Part E Water and Wastewater Sampling

Ambient Freshwater and Effluent Sampling

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Preface

Stakeholder Representation

This manual was prepared in cooperation with stakeholders as represented by the British Columbia Laboratory Quality Assurance Advisory Committee (BCLQAAC) and by the BCLQAAC Technical Subcommittee.

Purpose

All persons required to submit environmental monitoring data as a requirement of an order, permit, licence, approval or certificate under an enactment administered by the Minister of Water, Land and Air Protection should, wherever practical, only report data for samples collected and tested in accordance with methods specified in this manual. Nonetheless, this manual should be viewed as a general guide to sampling. Other sources should be referenced for detailed protocols, equipment options, and insights into the usefulness of a particular method for a given situation.

Abstract

The protocols in Section E describe the standardized field procedures including quality control practices such as the types of quality control samples to be incorporated into the sampling plan, field filtering, sample preservation and shipping protocols. The section further describes the sample collection techniques and equipment used for lake, stream/river surface, depth and shore sampling, as well as winter sampling. Effluent sampling protocols include the types and protocols for sampling waste streams, flow measurements and field tests such as DO, temperature, conductivity and pH.

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The Resources Information Standards Committee evolved from the Resources Inventory Committee which received funding from the Canada-British Columbia Partnership Agreement of Forest Resource Development (FRDA II), the Corporate Resource Inventory Initiative (CRII) and by Forest Renewal BC (FRBC), and addressed concerns of the 1991 Forest Resources Commission.

For further information about the Resources Information Standards Committee, please access the RISC website at: http://ilmbwww.gov.bc.ca/risc/index.htm.

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1. Introduction

This section covers the minimum requirements to ensure quality and consistency of the field aspects of ambient water and effluent data collection. The essential tasks in water sampling are to obtain a sample that meets the requirements of the program, in terms of location and frequency, and to prevent deterioration and contamination of the sample before analysis. The procedures outlined in this section are oriented primarily towards Ministry of Water, Land and Air Protection employees, consultants, or those under a legal requirement to undertake a sampling program for the Ministry. The protocols outlined in this section will aid field staff in collecting reliable, representative water samples.

The protocols presented here are the most acceptable ones used at present. It should be emphasized that in unusual circumstances, or with development of new methods, experienced professional judgment is a necessary component of method choice and application. It is intended that this document will be updated as the need arises to reflect new knowledge.

This section does not address the collection of samples for the purpose of providing legal evidence. For information regarding legal sampling, refer to Guidelines for the Collection and Analyses of Water and Wastewater Samples for Legal Evidence (Lynch and van Aggelen, 1994).

This section also does not address project design (site locations, frequency of sampling, duration, quality assurance program, etc.) or data interpretation. It also does not address the collection of groundwater samples. The protocols for the collection of ambient groundwater are documented in the Groundwater Sampling chapter of this manual.

The sample containers, preservatives and sampling procedures described in this section reflect those most widely used by Ministry of Water, Land and Air Protection. Shipping procedures and safety measures are also outlined. Different agencies or laboratories may have specifications that differ from those described here.

It should be acknowledged that funding for the initial manuscript upon which this section is based was provided by the Aquatic Inventory Task Group of the Resource Inventory Committee.

2. General Considerations

2.1 Preparing to Go to the Field

Preparation for each sampling trip is critical since oversights are not usually noticed until staff reach the first station. The most effective way to prepare for a sampling trip is with a **checklist** that is designed to meet the requirements of each project.

Other than considering site-specific instructions, the checklist should identify the following:

- Type and number of (labelled) bottles, including extras
- Field equipment such as meters (with adequate trouble-shooting equipment for small repairs), sampling tools (multiple samplers, through ice samplers, Van Dorns, automatic composite samplers) and filtration apparatus
- Preservatives
- Appropriate quantity of ice packs and coolers
- Logbooks
- Personal gear (for all possible weather conditions, e.g., survival suits, raincoats, protective footwear, waders, gloves, etc.)
- First aid kit
- Equipment (checked and calibrated, properly loaded to avoid damage during transport, batteries charged, probes not damaged or dried, etc.)
- Camera or video equipment as required
- Before going to the field:
- Contact a qualified laboratory to arrange for the required analyses
- A recommended operating procedure is to have the key equipment in a box or plastic “tote” that is dedicated to this activity. Appendix 1 of this chapter presents an example of a generic checklist.

2.2 Locating the Site in the Field

It is the responsibility of the field staff to locate all sampling stations accurately. Only if the same location is consistently sampled can temporal changes in the water quality be interpreted with confidence. Therefore, accurately written station location descriptions (that identify key landmarks and give the site a simple and unambiguous name) must be prepared on the first visit to every sampling site (see Appendix 2.1 for an example of a site identification guide sheet). Good photographic documentation is the best way of ensuring that each site is easily recognized.

A map that labels the sample sites should accompany the **site identification logbook**. This can be in the form of a 3-ring binder with a 1:50 000 map. The basic site location data (see Appendix 2.1 - latitudes, longitudes, map sheet #, site identification #, etc.) should be incorporated into the Water Quality database

(EMS in the case of Ministry of Water, Land and Air Protection). In many cases, a detailed site map may be helpful in describing the station location. Global Positioning Systems (GPS) are becoming common tools for locating position of sites and are recommended for this purpose.

2.3 Field Notes/Observations

Good sampling practice always involves the use of detailed field notes. Specific information about seemingly unimportant facts, such as the time of day or weather conditions, is often important when interpreting data. A **field logbook** (3-ring binder with water proof paper) for each project is mandatory (see Appendices 2 and 3 of this chapter for examples of data sheets). All field measurements should be entered directly into this field logbook while in the field. All information recorded in the logbook should be entered into the database immediately upon return from the field.

In addition to documenting standard conditions and measurements, field staff are responsible for noting any unusual occurrences. Any deviations from standard protocols (e.g., samples taken from a different location due to safety or access considerations or procedures used that differ from those outlined here) must be recorded in the database. Upon observing an anomalous condition, such as an unusual colour or odour of the water, excessive algal growth, indications that foreign substances have entered the system (oil slicks, surface films, etc.), or fish kills, the field investigator should take samples in addition to those required by the project design. The type of samples and their preservation should be consistent with the type of analyses that the investigator thinks are warranted by the prevailing conditions. If additional samples are collected, but not exactly at an established station, a new site location description should be accurately recorded and transferred to the database (EMS) as soon as possible. This information and additional samples will prove useful during the interpretive aspects of the study.

The field books are important documents and efforts should be made to ensure they are properly archived.

3. Quality Assurance/Quality Control (QA/QC)

3.1 Field Quality Assurance

The field quality assurance program is a systematic process that, together with the laboratory and data storage quality assurance programs, ensures a specified degree of confidence in the data collected for an environmental survey. The Field Quality Assurance program involves a series of steps, procedures and practices that are described below.

The quality of data generated in a laboratory depends, to a large degree, on the integrity of the samples that arrive at the laboratory. Consequently, the field investigator must take the necessary precautions to protect samples from contamination and deterioration.

There are many sources of contamination; the following are some basic precautions to heed:

- Field measurements should always be made using a separate sub-sample that is then discarded once the measurements have been made. They should never be made on a water sample that is returned to the analytical laboratory for further chemical analyses. For example, specific conductance should never be measured in sample water that was first used for pH measurements. Potassium chloride diffusing from the pH probe alters the conductivity of the sample. Similarly, pH should not be measured from a sample that will be analyzed for phosphorus, as some pH buffers contain phosphorus. Use a separate bottle for water temperature if not in-situ. **Dissolved oxygen measurements (by DO probe) should be made in-situ rather than in a separate container.**

- Sample bottles, including bottle caps, must be cleaned according to the recommended methods and certified by the issuing laboratory as 'contamination free' (if pre-cleaned by the laboratory), for the intended analysis. Sample bottles that are pre-cleaned by the laboratory must **not** be rinsed with the sample water being collected. Bottles must be supplied with cap in place. Note that cleaned reused bottles are not suitable for some trace constituents. If you are using a mixture of pre-cleaned, not pre-cleaned, and/or re-used bottles, label each bottle type to avoid confusion.

- Use only the recommended type of sample bottle for each analysis (see Appendix 4 of this chapter).

- Reagents and preservatives must be analytical grade and certified by the issuing laboratory to be contamination free (see Appendix 4). Containers holding chemical reagents and preservatives should be clearly labelled both as to contents and as to expiry date. No reagent or preservative should be used after the expiry date. Return expired reagents to the laboratory for proper disposal.

- If conditions dictate that samples from multiple sites be preserved at the same time (such as when returning to shore after sampling several deep stations), the possibility of adding the wrong preservative to a sample or cross-contaminating the preservative stocks should be minimized by preserving all the samples for a particular group of variables together. Colour-coded bottles and matching preservatives prevent mix-ups.

- The inner portion of sample (and preservative) bottles and caps must not be touched with anything (e.g., bare hands, gloves, thermometers, probes, preservative dispensers, etc.) other than the sample water and preservative. Remove caps only just before sampling and re-cap right away.

- Keep sample bottles in a clean environment, away from dust, dirt, fumes and grime. Bottles must be capped at all times and stored in clean shipping containers (coolers) both before and after the collection of the sample. Vehicle cleanliness is an important factor in eliminating contamination problems. During sample collection, store bottle caps in a clean, resealable plastic bag, not in pockets, etc.

- Petroleum products (gasoline, oil, exhaust fumes) are prime sources of contamination. Spills or drippings (that are apt to occur in boats) must be removed immediately. Exhaust fumes and cigarette smoke can contaminate samples with lead and other heavy metals. Air conditioning units are also a source of trace metal contamination.

- Filter units and related apparatus must be kept clean, using routine procedures such as acid washes and soakings in de-ionized water (see section 9). Store cleaned filter units in labelled, sealed plastic bags.

- Samples must never be permitted to get warm; they should be stored in a cool, dark place. Coolers packed with ice packs are recommended (most samples must be cooled to 4°C during transit to the laboratory). Conversely, samples must not be permitted to freeze unless freezing is part of the preservation protocol (Appendix 4). Cool samples as quickly as possible. A common mistake is to forget that a large volume of warm water soon melts a small amount of ice.

- Samples must be shipped to the laboratory without delay so that they arrive within 24 hours of sampling. Certain analyses must be conducted within 48 hours or within specified time limits set out in Appendix 4.

- Sample collectors should keep their hands clean and refrain from eating or smoking while working with water samples.

- Sample equipment and shipping coolers must be cleaned after each sampling round (see Section 9). Field cleaning is often not as effective as cleaning equipment at a support facility. Depending upon the analyte and concentration (i.e., metals or organics), it may only be possible to conduct effective cleaning procedures at a support facility, rather than in the field. Avoid using bleaches and strong detergents; specialty cleaning compounds are available.

- De-ionized water should not be used after 6 months (shelf-life period), and the containers should be clearly labelled with both the filling date and disposal date.

