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THE INSPECTOR'S FIELD SAMPLING MANUAL

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1.1 INTRODUCTION

1.1.1 ACKNOWLEDGEMENT

The 2nd edition of this manual continues the requirements of Environment Canada to achieve consistency and uniformity, while establishing national procedures for field sampling practices. In this new edition we address advancements in technology, methodology and updates to regulations and acts.

We hope the updated information will assist field sampling personnel; for training new employees and as a reference for experienced staff. Our intention is for this manual to help in promoting best practices and safe sampling procedures.

The national advisory committee included: John Holmes, Richard Strub (Pacific & Yukon Region); Kimberly Lam, Tim Lambert, David Noseworthy (Prairie & North Region); Carl Morden (Ontario Region); Bruno Lafortune (Quebec Region); Ron Hunter (Atlantic Region) and Gord Thompson. I would like to thank the committee and others who helped bring this manual to fruition. – Richard Strub, Project Coordinator, Richard.Strub@ec.gc.ca

1.1.2 LIABILITY AND LIMITATIONS

This manual is intended to provide guidance regarding recommended routine and legal sampling practices. The manual does not cover all situations. In some cases you may be required to amend these protocols to adapt to unique situations. Samplers

may also be required to consult subject matter experts for specific information. Where any conflict exists between the information in this manual and any relevant legislation or regulations, the latter shall take precedence.

DISCLAIMER: Throughout this document the use or reference to trade names, companies or trade marks is provided as examples and does not constitute endorsement of the products by Environment Canada.

1.1.3 INTRODUCTION

This manual has been written to provide national standards and uniformity in environmental sampling for samplers including enforcement officers, other public officers and lay samplers. Although some specific warnings on hazards involved in sampling procedures are included in this manual, inspectors should also familiarize themselves with **THE INSPECTOR'S SAFETY GUIDE** and the task hazard analysis (THA's) for specific tasks related to inspection work.

This manual draws on established procedures and protocols, legislative and regulatory requirements, scientific literature and the experience of Environment Canada inspectors, investigators and laboratory staff across Canada. Environmental sampling is a complex field and technology is evolving rapidly, you should stay aware of changes to legislation, regulations, methods and standard practices, as they occur.

Sampling can be undertaken for various reasons, including inspection of regulated facilities, investigation of suspected violations, routine monitoring, research projects and emergency response. This manual focuses on sampling for inspections, investigations and emergency response. The manual is divided into four parts, appendices and an index:

PART I: SAMPLE PLANNING AND DOCUMENTATION outlines practical information regarding how to prepare for sampling, including what information you should reference for planning your field trip and what to include when creating proper planning and field documentation. The differences between compliance and legal sampling are outlined here, including the type of equipment required for both types of sampling. Also covered is choosing a sampling site, taking quality assurance/quality control (QA/QC) samples and dealing with contamination issues.

PART 2: FIELD SAMPLING TESTS AND EQUIPMENT PROTOCOLS provides information on how to conduct field testing for temperature, pH, clarity, etc. Emergency response information, sampling spills and working with chemical categorization kits are discussed here. Sampling equipment protocols are covered to help you decide on the best tool for your sampling.

PART 3: SAMPLING MEDIA BACKGROUND deals with specific techniques and applications for sampling liquids, solids, gases, biota and hazardous wastes.

PART 4: SAMPLING PROTOCOLS provides new and updated information on sampling protocols for specific analytes, including an easy to use chart on container types, preservation techniques and holding times. Also included is a list of industry specific tests.

PART 5: APPENDICES have information on commonly used acronyms, formulas and abbreviations; website addresses for regulations; a summary of health and safety legislation; a field equipment checklist; laboratory contact numbers; flow calculations and regulatory tables. There is also a list of references for research used in this manual, plus an index.

1.1.4 PREPARATION

Thorough planning is important to ensure success in your sampling. The goal of any sampling program is to produce a set of samples representative of the source under investigation and suitable for analysis to identify the compound(s) in question.

