact local public health authorities. The physician should collect a blood specimen from the patient and send it with the baseline serum to the health department for hantavirus testing.

The Public Health Agency of Canada (PHAC) conducts testing of hantavirus infections in humans and analysis of trends in HPS cases in Canada.

Refer to Health Canada, & Public Health Agency of Canada (2009) for more information,
or contact: National Microbiology Laboratory, Public Health Agency of Canada
1015 Arlington Street Winnipeg, MB, R3E 3P6, Telephone: 204-789-2000

☐ Reserve, order, and pack all required equipment

- Obtain and prepare the necessary sampling and storage equipment, sample containers, materials, and documents.
- Check proper functioning and integrity of field equipment, including traps and generator (if used).
- For efficiency and to reduce field decontamination activities, all sampling equipment should be cleaned and decontaminated at the laboratory or field office before going to the sampling site. If possible, a separate set of cleaned and decontaminated equipment should be available for each sampling site.
- Prepare a field notebook for documenting all activities related to field sampling. Information to be recorded in the field notebook includes, but is not limited to, weather conditions, progress toward accomplishing sampling objectives, deviations from the field sampling plan, sample identification numbers consistent with what is recorded on the laboratory chain-of-custody forms, sample observations, preservatives used and shipping protocol.

☐ Determine whether permits are needed for small mammal trapping and/or handling, and obtain any required permits from the appropriate territorial, provincial, and/or federal regulatory agencies.

☐ If access to private property will be required, obtain written access permission from landowners.

☐ Obtain First Aid/CPR certification and appropriate safety training for all field personnel. Rabies vaccinations may be advisable for field personnel frequently exposed to wild animals.

Sample Collection:

General information

1) While placing clean traps, a long-sleeved shirt, long pants, socks, and lace-up shoes should be worn. These clothes should be laundered at the end of the day.

2) Prepare bait. In areas where ants are a problem, the oats may be used without the peanut butter.

3) Attach a strip of white tape (about 10 centimetres [cm] long) to the top of each trap on the side nearest the door. This tape can be used for numbering the traps or noting the trap line number or habitat when captured animals are collected.

4) Traps containing captured rodents should be handled only while wearing thick rubber gloves. Rubber gloves are preferred over leather gloves because they can be easily decontaminated with disinfectant. Latex gloves will not provide adequate protection because they are easily torn on sharp trap surfaces.

5) Traps containing captured rodents should be placed immediately into double plastic bags and the bags should be tied closed.

6) If a pickup truck is available, the bags containing captured rodents should be transported in the bed of the truck to provide extra security to those in the passenger section.

7) On hot days, the traps should be covered with a light-colour tarp to prevent the sun from overheating the animals.

Setting and Checking Traps

1) Density of small mammals and sampling success are greatly influenced by the quantity and quality of habitat present at a study area. When initially setting traps, it is advisable to establish arrays of traps (default = 10 traps) at several sampling locations, and to increase or decrease the number of traps per location based upon sampling success after the first night.

2) Place traps as level as possible on the ground, in areas that are out of sight of roads, sidewalks, paths, or other areas of human activity, if possible. Avoid large game (e.g., deer) trails and areas frequented by livestock, in order to prevent destruction or accidental tripping of traps. If possible, locate traps in areas along logs or in other areas that provide cover.

3) Place a small amount of bait (e.g., peanut butter with or without rolled oats) on or in each trap and set the trap. For live traps, addition of cotton ball or cotton batting may be desirable in areas with low overnight temperatures to protect small mammals from hypothermia. Small mammals are susceptible to hypothermia due to their high metabolism and the stress of being trapped. If bait is used, submit a sample to the analytical laboratory to ensure it does not contain detectable concentrations of COPCs.

4) Indicate the location of each trap with a pin flag or a small piece of flagging tape attached to overhanging or adjacent vegetation.

5) It is not necessary to record GPS coordinates for every individual trap location, provided that the overall trap array is co-located with soil samples. Otherwise, document the trap array location using GPS unit and/or manual measurements to known locations. Photodocument each trap array location and record the photograph number in the field notebook.