Note: Bottle cap liners of composite materials such as Bakelite must not be used due to high potential for contamination.

3.2 Quality Control

Quality control is an essential element of a field quality assurance program. In addition to standardized field procedures, field quality control requires the submission of blank samples to test: **1)** the purity of chemical preservatives; **2)** to check for contamination of sample containers, filter papers, filtering equipment or any other equipment that is used in sample collection, handling or transportation; and **3)** to detect other systematic and random errors occurring from the time of the sampling to the time of analysis. Replicate samples must also be collected to check that the sample is reproducible. Replicate samples allow the precision of the sampling and measurement process to be estimated, and are an additional check on sample contamination. The timing and the frequency of blank and replicate samples are established in the project design and will vary with each project. A minimum level of effort would be the use of blanks and replicates consisting of 10% of the samples. Another aspect of quality control is the use of certified or standard reference materials (CRM's or SRM's) and of spiked samples to assess laboratory process.

3.2.1 Blanks

Blanks are samples that do not contain the variable to be analyzed and are used to assess and control sample contamination. They are most often used to assess contamination of the trace measurements (metals and nutrients) but should also be used on occasion to test potential contamination of the other analyses (such as general ions). Most blanks are carried through the entire sample collection and handling process so that the blank is exposed to the same potential sources of contamination as actual samples. Ideally, blanks should be prepared by the analytical laboratory in the appropriate sample bottles under clean conditions.

Some of the blanks remain in the laboratory for analysis (laboratory blanks), while the remainder travel to the field for use as trip, field, equipment, and filtration blanks. Alternatively, blanks may be prepared in the field as outlined below.

3.2.1.1 Trip Blanks

Trip blanks are meant to detect any widespread contamination resulting from the container (including caps) and preservative during transport and storage. The recommended practice for organic parameters is to use carbon free de-ionized water for trip blanks.

PROTOCOL

(a) Prior to a field sampling trip, one or more sample bottles for each type being used during the trip are selected at random, filled with de-ionized water provided by an analytical lab (preferably one different from the one samples are sent to) and preserved in the field in the same manner as field samples (see section 7.2).

(b) These bottles are capped and remain **unopened** throughout the sampling trip. They are transported to the field with the regular sample bottles and submitted with the field samples for the analysis of interest.

3.2.1.2 Field Blanks

Field blanks mimic the extra sampling and preservative process but do not come in contact with ambient water. Field blanks are exposed to the sampling environment at the sample site. Consequently, they provide information on contamination resulting from the handling technique and through exposure to the atmosphere. They are processed in the same manner as the associate samples (i.e., they are exposed to **all** the same potential sources of contamination as the sample). This includes handling and, in some cases, filtration and/or preservation.

PROTOCOL

(field blanks)

(a) If the blank was prepared by the lab, then open the bottle to expose the de ionized water to the air for as long as the sample was exposed when it was collected. Otherwise, when the blank is prepared in the field, pour deionized water into the pre-labelled field blank bottle and recap it (this simulates sample collection). Document whether it was a lab prepared or a field prepared blank.

(b) Filter the sample as per the protocol outlined in section 7.1 (only if the associate sample requires filtration).

(c) Add preservative as per section 7.2 (only if the associated sample requires preservation).

(d) Ship to the lab with the remaining samples.

3.2.1.3 Equipment Blanks (prepared prior to the field trip)

A field equipment blank is a sample of de-ionized water that has been used to rinse sampling equipment. This blank (perhaps more properly described as a rinsate) is useful in documenting adequate decontamination of equipment. It is collected after completion of the decontamination process (washing) and prior to sampling.

PROTOCOL

(a) Pour the rinse (de-ionized) water used for the last rinsing into a prelabelled bottle that identifies the piece of equipment cleaned.

(b) Submit the blank with the regular samples for analysis.

3.2.1.4 Filtration Blanks

Filtration blanks (or rinsate blanks) are de-ionized water that is passed through the filtration apparatus in the same manner as the sample. Analysis of the filtrate provides an indication of the types of contaminants that may have been introduced through contact with the filtration apparatus. Filtration blanks are also used as a check for potential cross-contamination through inadequate field cleaning techniques (rinsing of the apparatus with de-ionized water between samples). It should be done both at the start and again at some point between samples (after the apparatus has been cleaned and immediately before the next 'real' sample is filtered). Each blank is preserved in the same fashion as the associate samples.

PROTOCOL

(a) Follow procedure outlined in section 7.1 (filtration).

3.2.2 Replicate Samples

3.2.2.1 Co-located Samples (field duplicate, triplicate, etc.)

Co-located samples are independent samples collected as close as possible to the same point in space and time and are intended to be identical. These samples are essential in documenting the precision of the entire sampling and analytical (laboratory) process.

For this procedure, simply follow (and repeat) the protocol outlined in section 4 (sample collection).

Note: Replicate samples have more information than either blanks or split samples, and are particularly recommended for QC studies.

3.2.2.2 Split Samples

Split samples are aliquots taken from the same container and analyzed independently by one or more laboratories. They are used to obtain the magnitude of errors owing to contamination, random and systematic errors, and any other variability, that are introduced after the time of sampling through analysis at the laboratory(ies). Split samples are commonly used to compare two or more laboratories. Care must be taken to ensure that the samples are split in a way to ensure homogeneity (a sample splitter must be used for samples containing suspended solids or effluents).

3.2.3 Spiked Samples (Field)

Spiked samples for each variable being tested are prepared by spiking aliquots of a single water sample with known amounts of the variable of interest. The information gained from spiked samples is used to reveal any systematic errors (or bias) in the analytical method. The spike solution is prepared by an analytical laboratory (preferably) or it can be prepared by the field staff (far less desirable) prior to the sampling trip.

PROTOCOL
(spiked samples)

(a) Collect the sample in a pre-labelled bottle as per section 4.

(b) Add the aliquot of spike solution, recap the bottle, mix and then treat the sample as if it were a regular sample (i.e., preserve and filter if requires)

3.2.4 Reference Samples

Reference samples are used to document the bias of the analytical (laboratory) process. There are two types of reference samples. The first, and simplest, is when an independent laboratory prepares a water sample with the addition of a known quantity of a variable of interest. In this case, the independent laboratory should provide calculated and measured concentrations on the variable.

The second type of reference material is a certified reference sample. It is obtained from a recognized national scientific body such as the National Research Council. The sample itself is an aliquot of a very large stabilized (may be preserved) batch sample that was collected from one place at one time. The batch sample has been subjected to a large number of analyses performed by independent laboratories using several different analytical techniques, but some reference materials are analyzed by different labs using the same methodology. Consequently, the distributing agency can provide a mean value and confidence interval for the variable of concern.

These samples are submitted blind to the analyzing laboratory along with the samples collected during a field trip. There is the option of submitting them blind (labelled as a regular sample) or non-blind with labelling that it is a certified reference material. The former is a more desirable QA tool.

4. Collecting Samples

Water samples are often obtained by filling a container held just beneath the surface of the water, commonly referred to as a dip or **grab sample**. Through the use of special depth samplers (such as a Van Dorn bottle), grab samples can also be obtained from deep waters. This is important as distinct thermal and chemical differences can occur throughout the water column. **Composite samples** are obtained by mixing equal volumes of discrete grab samples (collected at one point at regular time intervals or, collected from multiple points such as varying depths). A composite sample provides an estimate of average water quality conditions.

Note: If sample bottles have not been pre-cleaned by the laboratory, then they must be rinsed 3 times with either de-ionized water or sample water. The exception to this is when a sample is to be analyzed for suspended sediments, for contaminants likely associated with the suspended solids, or for oil and grease. In these cases, the bottles should not be rinsed with sample water as suspended particles or grease-like materials are retained on the interior surface of each bottle with each rinsing. For specialized analyses (trace metal, organics) and pre-cleaned bottles, containers should not be rinsed. Rinsing is not a recommended practice. Use of pre-cleaned bottles is recommended, where practical. Where bottles are rinsed, the rinsate should be discarded.

Special sampling and handling techniques known as "clean" and "ultra clean" methods are needed to achieve accurate results when measuring low-level trace metals in ambient waters. Clean methods are needed to quantify trace metals accurately when the concentrations are less than about 20 mg/L and down to 0.1 mg/L. Ultra clean methods are needed when the metal concentrations are less than 0.1 mg/L, as might be required for trace metals such as mercury, cadmium, or silver (Hunt et al., 1995). These methods are not in general use in British Columbia at this time, and detailed guidance on the methods has only recently become available. We expect that guidance on clean and ultra clean techniques will be added to the next edition of this field manual. In the interim, sample collectors should refer to the recent US Environmental Protection Agency report of the subject (USEPA, 1995).

4.1 Lake

Sample stations can be located either near-shore or in deeper waters (deeper sites are typically located over the deepest point of the lake). In general, the near-shore sites detect those effects that are associated with influences such as groundwater and runoff. Deep stations provide information about the water column, such as conditions associated with stratification (depth profiles). Additionally, near-shore sites tend to provide information on a relatively short time scale (days or weeks). The deeper sites allow for documentation on a seasonal or longer time frame.

4.1.1 Shore Sample

Sample collection at near-shore stations generally consists of grab samples at a specified location. It is critical that there be no deviation in location unless conditions at the site (e.g., severe weather, physical changes of the site, etc.) pose a threat to the sampler's safety. **If safety is threatened, then search for an alternative location nearby, or simply do not attempt to take the sample**. If an alternative location is found, then all details regarding the new site and the reasons why the alternative was necessary must be recorded in the field logbook. This information should be entered into the database as soon as possible after returning from the field.

To avoid contamination from suspended sediments, the sample collector should preferably sample from a boat or a dock or, if that is not possible, should wade out past the point where wave action affects the lake bottom. In most cases, this distance is not far from shore. But, in any case, **the sampler should not exceed a depth where there exists a reasonable possibility that water might enter the gum-boot or hip-wader**. This is particularly important during colder periods of the year when getting wet poses a health risk (such as hypothermia).

PROTOCOL
(for collecting shore samples)

(a) Obtain labelled bottles and wade into the lake at the most accessible point.

(b) Once you reach a sufficient depth (where bottom material will not interfere with the sample), stop and orient yourself towards the center of the lake.

If rinsing is required (see section 4, Collecting Samples), proceed from step (c), otherwise start at step (h).

(c) Remove the lid and hold it aside without touching the inner surface.

(d) With your other hand, grasp the bottle well below the neck. Lean out towards the center of the lake and, in one continuous motion, plunge the bottle beneath the surface and slowly force it through the water until it is partly full. This motion creates a current over the mouth of the bottle (such that water entering the bottle has not come in contact with your hand).

(e) Replace the lid and shake the bottle vigorously.

(f) Remove the lid and reach back towards shore to pour the water out.

(g) Repeat steps (c) through (f) twice more before collecting the sample.