There may be more than one objective when planning your sampling; the primary objective may be an emergency response to a spill, while a secondary objective may be a subsequent legal investigation.

You will be able to meet both objectives through preparation of a detailed sampling plan. It is important to research existing information sources before the start of a sampling trip to ensure that you understand the sampling protocols required for the permit or situation. You should also plan that you have enough sampling containers, the correct tools and you should be aware of required safety precautions. Be sure to understand what needs to be documented both BEFORE, DURING and AFTER conducting your sampling to ensure you have fulfilled all requirements.

PRELIMINARY ASSESSMENT

If you are able to conduct a preliminary site visit you will be able to more fully understand the sampling requirements. Your site reconnaissance can assist you in identifying what equipment you will need, what safety and hazard considerations to plan for and what your technical approach to site sampling will consist of.

After reading this manual, you should have the ability to answer the questions in each of the three stages:

SAMPLING PREPARATION

Where you are going?

Do you need assistance or a search warrant to conduct your work?

Do you have background information on the location or facility you are visiting?

Do you have all required safety equipment and information (see **THE INSPECTOR'S SAFETY GUIDE**)?

Are you familiar with which regulations apply and what are the requirements?

Do you have experience in all required techniques available?

What are the analytes you will be sampling for?

Do you know what sample size will be needed?

Have you talked to the laboratory for consultation?

What sampling containers and equipment will you need?

What are the quality requirements for data quality objectives, as well as for QA/QC?

How many sites will be sampled and do you have QA blanks for each site (travel, field and equipment blanks)?

Do you have complete chemical categorization kits and strips?

If this is a legal sample, do you have chain-of-custody forms, a lockable tool box, numbered seals, plastic bags and a cooler with a locking device?

Have you tested your equipment prior to going into the field?

Do you have the ability to clean equipment in the field?

Have you checked expiration dates on kits, containers, equipment, etc.?

Have you included all relevant information in your sample planning documentation?

SAMPLING

What approach will you use for your sampling (judgmental, systematic or random)?

Have you documented your sampling approach and which sampling protocols you will use?

Are you aware of the correct container choice and holding time for the analyte ?

Are you prepared for any safety requirements you may have (see **THE INSPECTOR'S SAFETY GUIDE**); do you have the correct personal protective equipment and other safety equipment?

Have you ensured that you will not introduce contamination to the samples?

Have you conducted your QC with your required travel, field and equipment blanks?

Do you have the correct preservatives with you?

Has your equipment been calibrated according to the manufacturer's guidelines?

Do you have extra batteries?

Is your camera or video camera working correctly?

Do you have the correct containers lids and seals (which will not contaminate the sample)?

Have you labeled and sealed each sample?

Have you photographed the sampling location?

Do you know who will be signing for your samples at the lab; is their name listed as the receiver on the shipping information?

Have you recorded all observation and labeling information into the notebook?

SAMPLE DELIVERY

Do you have all your shipping material?

Have you completed all forms?

Are all containers labeled and sealed correctly?

Are you sure you have used the correct containers, tools and lids for the analyte in question (for example for organic parameters, Teflon tubing should be used)?

For legal samples, have you filled out the chain-of-custody form?

For legal samples, have you correctly sealed the samples, tool boxes and coolers?

If you are sending samples by courier, have you kept a copy of the waybill?

Will your samples be delivered to the lab within the correct holding times for the analyte in question?

Have you contacted the lab to let them know what to expect or to check that your samples have arrived?