6) Traps should be checked as early in the morning as possible, especially in hot weather and when traps are exposed to direct sun. In hot seasons or climates, avoid placing traps in areas which will be exposed to direct sun. If this is impossible, traps may be covered with a board or with canvas cloth. If freezing temperatures are likely, add two cotton balls to each trap to provide nesting material during the night.

7) Check each trap for evidence of capture or visitation. If a trap appears to have been visited but not sprung (e.g., contains urine, feces, or nesting material in or on the trap), place the trap in a double plastic bag to be decontaminated and check for proper function. Replace the trap with a clean trap.

Collecting small mammals

1) Personnel not participating in the collection and not wearing respirators should remain upwind and at least 10 metres (m) from the collection area.

2) All personnel who handle rodents or traps should wear long pants and long-sleeved shirt, coveralls, socks and heavy shoes, one pair of non-powdered nitrile-type gloves, safety goggles, and an air-purifying respirator equipped with N-100 or P-100 filters.

3) After placing animals in the vehicle, wash rubber gloves thoroughly in soap and water, then remove gloves and wash bare hands in soap and water.

4) When handling live animals, personnel should wear rubber gloves, carefully grasp the organism, and perform cervical dislocation (if the animal is not to be released). Only experienced biologists should perform cervical dislocation.

5) If the small mammal is dead, carefully position the trap over a zip-top bag and open trap, allowing animal to fall into bag.

6) Squeeze excess air from bag and seal. Label bag with trap number/location, date, and time collected. Photodocument the sample.

7) All information regarding the sampling event should be accurately recorded in field notes, in order to support the subsequent preparation of a sampling event report.

IMPORTANT: Euthanasia by means of anaesthetic overdose generally cannot be used when submitting samples for chemical analysis, due to their potential to introduce artefacts. Therefore cervical dislocation is the recommended method of euthanasia.

Processing small mammals

1) Personnel not participating in sample processing and not wearing respirators should remain upwind and at least 10 metres from the processing area. If processing is conducted indoors, personnel who are not wearing respirators should not enter the processing room until work has been completed and any contaminated disposable materials have been properly stowed, and the room has been well ventilated for 30 minutes.

2) All personnel participating in the handling of rodents or traps should wear complete protective clothing, including surgeon's gown or coveralls (preferably disposable), disposable shoe covers, two pairs of latex gloves, safety goggles, and a half-face respirator or powered air purifying respirator equipped with HEPA filters.

3) For each individual small mammal, identify species and gender. Record weight, total length, body length, tail length, and hind foot length, and any visual gross abnormalities (e.g., deformities, scars, ectoparasites).

4) Place each sample on wet or dry ice and place in freezer or otherwise keep frozen until ready to ship to the analytical laboratory.

5) When the last animal has been processed, place all contaminated paper towels, plastic bags, and table coverings in a biohazard bag. Seal bag.

Decontaminating equipment

1) Prepare one 17-litre plastic bucket containing about 15 litres of 1:20 dilution of industrial strength liquid disinfectant.

2) When handling traps, wear heavy leather gloves over non-powdered nitrile-type gloves to avoid tearing the non-powdered nitrile-type gloves on sharp trap surfaces.

3) While wearing PPE described above, place used traps into the bucket of liquid disinfectant solution. Allow the trap to soak in the disinfectant for at least one minute.

4) Following disinfection:

    a. For disposable snap-traps, transfer traps from liquid disinfectant into biohazard bag, and seal the bag.

    b. For re-usable live traps (Sherman or Havahart), proceed to decontaminate with surfactant detergent and allow to dry.

5) Dispose of the used bath liquid by flushing down the drain with plenty of water.

6) Spray all contaminated surfaces with disinfectant and soak before wiping – this will prevent aeroionisation of infected particles. Wipe down all working surfaces, table and chairs, and all equipment on the processing table (balance, markers, even the disinfectant spray bottle) with disinfectant.

7) Place all disposable materials into biohazard bag.

8) Wash outer gloves well with soap and water and then spray with disinfectant. Remove outer gloves and allow to dry.

9) Remove inner gloves and dispose into biohazard bag. Close and seal bag.

10) Wash hands with soap and water.

11) Remove respirator. Clean and disinfect.

Shipping samples

1) Tissue samples should be stored at or below 4°C until shipped.

2) Ship to analytical laboratory daily or according to sampling and analysis plan.

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