(h) Remove the lid (without touching the inner surface) and grasp the bottle well below the neck. Lean out towards the center of the lake and, in one continuous motion, plunge the bottle beneath the surface and slowly force it through the water until it is full.

(i) Replace the cap immediately.

(j) Return to shore and pack the sample(s) in a cooler until time and conditions permit for other necessary procedures (filtration and/or preservation, that should be done as soon as possible after the samples are collected).

4.1.2 Sampling from a Boat

The collection of deep water samples requires that at least one member of the sampling group be very familiar with boat operation and safety. **If the sampling trip involves the use of a boat, then the weather forecast or marine conditions should be obtained prior to departure from home. If conditions are poor, then the sampling trip should be postponed.**

4.1.2.1 Site Identification

Deep water sampling sites are marked with a buoy or referenced by easily identifiable features (preferably two) on shore. Reference points should be described (both in writing and with photographs) in the site identification logbook. Once at the site, and if it is not too deep, anchor the boat (or tie it to the buoy) and wait until it settles with the bow (front) facing into the current (wind) before collecting the sample. If the water is too deep to anchor, then one person will have to maintain the location (with either the motor or with paddles) while the other person collects the samples and takes the field measurements.

4.1.2.2 Surface Water

PROTOCOL
(for collecting surface water)

(a) The person at the bow (front) should always collect the samples. This is because the bow is the anchor point and, even in slow moving water, the boat will drift so that the bow is upstream. In quiescent water the samples should be collected prior to anchoring and while the boat is slowly moving forward. These precautions reduce the potential of contamination from the boat or motor. The person in the stern (rear) can be responsible for holding the boat's position (when not anchored), taking the field measurements (see section 6) and field notes. Contamination is not as much of a concern for field measurements.

(b) Obtain a labelled sample bottle and remove the lid without touching the inside of the lid (or bottle!). If rinsing is required for the type of bottle, fill and rinse three times [see 4.1.1 (c) to (g)].

(c) Reach out an arm length from the boat to take the sample. Ensure that the person in the stern is providing counterbalance (working over the opposite side of the boat).

(d) Plunge the bottle under the surface and move it slowly towards the current (the direction the boat is facing). This should be done at a depth of approximately 0.5 meters.

(e) Recap the bottle immediately and proceed with the next sample.

(f) Samples requiring filtration and/or preservation (see sections 7.1 and 7.2) should be dealt with as soon as possible after returning to shore.

4.1.2.3 Deep Water

Lake water samples may be collected from any desired depth through the use of a **Van Dorn** (or similar) sampler (Figure 1). The Van Dorn bottle is designed for sampling at a depth of 2 metres or greater. A drain valve is provided for sample removal. Note that Van Dorn samplers are available in both horizontal and vertical configurations. The advantage of the vertical configuration is that the water within the open bottle is flushed out as the bottle is lowered, so one can be guaranteed the water collected was collected from the indicated depth. The advantage of the horizontal configuration is that a very narrow depth range is sampled. Vertical configurations are most commonly used. The horizontal configuration should be used when samples are taken near bottom at the sediment-water interface, or when samples are required from a narrow band of the depth profile (i.e., chemo cline, thermocline).

The sampling sequence recommended is to obtain the field measurements first (temperature, DO, conductivity - see section 6). These are often necessary prerequisite for locating the depths from which the water samples should be taken (i.e., if three deep samples are required at a site then it might be necessary to know the depths of the major stratified zones - epilimnion, thermocline, hypolimnion).

Although operation of the Van Dorn bottle varies slightly depending on its size and style, the basic procedure is the same.

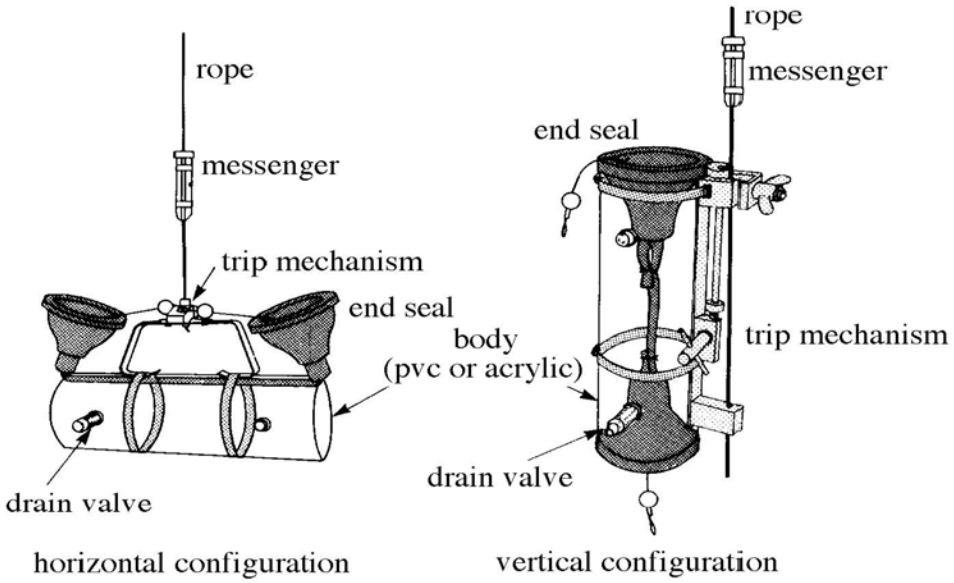

Figure 1. Van Dorn sampler

PROTOCOL

(for collecting deep water)

(a) Ensure the sampling bottle is clean.

(b) Open the sampler by raising the end seals.

(c) Set the trip mechanism.

(d) Lower the sampler to the desired depth.

(e) Send the messenger down to "trip" the mechanism that closes the end seals.

(f) Raise the sampler to the surface.

(g) Transfer the water sample from the Van Dorn bottle to individual sample containers via the drain valve. Take care to avoid contact with the drain spout as contamination at this stage often occurs.

(h) Rinse bottles 3 times (if they have not been pre-washed), and collect sample (see section 4.1.1).

(i) Filter and/or preserve the samples as required once at shore.

4.1.3 Winter Sampling

Sampling in winter presents extra elements of danger. **Always proceed with caution over ice and do not jeopardize your safety**. Check the ice for thickness with a rod or ice chisel every few steps (ice should be a minimum of 3 to 4 inches thick). Ice over moving water can be of varying thickness, and the strength of the ice cannot be estimated from its apparent thickness near the shore. **Always have someone accompany (follow) you, wear a life jacket, and carry a length of rope (tied around your waist) to use as a life line. If the ice is unsafe, do not take a sample. Never take unnecessary risks.**

Note: Ice near the outlet of a lake is often thin; therefore, use caution when sampling this area of a lake. Additionally, ice thickness on reservoirs, where water levels fluctuate, can be variable.

In springtime, ice can be thick, but not strong enough to walk on (often called "Frazzle" or "corded" ice).

PROTOCOL
(for sampling through ice)

(a) With safety considerations in mind, winter sampling locations should be as close as possible to the summer locations. The sites should be chosen where the water is known to be deep enough to avoid stirring up bottom sediments and to ensure that there is water movement under the ice at your selected spot. It is preferable to select a site where the ice is sagging rather than bulging.

(b) Clear loose ice and snow from the sampling location, and drill through the ice with a hand or motorized auger. Keep the area around the hole clear of potential contamination (e.g., dirt, fuel, oil, etc.). **At least one member of the sampling team should be familiar with the operation and safety of both motorized and hand operated augers**.

(c) Remove all ice chips and slush from the hole, using a plastic sieve.

(d) Use a Van Dorn (or similar) sampler to collect the sample (see section 4.1.2.3)

(e) Do not allow samples to freeze.

Note: An alternative to this method would be to use the Through-Ice sampler described in section 4.2.4 - Winter Sampling on rivers (this technique does not allow the collection of samples that are deeper than 2 metres). Any deviations from the above protocol must be noted in the field logbook.

4.2 River/Stream

The majority of samples collected from streams and rivers in British Columbia are grab samples taken near the surface at one point in the cross section of the flow. On rare, special occasions, more sophisticated multi-point sampling techniques known as equal-discharge-increment (EDI) or equal-width-increment (EWI) methods are used. Since these techniques are infrequently used they will not be discussed here, but further information about the protocols can be obtained from Clark and Shera, 1985.

Note: The collection of samples for the purpose of assessing the suspended sediment load in fast flowing waters requires specialized techniques/equipment. The equipment is not readily available; therefore, the protocols will not be discussed here. For information regarding the equipment and techniques, refer to Guy and Norman (1970) or Stichling and Smith (1968).

4.2.1 Access from the Stream Bank

Wherever practical, samples should be collected at mid-stream rather than near-shore. Samples collected from mid-stream reduce the possibilities of contamination (i.e., shore effects - back eddies, seepage from near shore soils, atmospheric components such as pollen concentrating in slow moving water, etc.). Samples should not be taken in back eddies or brackish waters unless required by the monitoring program objectives. The most important issue to consider when deciding where the sample should be collected from is **SAFETY**. If the flow is sufficiently slow that the collector can wade into the stream without risk, then the sample can be collected at a depth that does not pose a threat (discretion is the key - **never wade into water that appears deep or fast flowing**). When conditions dictate that the sample be taken from the stream bank, deviations from the standard protocol should be accurately documented in the field logbook and transferred to the database as soon as possible. **Samplers must be wary of non-visible bottom under turbid conditions.**

PROTOCOL
(for wading into flow)

(a) Obtain labelled bottles and wade into the river downstream from the point at which you will collect the samples, and then wade upstream to the sample site. This ensures that you will not disturb sediments upstream of the sample point. Attach safety line if conditions have any significant risk.

(b) Stand perpendicular to the flow and face upstream.

(c) Remove the lid and hold it aside without touching the inner surface. If rinsing is required for the type of bottle, fill and rinse three times (see section 4).

(d) With your other hand, grasp the bottle well below the neck. Plunge it beneath the surface in front of you with the opening facing directly down, then immediately orient the bottle into the current. Avoid collecting surface scum and film.

(e) Once the bottle is full, remove it from the water by forcing it forward (into the current) and upwards.

(f) Replace the cap immediately.

PROTOCOL
(for sampling from the stream bank)
(when the current is too strong, water is too deep, or ice is too thin)

(a) Secure yourself to a solid object on shore (with a safety harness and line if necessary). **As a safety precaution, the second person must remain nearby while the first is collecting the samples.**

(b) Remove lid from a labelled bottle and place into a clean resealable bag (e.g., Zip Lock) so both hands can be used to take sample. If rinsing is required for the type of bottle, rinse three times.

(c) Hold the bottle well below the neck or secure it to a pole sampler.

(d) Reach out (arm length only) and plunge the bottle under the water with the opening facing directly down and immediately orient it into the current.

(e) When the bottle is full, pull it up through the water while forcing into the current.

(f) Immediately recap the bottle.