1.1.5 INFORMATION SOURCES

There are a number of information sources to use in gathering data:

- compliance records
- other existing records
- applicable regulations, guidelines and codes of practice
- standard reference methods (SRMs) and scientific literature
- newspaper files
- other government agencies
- municipal archives
- hydrographic surveys
- harbour commission records
- past surveys
- topographical maps and charts
- similar circumstances in-house or at other agencies
- health and safety legislation

REGULATIONS, GUIDELINES AND CODES OF PRACTICE

Prior to sampling, you should review and understand the applicable legislation, including regulations, interim orders, guidelines, codes of practice and other legal or regulatory documents. For Environment Canada, the two principal pieces of legislation are the Canadian Environmental Protection Act 1999 (CEPA 1999) and the Fisheries Act. Some regulations and guidelines include specific sampling procedures or standard reference methods (SRMs) that must be followed. A list of current regulations under CEPA 1999 is found on the CEPA Registry (www.ec.gc.ca/ceparegistry/) and a list of current regulations under the Fisheries Act is found on the national enforcement web page (www.ec.gc.ca/enviroregs).

1.1.6 CONSULTATION

One of the most important aspects of the planning process is the joint involvement of the data users; the samplers and the laboratory analysts. Each group should be involved from the outset, because each has a critical role in defining data quality requirements. Laboratory analysts must understand the final objectives and ultimate goals of the sampling and analysis.

Understanding the principles of analytical methods is also important in the planning process, since methods can strongly influence the sampling protocols. For example, the sensitivity and type of an analytical method, the desired detection limits and similar considerations may directly influence the volume and type of sample to be taken. Analytical methods will also affect the choice of storage containers and preservation techniques.

One essential sampling decision that must be resolved by consultation during the planning stage and documented is if the sampling protocol involves Quality Control (QC) sampling. How many and what types of QC samples will you need to take? The answer will depend on the sensitivity of the test in question, the conditions of the sampling trip (for example, how widely separated sampling sites are) and the laboratory's requirements.

1.1.7 LABORATORY CERTIFICATION

When choosing a laboratory, samplers should check that the laboratory meets an international accreditation program (ISO/IEC 17025). Department of Environment (DOE) requires their laboratories and contract laboratories to have appropriate accreditation.

Formal recognition of the competence of the laboratory to carry out specific tests requires ongoing demonstration of performance through proficiency testing and bi-annual laboratory audits to maintain capabilities.

1.1.8 LEGAL OR ROUTINE

Submitters who are planning to collect samples for analysis at a laboratory should be aware of the following:

- proper labels, traceable clean containers and lids
- follow sampling and handling protocols (preservation, holding time, transport, etc.)
- QA/QC (blanks, replicates, etc.)
- documentation
- consultation with lab

The distinction between legal sampling and routine sampling is the ability to prove in court the chain-of-custody of a sample. This is documentation showing how evidence was collected, who collected it, where it was collected and who has had custody of the evidence during all stages from collection to laboratory analysis to return of the sample to the submitter.

1.2 PLANNING DOCUMENTATION

1.2.1 SAMPLING PLAN DOCUMENTATION

Your documentation is an important record of both your planning and field work. Keeping detailed notes will enable you to have a successful sampling trip, as well as provide much needed information should your sampling information be used in other enforcement activities or in a court case. In the event that a case proceeds to prosecution your raw notes, documents, notebooks and report will be used in preparation of the court brief and may be entered as evidence into legal proceedings.

The following examples of a sampling plan will serve as a guide to show you where detailed reference information can be found in this manual. Not all elements of the sampling plan may be required for every field sampling trip and you will also want to combine this example with the existing templates used in your office.

Creating a detailed sampling plan will leave no doubt as to why you have chosen a particular approach. You will also ensure you have a documented systematic approach and should you need to testify in court, your documentation will assist you in recalling important details.

SAMPLING PLAN

CATEGORY	FURTHER DETAILS	MANUAL REFERENCE
Title (short, but easily indexed)		
List of references	A summary of background information on the site.	Part 1.1.5: Information Sources
Introduction and objectives	Describe issue and purpose of sampling. Typical goals include identification of type and concentrations of substance(s) of concern and verification of substance migration.	
Scope	The breadth of coverage of a sampling plan.	
Sample matrix and sampling site	Investigation methods required to characterize the site. Often this includes sample types, sampling location and field quality control.	Part 1.3: Site Selection and Documentation Part 3: Sampling Media Background
Method of sampling	Sampling protocols.	Part 4: Sampling Protocols and procedures.
Administrative arrangements - health, safety and security provisions	Personnel requirements.	Appendices THE INSPECTOR'S SAFETY GUIDE
Preliminary site inspection		
Sampling equipment	List of standard and non-standard equipment.	Part 2: Field Sampling Tests and Equipment Protocols
Sample containers, preservatives, holding times and shipping	List of container needed.	Part 4: Sampling Protocols
Additional information	This might include methods used to control contaminated materials including decontamination procedures, solutions to be used and storage or disposal obligations.	
Coordinate with laboratories	Use a certified lab and ensure you understand their needs for holding times, etc.	Part 1.1.7: Laboratory Certification Part 4: Sampling Protocols
Other	Is special training required, such as in the case of some microbacterial sampling or will you need other contract services to complete the investigation. Finally, notify your office and leave word on how you can be contacted in the field.	

1.2.2 DOCUMENTING SAMPLING PROTOCOLS

Sampling protocols are pre-established written descriptions of the procedures to be followed in collecting, packaging, labeling, preserving, transporting and storing samples. Protocols are essential in achieving consistency at every step and for reducing the chance of error or erroneous assumptions.

Proper documentation of sampling protocols is an essential component of the field quality assurance program. The overall sampling protocol should identify sampling locations and include all of the equipment and other information needed for sampling. Proper protocol documentation may include:

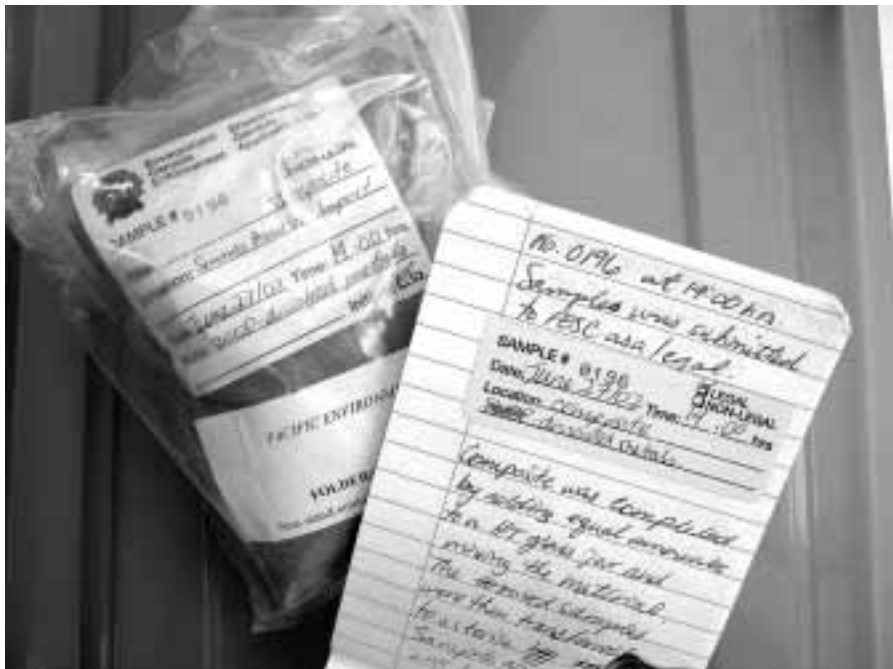
- sample type (e.g., water, sediment, biota, air)
- number of samples and volume
- size of sample containers
- seals, labels, field logs
- types of sampling devices
- numbers and types of QA/QC samples, including blanks, splits, etc.
- method used to collect samples
- specific preservation instructions for each sample type and parameter in question
- chain-of-custody procedures for legal samples
- transportation plans
- any field preparations (e.g., filtering)
- field measurements such as pH, dissolved oxygen, temperature, etc.
- field conditions (e.g., physical, meteorological, hydrological)

Digital photography is helpful in assisting the documentation process – however it should not be used to the exclusion of written documentation.