4.2.2 Access from a Bridge

Some sample stations are designed to be sampled from a bridge. This allows the collection of samples from the central flow of rivers where wading is not an option. The samples can be collected using an apparatus called a **multiple sampler** (Figure 2) that is lowered over the side of the bridge. Since the multiple sampler holds more than one bottle, it has the advantage of allowing all containers (therefore, all variables) to be sampled at the same time and at the same place. This allows for more precise cross-referencing among the variables. Other pieces of equipment for single bottles are also available and can be used in situations that are appropriate.

The precise location at which the sampling device is lowered from the bridge should be marked to ensure that the same section of the river is sampled each time.

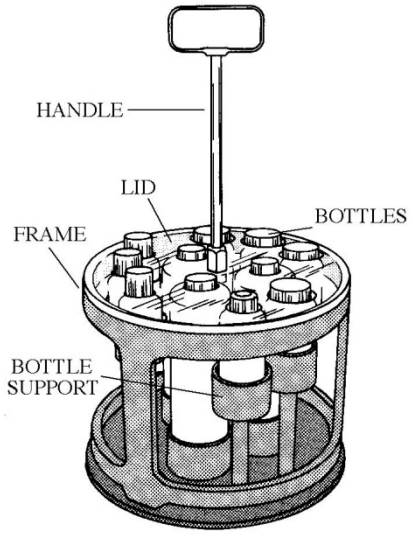

Figure 2. Generalized multiple sampler

PROTOCOL

(from bridge with multiple sampler)

(a) Remove the lid (with handle) from the multiple sampler.

(b) Secure all sample bottles (lids on) into the multiple sampler (as in Figure 2).

(c) Refit the lid to the sampler.

(d) Secure the free end of the sampler's rope to bridge before attempting to take the sample.

(e) Remove lids from the sample bottles and place in a clean resealable bag

(f) (e.g., Zip Lock).

(g) Whenever possible lower the multiple sampler over the upstream side of the bridge (side that the water reaches first), being careful not to disturb bridge surfaces with the rope or sampler. This avoids contamination of the sample from the bridge itself or substances falling into the water or into the open bottles from the bridge (e.g., fuel, oil, dust, wood chips, etc.).

(h) Allow the sampler to submerge to the point that all the bottle openings are below the surface.

(i) After a sufficient period has elapsed to fill all bottles, haul the sampler up, add preservatives where required, and recap each bottle before disassembling the sampler.

4.2.3 Sampling from a Boat

Due to the fact that fast-flowing waters pose a serious threat, it is essential that the person operating the boat be very experienced with river boating. Ideally, there should be three people along on the sampling trip when it involves boating on a river. Two people are responsible for collecting the samples, taking field measurements and recording field notes. The remaining person is responsible for boat operation only.

Sampling trips should start at the site that is most downstream and work upstream. If mechanical problems should arise then the current will work to your advantage and assist you to return to the vehicle.

PROTOCOL

(in flowing waters)

(a) When a sample site is reached the boat operator idles into the current so as to maintain the boat in one location. Use a reference point on shore to do this.

(b) The person in the bow is responsible for collecting the water samples (see section 4.1.2).

(c) The third person is responsible for the field measurements (see section 6).

4.2.4 Winter Sampling

Due to the fact that flow patterns in rivers and streams are generally more complex than in lakes, there are additional safety factors to consider. Honeycombed ice and areas over rapids should always be avoided. Be aware that ice downstream from bridge supports may be thin as a result of modified flow patterns and de-icing agents. At least two people must proceed onto the ice, one ahead of the other. The person in the rear should carry a rope and each must wear a life jacket.

Generally, winter sampling on rivers follows a similar protocol as for sampling lakes in winter (see section 4.1.3). The primary exception occurs when the ice is unsafe; when this is the case, sample stations that are accessible from a bridge are the only option.

When the ice is safe, there are two tools that are commonly used for the collection of water samples, the **Through Ice Sampler** (Figure 3) and the **Flip Sampler/Duncan Sampler** (Figure 4).

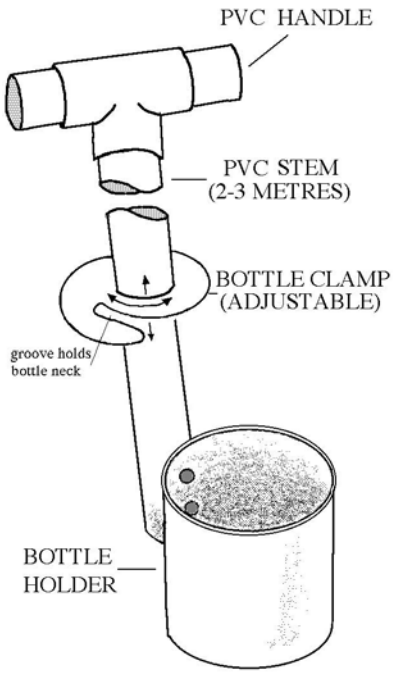

Figure 3. Through ice sampler

PROTOCOL

(Through Ice Sampler)

(a) Clear loose ice and snow from the sampling location, and drill through the ice with a hand or motorized auger. Keep the area around the hole clear of potential contamination (e.g., dirt, fuel, oil, etc.). **At least one member of the sampling team should be familiar with the operation and safety of both motorized and hand operated augers**.

(b) Remove all ice chips and slush from the hole, using a plastic sieve.

(c) Load a pre-labelled bottle into the bottle holder of the **Through Ice Sampler** (Figure 3). Remove the bottle cap and insert stopper (with attached cord) into the bottle opening.

(d) Lower the sampler and bottle through the hole until it is clear of the bottom of the ice surface, and into freely moving water.

(e) Remove the stopper by pulling the cord, and allow the bottle to fill. For the bottle to fill in fast flowing water the sampler may have to be held at different angles.

(f) Bring bottle back up and decant water into the appropriate sample bottles (rinsing if required). For low-level metals analysis, a separate pre-cleaned (acid-washed) collection bottle must be used in the through ice sampler.

There are a variety of unusual conditions that may be encountered in sampling through ice. There may be meltwater below the snow on the ice surface, or there may be a slushy stratum within the ice itself. If these or other conditions occur, they should be noted in the field book and a judgment made as to whether the sample is worth taking.

Note: In streams where the ice is not too thick (20 -50 cm), it may be possible to sample with shoulder length gloves and reach below the ice into the flowing water.

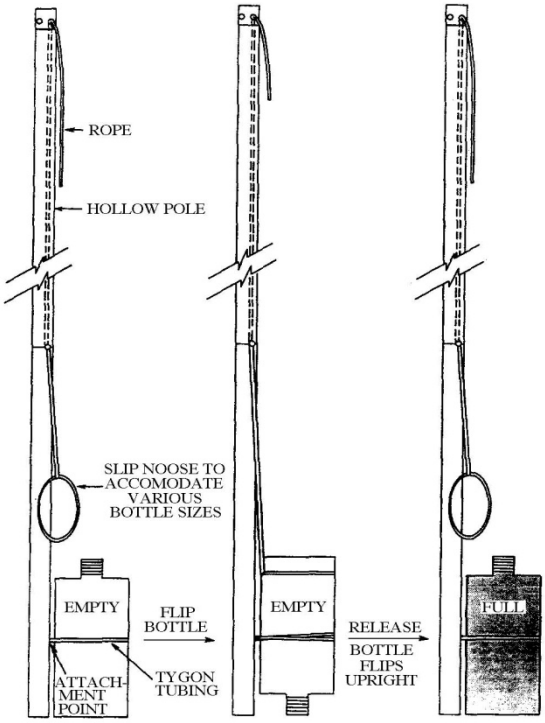

Figure 4. Flip sampler (Duncan sampler)

PROTOCOL

(Flip/Duncan sampler)

(a) Clear loose ice and snow from the sampling location, and drill through the ice with a hand or motorized auger. Keep the area around the hole clear of potential contamination (e.g., dirt, fuel, oil, etc.). **At least one member of the sampling team should be familiar with the operation and safety of both motorized and hand operated augers**.

(b) Remove all ice chips and slush from the hole, using a plastic sieve.

(c) Load a pre-labelled bottle upright into the bottle holder (tygon tubing) and rotate it so the mouth is facing down (Figure 4). Slip the noose over the bottom of the bottle.

(d) Hold the rope and pole at the top while you lower the sampler through the hole to the desired depth.

(e) Pull the rope to pivot the bottle so that the mouth faces upwards. Allow the bottle to fill and return it to the surface. Cap it immediately.

PROTOCOL

(from a bridge when ice is dangerous)

(a) When river ice is thin, a hole of sufficient size to collect a sample may be produced by dropping a weight attached to a hand line.

(b) Once the current has cleared the hole of debris, the protocol for sampling from a bridge (see section 4.2.2) should be followed.

Note: Extra care must be taken to avoid contamination in winter. De-icing agents such as salt can be easily transferred to the sample (particularly when working from a bridge).

5. Collecting Effluent and Receiving Water Samples

Effluent sampling has a particular series of protocols associated with it and this type of sampling is usually conducted by the waste discharge permittee. The conditions of sampling (frequency, site locations, etc.) are determined through consultation between Ministry of Water, Land and Air Protection and the permit holder. These conditions are then outlined in the permit itself. **The sampling site must conform to Workers' Compensation Board Regulations and other applicable safety requirements, and be readily accessible under all expected weather conditions**.

An overview of the types of sampling and flow measurement procedures are presented in "Field Criteria for Sampling Effluents and Receiving Waters" (Bollans, et. al., 1989). The following protocols outline the steps required to ensure that the samples that arrive at the laboratory are representative of the true conditions in both the effluent and the receiving waters.

Blanks, as discussed in section 3.2.1, also apply to effluent sampling programs.

Appendix 4 of this chapter lists the container size and type, and preservation technique required for the individual parameters.

5.1 Effluent Stream

The sample must always be collected at the same location within the effluent stream to ensure that each is representative. Representative sampling locations occur where the effluent is well mixed in the river or stream (i.e., typically near the centre of the effluent stream in order to avoid boundary effects and biasing due to material that has a strong tendency to sink or float). Grab samples are generally specified when the concentration of a parameter under consideration is not expected to vary significantly with time; or when values associated with extreme events are desired or when the analyte is such that the procedure of compositing would destroy the sample integrity or representativeness (VOC's, oil and grease) where the sample must be shipped for the lab in the original sample bottles. Composite samples are generally specified when the concentration of the parameter under consideration is expected to vary with time (or location). The individual samples that make up the composite may be of equal volume or be proportional to the flow at the time of sampling. The compositing period is defined according to the terms of the Permit (i.e., daily, over a four-hour period, etc.).

Note: When sampling effluents or receiving waters, the collector must wear protective gear (gloves, goggles, waders, etc.).