Sample protocol documentation should be saved for future reference; you may need to consult these in court or they may be useful for planning future sampling trips.

1.3 SITE SELECTION AND DOCUMENTATION

1.3.1 SITE SELECTION



The selection of sampling sites is one of the most critical steps in any monitoring or surveillance planning. Selecting a representative sampling site will depend on the sampling objectives and the analyte of interest. Some of the factors and concerns to consider are:

- Are regulatory requirements a factor?
- What volume of sample is required?
- Does substance mix with water, float on it or sink?
- Should you collect a grab or composite sample?
- Do you use an automatic sampler or collect samples manually?
- At what depth will you collect the samples?

- Is access a problem?
- Will it be easy to re-sample if required?
- Can you find the location again?

The selection of sampling method will depend on the size of the study site and the desired degree of statistical certainty and accuracy.

The sampling points are often pre-determined for compliance monitoring. In situations such as spills or specialized surveys, sampling locations must be chosen to reflect the objectives of the sampling program. In the case of a spill, the primary objective may be to collect sufficient material for identification in the lab, while a secondary objective may be to assess the possible impact on aquatic organisms.

You should be knowledgeable about conditions on-site prior to sampling. When possible, physically inspect the site before taking any samples, or research any available records such as file information or previous reports. Consult with others who may know the site. In some cases, aerial reconnaissance or satellite imagery may help in targeting sampling sites.

Ensure your sampling objective matches your strategy for choosing sampling points, for example:

SAMPLING OBJECTIVE: to find unknown sources of known contamination.

STRATEGY: set up a grid (regularly spaced parallel or intersecting bars) and take samples at regular intervals (consistently sample at axis, or center point in grid segments). If the area is large, consider doing a large-scale grid first, followed by a smaller-scale grid once the target area has been identified.

SAMPLING OBJECTIVE: to determine the extent of contamination from an industrial outfall in a river.

STRATEGY: assume that concentration of the contaminant will decrease with increasing distance from the source; also consider factors affecting dispersion of materials from the point source (e.g., current). Select sampling stations at fixed distances downstream, following a geometric progression (i.e., distance = x , $2x$, $4x$, $8x$).

SAMPLING OBJECTIVE: to determine contamination from a sunken ship.

STRATEGY: locate sampling stations in concentric rings at fixed distances and specific compass angles (e.g., 45° or 90°).

1.3.2 SAMPLING APPROACHES



There are three primary approaches in sampling: random, systematic (or stratified) and judgmental. It may be useful to use two of these three approaches in combination.

RANDOM SAMPLING relies on the theory of random chance to choose representative samples. This process is useful when numerous sampling locations are available but there are no particular reasons for choosing one over another.

For sampling a dump site involving many drums, separate the drums into groups according to their content, if known. Sample from each group randomly.

Random sampling is also often used for sampling lagoons, ponds and other surface waters. Here, the area of concern is divided into a two- or three-dimensional grid and the sampling points are chosen randomly.

SYSTEMATIC SAMPLING involves taking samples over regular distances or intervals – horizontal or vertical.

In horizontal sampling, shellfish samples are taken at 1 km intervals along a shore. In vertical sampling water samples are taken from varying depths in the water column.

Systematic samples taken at regular time intervals can be used for geostatistical data analysis, to produce site maps showing analyte locations and concentrations.

Geostatistical data analysis is a repetitive process, showing how patterns of analytes change or remain stable over distances or time spans.

JUDGMENT SAMPLING is often the method of choice for regulatory and emergency response sampling. Determining locations that will provide the most representative samples requires knowledge of the distribution of the parameter(s) in question; sample validity will depend on the accuracy of this knowledge. Because the validity of judgment sampling can be questioned in court, inspectors should always document

their reasons for choosing a particular sampling location. Field measurements, such as pH, conductivity or temperature, can also be used in selecting the most appropriate sample location.