When variability in effluent flow rates exists, flow proportional composite sampling is a technique that must be used. In order to accomplish this, accurate (preferably continuous) flow measurements must be made. Automatic sampling devices (to collect grab or composite samples) are acceptable providing that the sample is in contact with only components made of acceptable materials (stainless steel, glass, plastic or Teflon). Plastic is acceptable except where samples are taken for organic analyses. **Automatic** sampling devices must be equipped with a purge mechanism to enable the sample line to be evacuated prior to sample extraction. The velocity in the sampling line should be a minimum of 0.75 m/s to prevent the settling of solid material.

PROTOCOL
(grab samples)

(a) Obtain a pre-labelled sample bottle and remove the lid without touching the inner surface of either.

(b) Grasp the bottle well below the neck and plunge it into the effluent. Ensure that your hand is always downstream of the bottle opening.

(c) Recap the bottle and place it in a cooler containing a sufficient quantity of ice packs (twice the volume of ice to sample in the summer, one to one in the winter).

(d) Once all the samples have been collected, process accordingly (see Appendix 3 of this chapter) and ship to the laboratory without delay (see section 8).

PROTOCOL
(composite sampling - flow proportional)

Note: Flow proportional composite sampling is necessary when effluent flow rates vary significantly (variations exceeding +15% of the daily mean more than 10% of the time) and will normally be specified as a condition of the Permit.

Follow the protocol outlined above for the actual acquisition of the sample. The only variable will be the quantity collected each time. The following is a hypothetical example of calculations for quantity collected:

If you are required to collect 1% of the effluent discharge (expressed per second) and the discharge is 10 L/sec then you would collect 100 mL. If the discharge doubles to 20 L/sec then in order to collect the required 1% you would have to collect a 200 mL sample.

It will be necessary to store component samples in an interim storage container over the prescribed composite period. This container must be made of acceptable materials, and the procedures for cleaning and re-use must conform to the protocols outlined in section 9. The sample must be kept cool (4°C) throughout the collection process.

Interim discrete samples should be preserved if required after they are taken, rather than waiting until the end of the composite period for adding preservative.

It is important to maintain a record or the volume and time of collection of the discrete subsample.

5.2 Receiving Waters

The sampling of receiving waters consists primarily of the same protocols and safety considerations as those discussed for ambient water sampling (see section 4). **The possibility of elevated levels of contaminants at some locations warrants further safety practices (see WHMIS and Workers' Compensation Board Regulations)**.

The ambient conditions at each effluent discharge location dictate which sites are ideal as sampling stations. These sites, for testing the impacts of effluents on the receiving waters, are determined through consultation with the permittee. They will include the following considerations:

- A control site (receiving water in a location not affected by the discharge);
- A site intended to monitor discharge impacts after complete mixing with the receiving water;
- A site intended to monitor outside a defined initial dilution zone.

Refer to Bollans et al., (1989) for a description of dilution zones.

Refer to Section 4 (Collecting Samples) for the protocols required for the acquisition of receiving water samples. Samples can be collected as either grab or composites. The rationale for composite sampling provided for effluents also applies to receiving waters. Receiving water flow variations are not usually significant over the sampling period; therefore, a flow proportional composite is not necessary.

6. Field Measurements

Field measurements involve the use of specialized equipment. Since different models are available for each variable, this section will discuss their use from a general perspective only. Field staff are directed to the reference documentation provided by the instrument manufacturers. An equipment logbook that documents instrument calibration, operation, and maintenance (yearly, at a service shop) records must be carried by the sampling staff at all times. This logbook must contain information about each instrument available to the sampling group.

All field data are to be recorded in the field logbook and entered into the database (e.g., EMS for Ministry of Water, Land and Air Protection) as soon as possible upon return from the field.

6.1 Temperature

Temperature can be measured with an alcohol-filled thermometer or with an electronic thermometer that has been calibrated against a certified thermometer. All thermometers must be checked against a reference thermometer by a laboratory before use and annually thereafter. Thermometers that do not meet the data quality objective of the project (e.g., ± 0.5°C of the true temperature) must be discarded.

PROTOCOL
(thermometer)

(a) Measure surface water temperatures directly in the water, allowing the thermometer to come to equilibrium before recording the value.

(b) For deep waters, collect a grab sample (e.g., with a Van Dorn - section 4.1.2.3) and decant some water into a 1 litre "field bottle" (never measure the temperature in a sample bottle that is being submitted to the laboratory for other analysis). Measure the temperature immediately, allowing the thermometer to come to equilibrium before recording the value.

Note: Ensure that the corresponding depth is identified for each temperature recorded in the field logbook.

PROTOCOL
(temperature using meters)

Note: Many meters have the capacity to measure temperature. Typically, though, temperature is measured with a combined temperature-dissolved oxygen meter. Temperature changes that occur with depth strongly influence the solubility of oxygen and therefore, the two need to be correlated (% saturation of dissolved oxygen).

(a) Calibrate the meter as per the operating instructions issued for each model.

(b) Check meter temperature readings, both in air and in water, against a thermometer of known accuracy as a quality control measure. If the measures do not agree, the meter can be adjusted to the thermometer reading. This check should be repeated throughout the day to determine if the meter is "wandering". All adjustments must be recorded in the field logbook. Temperature data are typically recorded to the nearest 0.5 degree.

(c) For depth profiles, record readings for increments of 1 - 2 metres. As a quality control measure, record the readings twice, once as the probe descends, and then again as it ascends.

6.2 Dissolved Oxygen (DO)

Dissolved oxygen can be measured by either chemical titration (Winkler method) or the membrane electrode method. Both have the potential of being accurate and reliable, but both methods require some training so that accurate measurements can be made. Meters provide a convenient and inexpensive way of measurement and are the most commonly used method. A well-calibrated oxygen meter membrane electrode system is preferred for obtaining a depth-profile of DO in a lake or deep river. Sampling for DO measurements requires particular care, since any contact between the sample and the air will modify the results. If percent saturation is to be determined, then the water temperature must be measured at the same time and location. Additionally, barometric pressure or altitude is required to determine percent saturation accurately.

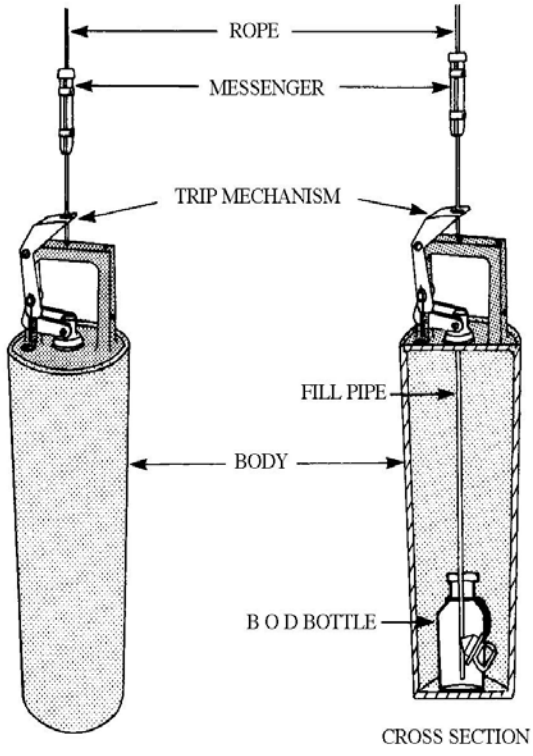

Figure 5. Dissolved Oxygen sampler

PROTOCOL

(Winkler method)

(a) If a DO sampler (Figure 5) is available the sample can be collected directly into a BOD (biochemical oxygen demand) bottle that is used for DO sampling. This sampler flushes 3 volumes of water through the bottle before it is filled (minimizing air-water contact). If this sampler is used, then proceed directly to step (c) after acquisition of the sample. Otherwise, a Van Dorn bottle can be used to collect water samples for DO analysis. In shallow waters (where a water-bottle sampler cannot be used), use a hand pump or a bucket with a clamped drain tube installed at the bottom.

(b) When the sample has been collected with a Van Dorn bottle or into a bucket, then transfer the sample to a 250 or 300 mL BOD bottle immediately. Allow the water to flow continuously through a delivery tube placed to the bottom of the bottle, taking care to prevent turbulence and bubble formation. Wait until at least 3 times the capacity of the sample bottle has overflowed before gently removing the tube (count the number of seconds for the bottle to fill initially, and then repeat twice).

(c) Immediately and gently add the flocculating agent (typically a pre-measured powder pillow containing manganous sulfate and alkali-iodide-azide, available from HACH*). Insert stopper, being sure that no air becomes trapped in the bottle. Mix vigorously by inversion. Allow the precipitate to settle and shake vigorously again. At this point analysis can be suspended for up to 8 hours (when samples from all sites can be processed at the same time). Care must be taken to ensure that the samples are not exposed to light during the interim. Place in a cooler for transport to shore or laboratory

* If pre-packaged chemicals are not available, directions for preparation of the chemicals are given in Standard Methods.

(d) Add 1 mL of concentrated sulfuric acid (H2SO4) with an automatic pipette by inserting the tip just below the surface of the sample. Carefully insert the stopper and shake the bottle until all of the precipitate has dissolved.

(e) Measure 100 mL of the sample with a volumetric pipette and then transfer to a 250 mL Erlenmeyer flask. Touch the tip of the pipette to the side of the flask during delivery.

(f) Titrate with 0.005M standardized sodium thiosulfate solution. Mix the sample during titration until a very pale yellow is observed.

(g) Add 2 drops of stabilized starch solution, mix to get a uniform blue color, and titrate carefully but rapidly to a colourless end point. Record the volume of the titrant used in mL to two decimal places.

(h) Calculate the concentration of dissolved oxygen in the sample as follows:

$$mgO_2/L = \frac{(mL\ titrant)\ (molarity\ of\ thiosulfate)\ (8000)}{(mL\ sample\ titrated)(mL\ of\ bottle - 2/mL\ of\ bottle)}$$

PROTOCOL

(DO meter - most common model YSI 57)

(a) Follow manufacturer's directions for storage, transportation, calibration, and use.

(b) Obtain DO readings for increments of 1 - 2 metres both during the descent and the ascent of the probe. Allow probe to equilibrate (a steady reading on the meter) at each depth before recording the value. When passing through a zone of rapid temperature or DO change (a lake thermocline for instance), two to five minutes may be required for equilibration.

Notes:

1. **When membrane function deteriorates, it should be changed to avoid contamination of the sensing element. Air bubbles must not be trapped under the membrane.**
2. **When measuring DO in lake hypolimnia, do not allow the probe to remain in waters of low DO (<0.5 mg/L) as the probe will become damaged.**

Use high sensitivity membranes where possible. Service meters annually. Meters should never be stored for long periods with batteries inside. Probes need cleaning too. Attach tag indicating service date and battery change date. Always carry spare parts, including batteries.

A simplified but thorough set of instructions for operating and calibrating a DO meter should accompany the meter - preferably laminated in plastic.