SAMPLING APPROACHES TO SITE SELECTION

APPROACHES	AMOUNT SAMPLES	RELATIVE BIAS	BASIS OF SELECTING SAMPLING SITES
Random	Largest	Smallest	Simple random selection
Systematic	Larger	Smaller	Consistent grid or pattern
Judgmental	Smallest	Largest	Prior history, visual assessment of technical judgment

1.3.3 CONTROL SITE SELECTION

In addition to samples substance(s) of interest, control site or background samples may be required to verify the source of contamination, eliminate other possible sources or to determine background concentrations. Control sites are used for determining if the analyte of interest is present in the test samples but absent in the control sample. These should be taken upstream or upwind from a point source. In some situations, it may be best to take samples from a separate but similar area. Sampling points must be chosen to avoid possible contamination, avoid sampling downstream from a bridge or boat motor. There are two types of control sites:

LOCAL CONTROL SITE(S) are near sample site, usually far enough upstream of the source of contamination for running water or far enough distance from pollution source for stationary water.

AREA CONTROL SITE(S) are town or watershed not adjacent to sampling site. Use only as background samples when local control site is not available.

PROTOCOL

1. Select site(s) with common characteristics with the affected area, except for the pollution source.
2. Always take local control site samples first if available before sampling effluent or spill site(s) to avoid contamination.
3. Minimize travel between control site and sampling site to avoid contamination.

1.3.4 METHODS FOR FIXING POSITION

GLOBAL POSITIONING SYSTEMS

Use global positioning system (GPS) to identify the location of the sampling station in terms of latitude and longitude, so that the user can return to the same position, including:

- locate old mine sites during environmental audits
- mark the location of sampling sites on or near streams, rivers, outfall areas, estuaries, and shorelines
- mark affected shorelines during spills or to record the location of oil spills off shore
- estimate distances, elevation, gradients and areas in the field

Most GPSs are now accurate to within 3 m – 5 m. Many systems offer multiple station locations, data recording, way marking and navigation capabilities.



GPSs receive signals transmitted from three or more orbiting satellites and then calculate their position relative to the satellites. The accuracy of the calculation is determined by the number and position of the satellites used. Locations with a restricted view of the sky will have reduced accuracies. Hand held receivers commonly report the number and position of the satellites they are receiving along with the accuracy of their reading.

1.3.5 SITE DOCUMENTATION AND NOTEBOOK

Proper site documentation of all activities is an integral part of field inspection and investigation. Samplers must keep individual notes on sampling operations. This is a written record of all field data, observations, field equipment calibration, sample and chain-of-custody forms. These notes should be kept in a notebook, which is bound, water-resistant and has sequential page numbers (printed or hand written). The notes should ideally be kept by one person, written in waterproof ink. The notebook should contain the following information for each sampling program.

SITE INFORMATION

- name of sample collector
- name of company
- site name and GPS coordinates
- general description of the area, including land use practices upstream and downstream of sampling locations
- information on processes or products observed, waste generations, etc.
- reason for visit
- date and time of arrival and departure
- names and affiliations of persons present (e.g., business cards)
- field instrument calibration information
- weather conditions on the day of sampling
- all pertinent observations (e.g., flow rates)

SAMPLE INFORMATION

- sample numbers (including QC samples) and time of collection
- nature of spilled materials or contaminating substances, composition and concentration, if known
- number of samples and sample identification
- time (hour, day, date) of collection
- number and nature of QC samples
- specific location and diagram of sampling points
- method of sample collection and any factors that may affect sample quality
- equipment used
- information on any background or control sites

The collection of field data such as pH, conductivity, temperature, static water levels and ambient air characteristics is critical to most sampling and investigative work.

When inspecting industrial facilities (e.g., pulp and paper mills), maps, site diagrams and similar information are generally available from the plant, if you do not already have this information. This should be kept as reference for future inspections.