6.3 Conductivity/Salinity

Conductivity and salinity can be measured with a specific conductance meter or a multi-purpose meter (e.g., a Hydrolab).

PROTOCOL

(a) Follow instructions as per the manufacturer's directions for storage, transportation, and use. Check the accuracy of the meter against a conductivity standard.

(b) Obtain readings for increments of 1 - 2 metres both during the descent and the ascent of the probe. Allow probe to equilibrate at each depth before recording value.

(c) Check readings periodically by having water samples measured in a laboratory.

Notes:

1. **Conductivity is a numerical expression of the ability of matter to carry an electric current. If the matter is an aqueous solution the term conductance is synonymous with conductivity. Either term is correct.**

2. **Since the conductance of solutions changes with temperature, a correction is made (usually an internal automatic correction by the instrument) to estimate the conductance at 25 C, called the 'specific conductance.' Note that not all meters have temperature compensation. Also meters having temperature compensation can be damaged such that the temperature compensation is not working. Therefore instrument maintenance checks should include evaluation of the temperature compensation.**

6.4 pH

Either an electronic pH meter or a multi-purpose meter is used to measure pH. Most pH meters require that the sample be brought to the surface, while the Hydrolab can be lowered through the water column. This measurement is accurate for the current conditions only in a fresh sample. Rapid pH changes that occur as a result of gas diffusion, biological activity, and chemical reactions dictate that the measurements be performed immediately.

pH electrodes are available for specific measurement of pH in waters of low ionic strength and high ionic strength. It is imperative when measuring pH in water of low ionic strength that an electrode designed for measurement in solutions of low conductivity or dissolved solids be used. Caution should also be taken that the pH electrodes are functioning correctly - ones in long term use or storage can loose the internal electrolyte and provide inaccurate data. pH is a deceptively easy measurement to make but without understanding of how to use the equipment correctly, the risk of inaccurate data is very high.

PROTOCOL
(pH meter)

(a) Follow the pH meter manufacturer's instructions for storage and preparation of the electrodes.

(b) Remove electrodes from the storage solution and rinse with distilled water. Electrode fill plug, if present, should be removed before taking readings.

(c) In the field, calibrate the pH meter using two buffer solutions which will bracket the pH range of the samples [one at pH 7, one at acidic pH (4.0 or 5.0), or one at alkaline pH (8.0 or 9.0)]. Place the electrode in each solution for at least 1 minute (rinse well with distilled water between buffer solutions). If the reading does not correspond to the value of the buffer solution, adjust the meter and record the discrepancy in the field logbook. Repeat this process before the end of the sampling day. Samples should be at or near the temperature of the buffers used for calibration or the meter be equipped with a temperature compensation probe.

Note: Never calibrate with just a single buffer solution.

(d) Immerse the electrode directly into the surface water or into the field bottle (for samples collected from depth). Allow it to equilibrate before recording the value. Values are typically recorded to the nearest 0.1 pH unit.

(e) Check the field readings by having water samples measured periodically in a laboratory

PROTOCOL

(pH using a multi-purpose meter)

Note: These meters have automated internal calibration mechanisms that must be checked at time of overhaul maintenance, and the probes must be calibrated for each parameter.

(a) Follow instructions as per the manufacturer's directions for calibration, storage, transportation, and use.

(b) Obtain readings for increments of 1 - 2 metres both during the descent and the ascent of the probe. Allow probe to equilibrate at each depth before recording value.

6.5 Clarity

Water clarity in lakes is most commonly measured with a Secchi disc. The Secchi disc is a weighted disc, 20 cm in diameter, that is divided into black and white quadrants. The measurement is called the 'extinction depth'.

PROTOCOL

(a) Lower the Secchi disc over the shaded side of the boat.

(b) Record the depth at which the pattern of the disc is no longer observable. The disc should then be lowered beyond this depth to determine, when it ascends, the depth at which it reappears. Average the two depth readings to calculate the extinction depth.

(c) Record the value in the field logbook along with the weather and water surface conditions (e.g., cloudy, sunny, windy, surface chop, etc.). Measurements should be to the nearest 0.1 meter.

Note: Secchi disc readings should only be taken from 2 hours after dawn to 2 hours before dusk. During winter months, readings should only be taken between 10 A.M. and 2 P.M. Sunglasses should not be worn while taking the measurement.

6.6 ORP

Oxidation-Reduction potential (ORP) is most commonly measured with a multipurpose meter (e.g., Hydrolab).

PROTOCOL

(a) Follow instructions as per the manufacturer's directions for storage, transportation, calibration and use.

(b) Obtain readings for increments of 1 - 2 metres both during the descent and the ascent of the probe. Allow probe to equilibrate at each depth before recording value.

(c) As the meter approaches the lake bottom (use bathymetric maps or a depth sounder to assess depth), the readings may drop rapidly. At this point, take care that the probe does not contact the sediment.

6.7 Stream Flow

The most accurate measure of stream flow is achieved with a current meter used at multiple points along the cross section of the stream. However, simpler methods may be used if the flow estimates need only be approximate (cross-sectional area, a roughness factor, and floating object provide a very gross estimate of flow).

PROTOCOL
(current meter)

(a) Follow flow meter instructions as per the manufacturer's directions for storage, transportation, calibration, and use.

(b) Extend a measuring tape at right angles to the direction of flow and measure the width of the cross section. Record measurements on a data sheet. Leave the tape strung across the stream.

(c) Divide the width into segments using at least 20 points of measurement. If previous flow measurements have shown uniform depth and velocity, fewer points may be used. Smaller streams may also require fewer points. Measuring points should be closer together where depths or velocities are more variable. Cross sections with uniform depth and velocity can have equal spacing.

(d) Record the distance (from the initial starting bank) and the depth of each point.

(e) Record the current velocity at each measuring point.

Note: Horizontal and vertical variation of stream velocity may influence stream-flow measurements. To correct for vertical differences, hydrologists have determined depths that can yield acceptable estimates of the mean velocity over a vertical profile. If the depth exceeds 0.8 m, it is recommended that velocities be measured at 20 percent and 80 percent of full depth and averaged to estimate mean velocity. In the depth range 0.1-0.8 m, take the velocity at 60 percent of the full depth (measured from the surface) as an estimate of the mean over the profile.

(f) Calculate flow as a summation of flows in partial areas using the following equation:

$$\frac{q_n = v_n\, d_n\, (b_n+1 + b_n-1)}{2}$$

where:

q = discharge in partial area n [m^3/sec]
v = average current velocity in partial area n [m/sec]
d = mean depth of partial area n [m]
b_n+1 = distance from point to the following point [m]
b_n-1 = distance from point to the preceding point [m]

PROTOCOL
(floating object)

(a) Measure stream width (w in meters) and average depth (d in meters). Width is width of the water exclusive of dry stream bed. The average depth must be estimated, but is typically 0.4 - 0.6 of maximum depth (for shallow streams and deep streams respectively).

(b) Measure a three meter strip (l) along the stream bank that bisects the area measured in step (a) (very fast streams will require a strip longer than 3 m.) Choose a location where both flow and substrate are fairly uniform and representative of the stream reach. Curved areas should be avoided.

(c) Toss a floating object (e.g., cork, twig, etc.) into the flow upstream of the three meter measure area. Time the float as it travels the three meter segment. Repeat this step five times to obtain a mean of the time interval (t expressed in seconds). It is recommended that you re-measure until you get 3 measurements very nearly the same.

(d) Discharge is then calculated as follows:

$q = wdla/t$

where:

q = discharge (in m^3/second)
a = roughness coefficient [0.8 if rough (boulders), 0.9 if smooth (mud, sand)]

7. Field Filtration and Preservation

When the sampling objective is to determine concentrations of dissolved metals, low-level nutrients (e.g., phosphorus), or chlorophyll a in a water system, the sample must be filtered through a non-metallic 0.45 µm membrane immediately after collection. The guiding principle is to filter and preserve as soon as possible.

7.1 Filtration

The field filtration apparatus recommended is a portable vacuum system designed for ease of use in the field, thereby minimizing the time between sample collection and filtration (Figure 6). When filtering more than one sample, always filter the samples in the order of lowest expected variable levels to the highest. This minimizes the risk of cross-contamination between samples.

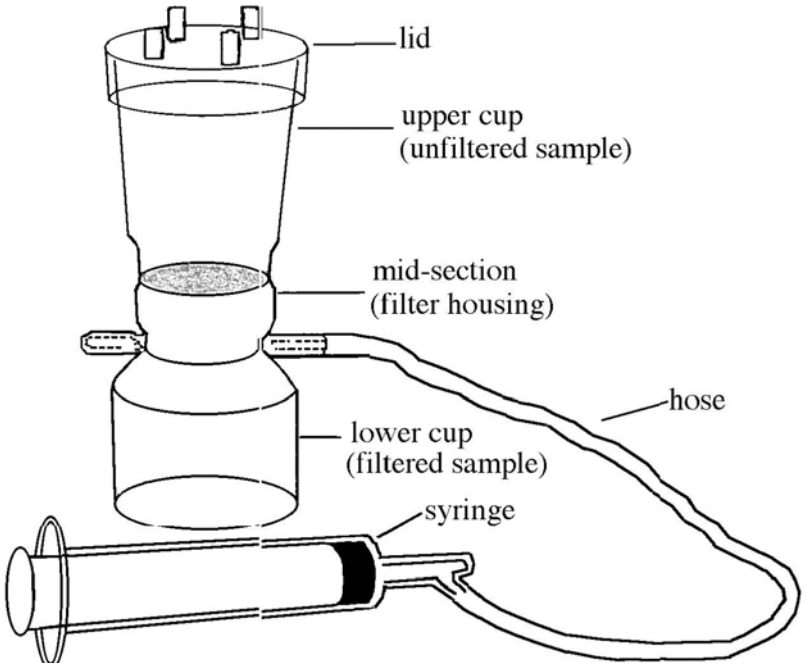

Figure 6. Filtration apparatus

PROTOCOL

(a) Rinse filtration apparatus with de-ionized water.

(b) With a pair of clean, non-metallic tweezers, place a filter paper on the surface of the mid-section of the filter apparatus. Assemble the apparatus as per Figure 6.

(c) Pour 250 mL of de-ionized water in the top section of the apparatus.

(d) Generate a partial vacuum by withdrawing the plunger of the syringe. Reject the initial filtrate (50 mL), and then filter the remaining water through to the lower section of the apparatus.

(e) Disassemble the apparatus and pour the filtrate into a labelled sample bottle. This is the first filter blank.

(f) Reassemble the apparatus and filter first sample (see instruction (d)) and pour the filtrate into a new labelled bottle. Always use standard amount of sample water (i.e., 250 mL) unless otherwise noted.

(g) Rinse the entire apparatus twice with de-ionized water and proceed to next sample. **Always rinse the apparatus thoroughly between sites**.

(h) At some point between samples (or after the last sample - if not filtering many samples), rinse the apparatus twice, change the filter paper, and filter 250 mL of de-ionized water. Transfer the filtrate to a labelled 'blank' bottle (e.g., 2nd filter blank or final filter blank).

Note: Other filtration techniques are also available and acceptable (e.g., Nalgene hand operated vacuum pump, disposable luer-lok syringes, etc.). Dedicate different sets of filtering apparatus for ambient, receiving water and effluent.

Note: The apparatus should be cleaned in a lab between field uses by soaking in dilute nitric acid solution followed by de-ionized water rinse and placing the dry and clean apparatus in a resealable bag (e.g., Zip Lock) for transportation.

7.2 Preservation

Many preservatives are considered hazardous materials and, therefore, the regulations outlined by WHMIS (Workplace Hazardous Materials Information System) must be adhered to. Read safety instructions and WHMIS material safety data sheets supplied for each preservative.

Deteriorated samples negate all the efforts and cost expended in obtaining representative samples. In general, the shorter the elapsed time between collection and analysis, the more reliable the analytical results.

Bulk dispensers for preservatives are not recommended due to the risk of contamination and deterioration over time. Preservatives should be pre-packaged in the laboratory in single-sample vials or ampoules to reduce the risk of contamination. Each of these ampoules should be labelled and have an expiry date beyond which they must be discarded in accordance with WHMIS regulations.

Note: Never use vials having Bakelite, or like material, as filler behind the cap liner of the lid.

Refer to Appendix 4 of this chapter for the quantity and type of preservative required for each individual analysis. Avoid pouring preservative down inside surface of sample bottle.

PROTOCOL

(a) **Before beginning, put on latex gloves and safety glasses or goggles.**

(b) Add preservatives to those samples that need preservation, being sure to match each preservative with its similarly labelled sample bottle. Preservative containers must not come in contact with the sample or inside of the sample bottle/lid. Minimize the length of time that the sample or preservative is exposed to the atmosphere.

(c) Recap sample bottles tightly and invert twice to mix.

(d) Recap the preservative bottles/vials tightly and place into a protective container. Ship these and latex gloves back to the lab with the samples for disposal.

Note: Consult WHMIS for recommended procedures for spill cleanup. Samplers should become familiar with WHMIS procedures before going into the field.

8. Shipping

The day's sampling schedule must be designed to ensure that the samples arrive at the shipping agency's terminal well before the end of business hours. Since some variables have very limited hold times (see Appendix 4), every effort must be made to avoid delays in shipping. The following is the procedure to be followed to maintain the integrity of the samples during transit.

PROTOCOL

Note: Ice packs should be used as opposed to loose ice or bagged ice. When loose ice melts, the contents of the cooler are free to shift, potentially allowing contamination of samples with melted ice water and/or breakage of glass bottles.

(a) Pack the samples upright in the cooler with at least 1 (winter) to 2 (spring, summer, fall) times as much ice as the total volume of the samples. Ensure that the samples that are most likely to deteriorate are closest to the ice packs (i.e., those that are not chemically preserved). Also, ensure that the glass bottles are separated from each other by ice packs, plastic bottles, or clean packing material to prevent them from shifting, falling over and/or breaking.

(b) Complete the laboratory requisition forms, enclose them in a sealed plastic bag, and then tape them to the inside lid of the cooler or place them in the cooler on top of the samples. The recommended minimum information that should accompany samples to the laboratory (on each requisition form) includes:

- Name of the source
- Site name
- EMS site numbers
- Date and time of collection
- Name of collector
- Field measurements
- Comments on sample appearance, weather conditions, and any other observations that may assist in interpreting water quality data

Additionally, a request should be made to the laboratory that they record the time and temperature of the samples at arrival (whenever samples requiring preservation by cooling to 4°C are shipped).

(c) Seal the cooler with heavy duty packing tape to reduce the possibility of it accidentally opening and to prevent tampering with the samples. Coolers arriving at the laboratory with torn or absent tape alert the lab staff that tampering might have occurred during transit.

(d) Attach a label prominently displaying the destination.

Note: If data on temperature on arrival is requested (to document that samples arrived at the laboratory at proper temperatures), a separate labelled bottle with water in it should be shipped in each cooler.

9. Cleaning Equipment

Equipment cleanness is an essential factor in ensuring that samples remain contaminant-free. All sampling devices (Van Dorn, multiple sampler, through ice sampler, tow nets, etc.) must be thoroughly cleaned and scrubbed with de-ionized water after each sampling trip. This process should be followed by two or three rinses with de-ionized water. The last rinsate should be collected and shipped for analysis as an equipment blank (see section 3.2.1.3).

Note: The Van Dorn sampler should be stored in the open position to prevent moisture from being trapped (might promote fungal or bacterial growth).

General cleanliness considerations include:

- Shipping containers (coolers) wiped free of dirt and rinsed with de-ionized water
- Vehicle neat and tidy
- Trailer, boat and motor free of aquatic plants before use on another body of water

The filtration apparatus must be soaked in an acid bath (10% HCl) and rinsed three times with de-ionized water. The final rinsate should be submitted periodically as an equipment blank.

Equipment used for ambient sampling should not be used for effluent sampling. Each type of sampling should have equipment dedicated to that use.

10. Sources of Further Information

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Appendix 1 Generic Field Checklist

(including water, sediments, biota and effluents)

General:

Logbooks____
Cooler (with ice packs) ____
Rope____
Camera (film) ____
Way bills____
De-ionized water (4L) ____
Resealable bags____
Pencils____
Felt Markers (waterproof) ____
Tape____
Requisition forms____
Shipping labels____
Squirt bottle____
maps____

Labelled Sample Bottles:

General chemistry (1 L) #____
Dissolved Metals #____
Total Organic Carbon #____
Coliforms #____
Zooplankton #____
Periphyton #____
Tissue cups #____
Extras - two of each
General chemistry (2 L) #____
Total Metals #____
Low-level nutrients #____
Sediments #____
Phytoplankton #____
Invertebrates #____
Macrophytes____

Sampling Equipment (clean, in working order, batteries charged):

DO Sampler (BOD bottle, Winkler reagents)
Thermometer____
pH meter____
Hydrolab____
Van Dorn, rope____
Auger (bit sharpened, skimmer) ____
Sediment grab____
Sieves____
Benthic invertebrate sampler (Hess, drift net, Surber) ____
Periphyton kit (cup, denture brush, baster) ____
Macrophyte sample kit (buckets, garbage bags, float tray, plant press, blot paper, herbarium Sheets, newsprint, corrugated cardboard) ____

Filtration and Preservation Equipment:

Filter Pots____
Tweezers____
Preservative Vials with acid____
70% ethanol____
Lugol's solution____
Syringe(s), Hose____
0.45/1.0 µ membrane filters____
Disposal Container (for used vials) ____
Formalin_____
Magnesium carbonate____

Boat Equipment:

Canoe (or boat) ____ Paddles____
Motor____ Fuel____
Life jackets____ Rope____
Anchor____ Tool kit____

Personal Gear:

Lunch____ Survival suit____
Rain gear____ Gum boots____
Waders (hip, chest) ____ Sun screen____

Safety:

WHMIS guidelines____ First Aid Kit____
Goggles (or safety glasses) ____ Rubber gloves____
Hard Hat (for industrial sites) ____

Appendix 2 Site Identification

Appendix 2.1 Site Identification Guide

Appendix 2.2 Site Identification Guide

Appendix 2.3 Site Identification Guide

Appendix 2.4 Site Identification Guide

Appendix 2.1 Site Identification Guide

Lake / river name _____

EMS site number _____ Latitude _____

Longitude _____ Map sheet number _____

Elevation _____

Access road names or numbers _____

NOTES: Distinguishing features

_____

_____

_____

Best access point to water

_____

_____

Photograph/Access Map

Appendix 2.2 Site Data Sheet (Lake)

EMS site number _____
Date _____
Time _____
Weather _____
Air temperature _____

Field Measurements:
Secchi depth _____

Depth (m)	**Temp**		**D.O.**		**pH**	**Cond**	**ORP**
	down	up	down	up			
0							
2							
4							
6							
8							
10							
12							
14							
16							
18							
20							
22							
24							
26							
28							
30 (or depths appr. to lake)							

Appendix 2.3 Site Data Sheet (River)

Site number _____

Date _____

Time _____

Weather _____

Air temperature _____

Field Measurements:

Water temperature _____

D.O. _____

pH _____

Conductivity _____

Flow / discharge _____

Stage (rising / falling) _____

Substrate type _____

Flow Data Measurements for Cross-Sections:

Appendix 2.4 Site Data Sheet (Effluent)

Site number_____

Date_____

Time_____

Weather_____

Site Observations

Effluent description_____

Site Observation_____

Maintenance/process considerations_____

Appendix 3 Sampling For The Most Common Variables

Appendix 3.1 General Chemistry (including nutrients)

Appendix 3.1 Metals

Appendix 3.1 Carbon

Appendix 3.1 Chlorophyll a

Appendix 3.1 General Chemistry (including nutrients)

3.1.1 General Chemistry (including acidity, alkalinity, chloride, colour, fluoride, hardness, nitrogen, pH, phosphorus, potassium, silica, sodium, specific conductance, sulfate and turbidity)

PROTOCOL

(a) Collect sample for all nutrients (as per sections 4 & 5) in a pre-labelled, plastic bottle (250mL to 2L depending on how many tests needed).

(b) Secure lid tightly and place in cooler with ice packs immediately.

(c) Do not field filter or preserve.

3.1.2 Low-level nutrients (phosphorus and nitrogen)

PROTOCOL

(a) Collect sample (as per sections 4 & 5) in a pre-cleaned (do not rinse), prelabelled 250 mL brown glass bottle.

(b) Field filter all low-level nutrient samples. Always return filtered sample to a new (clean) pre-labelled bottle.

(c) Secure lid tightly and place in cooler immediately.

(d) Do not field preserve.

Appendix 3.2 Metals

3.2.1 Total Metals

PROTOCOL

(a) Collect sample (as per sections 4 & 5) in a pre-cleaned (do not rinse), prelabelled 500 mL plastic bottle.

(b) Preserve the total metals samples with nitric acid (HNO3 provided by the analytical laboratory in individual ampules).

(c) Secure lid tightly and place in cooler immediately.

(d) Do not field filter.

3.2.2 Dissolved Metals

PROTOCOL

(a) Collect sample (as per sections 4 & 5) in a pre-cleaned (do not rinse), prelabelled 500 mL plastic bottle.

(b) Field filter all dissolved metal samples. Always transfer the filtered sample to a new (clean) pre-labelled bottle. Field filtration is a procedure where contamination often occurs. Extreme caution should be exercised.

(c) Once the sample has been filtered and transferred to a new bottle , then preserve with nitric acid (HNO3 provided by the analytical laboratory in individual ampules).

(d) Secure lid tightly and place in cooler immediately.

Appendix 3.3 Carbon

3.3.1 Total organic/inorganic carbon

PROTOCOL

(a) Collect sample (as per sections 4 & 5) in a pre-labelled 250 mL plastic bottle.

(b) Secure lid tightly, ensuring that no air is trapped in the bottle, and place in cooler with ice packs immediately.

(c) Do not field filter or preserve.

3.3.2 Dissolved organic/inorganic carbon

(a) Collect sample (as per sections 4 & 5) in a pre-labelled 250 mL plastic bottle.

(b) Field filter each dissolved carbon sample. Always transfer filtered sample to a new (clean) pre-labelled bottle.

(c) Secure lid tightly, ensuring that no air is trapped in the bottle, and place in cooler with ice packs immediately.

(d) Do not field preserve.

Appendix 3.4 Chlorophyll a

PROTOCOL

(a) Collect sample (as per section 4) into a pre-labelled plastic bottle.

(b) Secure lid tightly and immediately place in cooler with ice packs.

(c) When all samples for the day are collected, filter (using a .45 micron membrane filter) an appropriate portion of the chlorophyll a sample. This can be done in the field or in the lab within a few hours of collection if the samples are kept dark and cool. The filtration should be done in cool temperature and subdued light (not on the tailgate at the boat ramp!). The amount of sample filtered depends on the density of the algae present (productive lakes may require only 50 mL, unproductive lakes may require 1 L to be filtered). **Always record the volume of sample that was filtered (both in the field logbook and on the Laboratory Requisition Form)**.

(d) As the water sample is filtered, observe the filtration pressure or vacuum (<5psi) and the water level. When all but the last few mLs of water are drawn through the filter, rinse the top holding cup with de-ionized water and continue to filter. Before the rinse water is fully filtered, add 2-3 drops of MgCO3 suspension (1g magnesium carbonate / 100 mL de-ionized water) and gently swirl the apparatus to distribute the MgCO3. Magnesium carbonate is a buffer to stabilize the pH of the algal cells above 7. The cells are very sensitive to acid pH as the chlorophyll will then be degraded to other pigments like phaeophytins.

(e) With clean tweezers, carefully remove the filter and place it in the center of a larger (9 cm) 'Whatman' filter paper. Fold the two papers in half and then in half again (with the smaller filter paper inside the larger). Secure the filter papers shut with a plastic paper clip. With a **pencil**, label the 'Whatman' filter paper as a chlorophyll sample. Also, for each sample, identify the date, site number and the volume of water filtered directly onto the 'Whatman' filter paper.

Note: Some brands of filter papers have throw-away plastic separators. On occasion, it has happened that people have confused these plastic separators with membrane filters

separated by throw-away paper. Be sure you know which is the filter and which is the throw-away!

(f) Place the filter paper in a pre-cooled dark bottle (amber glass, wrapped with aluminum foil and black tape - chlorophyll is very sensitive to degradation by light) that contains a desiccating agent (i.e., silica gel).

Note: Silica gel will take up water until it is saturated, at which point it must be rejuvenated by heating it in an oven for several hours. Ordinary silica gel is white, whether fresh or saturated. However, dye is often added to warn you when the gel has been saturated. Usually fresh silica gel is blue and completely saturated gel is pink. Partially saturated gel is both blue and pink (i.e., purple). Note that some brands of silica gel use other colours so be sure what color change you should expect. This is readily done by wetting a gel crystal to check the colour for saturated silica gel. Never use saturated silica gel.

Two common errors by untrained staff are to use saturated gel, or to attach the gel outside the bottle.

(g) Pack the bottle containing all chlorophyll a samples in a cooler with ice packs (or dry ice) so that they remain frozen until they reach the analyzing laboratory.

(h) Filters stored inside a dark bottle with desiccant can be stored in a deep freeze for a week or two but it is far preferable to ship them to the lab immediately.

Appendix 4 Sample Container, Preservation, and Hold Times for Water and Effluent Samples

TYPE OF ANALYSIS	MINIMUM SIZE	CONTAINER TYPE	PRESERVATION	MAXIMUM HOLD TIME
WATER - INORGANIC ANALYSIS				
General chemistry and Anions	1 to 4 L	P	keep cool, 4°C	72 h
Mercury, Total	1 L or 500mL	G, A, R	6 mL 10% K2Cr2O7+6 mL H2SO4 per L	28 d
Metals, Dissolved	250 mL	P, R	field filter & pres HNO3 (to pH2)*	6 mo
Metals, Total	250 mL	P, R	HNO3 (to pH2)	6 mo
Carbon TIC/TOC, Inorg/Org	100 mL	P or G	4°C	72 h
Biochemical Oxygen Demand, BOD	1 L	P	4°C, exclude air	72h
Chemical Oxygen Demand, COD	250 mL	P	0.2 mL H2SO4/250mL	72 h
Cyanide, SAD and/or WAD	1 L	P	NaOH (to pH12)	72 h
Oil & Grease	1 L	G	HCl (to pH2)	28 d
Phenolics, Total	1 L	G, A	H3PO4 to pH 4 + 5mL 20% CuSO4	28 d
Phosphorus, Low-level	100 or 250	G, A, R	keep cool, 4°C	72 h
Sulfide, Total	500 mL	P or G	1 mL 2N Zinc Acetate, exclude air	72 h
WATER - ORGANIC ANALYSIS				
AOX (Adsorbable Organic Halides)	500 mL	G, A, R, B	HNO3 (to pH 2)	30 d
Chlorophenols PCP, TTCP, TCP	1 L	G, A, Solv	4°C	30 d
Dioxins / Furans	3 x 1 L	G, A, Solv	4°C	30 d
EPA 624, Volatiles or BTEX	3 x 40 mL	vial, G, B, P&T	Headspace-free, 4°C	14 d
EPA 625, CP/OC/PAH/PCB	1 L	G, A, Solv	4°C	30 d
AEH, TCMTB	1 L/analysis	G, A, Solv	4°C	30 d
Hydrocarbons	500 mL	G, A, Solv	4°C	30 d
Copper quinolate	250, 500 mL	G, Solv, Fc	4°C, HCl (to pH 2)	30 d
Resin Acids	1 L	G, A, Solv	4°C, NAOH (to pH 12)	21 d
Trihalomethanes	500 mL	G, A, Solv	Na2S203, headspace-free, 4°C	14 d
IPBC / DDAC	1 L	P or G	4°C, 6N HCI, 2mL/L	28 d

ANALYSIS WITH LIMITED SHELF LIFE	
pH, Turbidity, Acidity, Alkalinity	72 hr
Ammonia, TKN, Nitrate, Nitrite	72hr
P ortho, total, total dissolved	72hr
Specific Conductance	72hr

LEGEND	
P = plastic	Ster = sterilized
G=glass	Solv = solvent cleaner
A=Amber	Fc = foil-lined cup
W=wide mouth	R = acid rinsed
T=Tissue Cup	B = Baked

Note: The preservation acids/bases specify a pH endpoint (pH ²2 or pH³12). The appropriate amount of preservative for a set of samples should be determined by titration on water samples collected specifically for that purpose. The amount of preservative needed should never be arrived at by titrating and measuring the pH of the actual sample!!! All preservatives should be high purity, lab approved materials.

The preservatives used should be supplied from the analytical lab in ampules. The lab will verify their purity and provide an expiration date, beyond which they should not be used.

Note: These are the preservation and hold times for the present (1995) contract laboratory for the Ministry. Different labs, organizations and protocols may differ, as may future laboratory procedures.

Appendix 5 Effluent Sampling Checklist Guide

Location _____

Sample Point _____

Date _____

NOTE: Ensure sampling point is agreeable to MWLAP and Permittee for determining compliance and in compliance to WCB regulations and safety requirements. Take samples for what is allowed by Permit, or for what is requested. All sampling bottles should be clearly labelled and dated; and shipped to the designated lab immediately.

TEST PARAMETERS OF SAMPLE

Test Parameter	Sample Frequency and Type	Allowable Level

A. SAMPLE POINT	**YES**	**NO**
Is the sample point:	____	____
1. Accessible under all weather and tide conditions?	____	____
2. Near the centre of the stream?	____	____
3. In a turbulent mixing zone (immediately downstream from a flow disturbance such as a pipe constriction, bend or flow control device)? (describe disturbance in Comments)	____	____
4. At least 6 pipe diameters downstream where two separate pipe streams combine (point of confluence)?	____	____

Comments:

_____

_____

_____

Effluent Sampling Checklist

B. TYPE OF SAMPLE	**YES**	**NO**
1. Grab Sample		
a) Does permit allow grab sample?	____	____
b) Is volume collected ≥ 1 litre?	____	____
c) Is collection time ≤ 15 minutes?	____	____
2. Composite Sample		
a) Does permit allow composite sample?	____	____
b) Compositing period:	____	____
c) Is sampling frequency > 4x / hour?	____	____
d) Are individual grabs of equal volume?	____	____
e) Is flow variation less than ± 15% of daily mean more than 10% of the time?	____	____
f) Is flow proportional sampling performed?	____	____
3. Automatic Sampling Device (Grab or Composite)		
a) Type: _____ _____	____	____
b) Is the automatic sampling device equipped with a purge mechanism?	____	____
c) Is the velocity in the sampling line at least 0.75 m/s? (velocity = $4f/\pi d2$; f = sample flow; d = sample line diameter)	____	____
d) Do the components of the sampling device consist of acceptable materials for the parameter being sampled? (plastic for BOD and TSS analysis)	____	____
4. Continuous Sampling		
Parameters sampled: _____ _____		
5. Split Sampling		
a) Is the sample splitting device appropriate?	____	____
b) Has it been approved?	____	____
c) Was the splitter cleaned, as prescribed, prior to use?	____	____
d) Was the entire sample directed through splitter?	____	____

Comments:

_____
_____
_____

Effluent Sampling Checklist

C. SAMPLER DESIGN AND OPERATION	YES	NO
1. Are sample bottles lab pre-cleaned?	____	____
2. Are components of storage container compatible with effluent parameter to be tested? (e.g. plastic for BOD, TSS; glass for oil)	____	____
3. Are all parts that come in contact with effluent cleaned regularly?	____	____
4. Are buckets, storage vessels, etc. that are reused	____	____
a) rinsed 3 times with de-ionised water (W) or sample (S)? (if YES, specify whether (W) or (S) was used)	____	____
b) rinsed 3 times with acetone and once with de-ionised water, if organic parameters are sampled?	____	____

Comments:

_____

_____

D. SAMPLE PRESERVATION AND STORAGE		
1. Is sample immediately cooled to 4°C (± 2°C), if required?	____	____
2. Is elapsed time for testing sensitive parameters ≤ 48 hours? (elapsed time for composite sample begins with the last sample collected)	____	____
3. Is sample filtered prior to preservation (dissolved metals and nutrients)	____	____
4. Is preservation required?	____	____
5. Are blanks submitted with samples?	____	____
6. Parameters to be analysed:	____	____

Comments:

_____

_____