Notebook entries should be a chronological recording of information and activities. Each page should be numbered and contain accurate and inclusive documentation of the sampling activity. Each entry should be marked with date and time. Because the notebook information forms the basis for later reports it should contain objective factual information, free of personal feelings or other terminology which might be inappropriate. Notebooks should not be shared and only contain notes of the person who the notebook belongs to. Entries should never be scratched out. If an error is made a single line should be put through the entry and initialed. Pages should not be removed. All notes should be made at the time or as soon as possible thereafter.

If a sample label is lost in shipment or was never prepared:

Prepare a written statement detailing the collection and transportation of the sample from the field to the lab. Include description of the known chain-of-custody information, as well as references to any associated entries in the notebook about that sample.

1.3.6 PHOTOGRAPHIC DOCUMENTATION



STILL PHOTOGRAPHS (CONVENTIONAL OR DIGITAL)

In many cases you will want to document sampling activities using photographs. Photographs are often the easiest, most accurate and convenient way to demonstrate your observations and provide positive identification of the sampling point. Photographs documenting sampling points should include two or more reference points to make it easier to find the point at a later date. Photographs should be taken of the samples showing their labels with the sample number clearly visible and linking the samples to its source.

For each photograph, record in the notebook:

- date and time of the photograph
- photographer's name
- name of site
- compass directions or GPS location and description of the subject taken
- photograph number and/or film roll number

VIDEO

Video coverage can also be very valuable. It can be used to establish where samples were taken and that samples were taken properly. It can also record site conditions and can give those who have not been on-site an idea of the circumstances. Care should be taken in choosing the camera angle. Be sure to label the videotape with the place, date, incident number and your name. If you decide to include a verbal documentary of the scene on the video camera's sound track, ensure that your descriptions of the scene are accurate.

1.3.7 CONTAINER LABELS

Once the sample has been sealed correctly, it should be properly labeled.

Use pre-numbered, waterproof permanent adhesive polyester labels (or equivalent). They can be ordered pre-printed and in two parts with corresponding unique numbers; one part for the bottle, the other for your documentation.

Another way is to etch the container with a glass etcher. The etchings are marked directly into the glass and form a permanent record.

The label should contain:

- site name
- date and time
- initials of sampler
- type of analysis requested

Transfer information from the label to the notebook. Use a numerical sequence consistently, for example going from year, month, day, hour and minute.



1.4 LEGAL SAMPLING

1.4.1 CONDUCTING LEGAL SAMPLING

Legal samples are those collected as evidence for a possible prosecution under the Fisheries Act or Canadian Environmental Protection Act (CEPA). Samplers must demonstrate the reliability of evidence by providing a documented record of custody of the samples which are offered for evidence. Collection, transport and analysis must be defensible from the standpoint of objectivity, continuity and quality of results.

In the field, legal sampling requires the following factors to distinguish legal samples from routine samples:



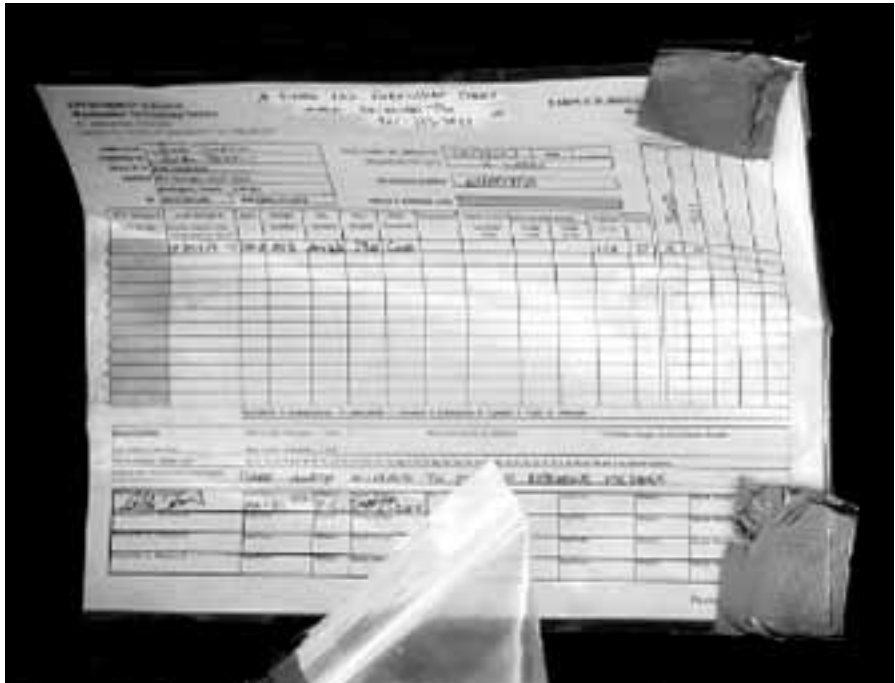
Ability to demonstrate the continuity of custody of the samples from the time of collection to the time of analysis, to show that the samples could not have been tampered with at any stage in their collection, handling and shipping.

When samples are submitted to the laboratory is when the distinction between legal and non-legal samples arises; legal samples require additional work in the laboratory. Although legal protocols may be followed during the collection of samples on submission to the laboratory, officers will have to use judgment as to whether samples are legal or not.

Legal sampling should be conducted under the following circumstances:

- any known or suspected violation
- spills or environmental accidents
- when you have no previous knowledge about compliance history
- when taking bacterial samples

1.4.2 CONTINUITY/CHAIN-OF-CUSTODY DOCUMENTATION



A chain-of-custody form is a written record for legal samples to document continuity by tracing the possession of the sample from collection through introduction into evidence. The form is signed by the sampler to attest that the samples have always been secure while in their possession and could not have been tampered with at any stage. A chain-of-custody form must always accompany legal samples.

Chain-of-custody is required when samples are collected, transferred, stored, analyzed and finally destroyed. A sample is in custody if it is in actual physical possession, in view after being in physical possession or in physical possession and locked up so that it can not be tampered with.

The following steps must be taken to ensure sample continuity:

SEALS – Ensure the sample has been sealed using proper techniques, such as custody seals, masking tape, etc.

PERMANENT MARKING – Label the sample using a permanent water proof uniquely numbered label and/or using a diamond-tipped scribe, waterproof marker, or other means of permanent identification with sufficient information to enable you to identify the sample later in court.

NOTEBOOK – Transcribe information from the sample labels into the notebook, including reference to unique identifier.

SECURITY – Lock the sample in a secure container, refrigerator, etc. or keep the sample in your possession or in view at all times until it can be secured. Limit the number of people handling the sample and ensure that only one person at a time has access to the sample.

CHAIN-OF-CUSTODY FORM – Complete the chain-of-custody form and include it with the samples.

PACK – Place the samples properly in appropriate shipping containers.

PHOTOGRAPH – Provide a visual record of the sample container(s) before shipment to document your shipping preparation.

DELIVER – Send the sample(s) to the laboratory by the appropriate means, ensuring that the sample(s) arrive within the required holding period. Samples should be delivered to the laboratory as soon as possible to minimize sample degradation.

RECORD CONTACT INFORMATION – If shipping samples by courier, air freight, or similar means, make a note of the waybill number and laboratory address.

CONTACT – Advise the laboratory that the sample has been shipped and give the waybill information. Keep the original waybill on file.

NOTE TAKING – Keep detailed notes of the sample collection methods, container markings, packaging and shipping details.

1.5 EQUIPMENT AND QUALITY ISSUES

1.5.1 PLANNING EQUIPMENT AND CONTAINER NEEDS

You should prepare an equipment checklist, an example can be found in Part 5: Appendices, which include sampling and safety equipment, sample containers, shipping materials and legal sampling equipment.

Decide how the equipment will be shipped to and from the sampling site (such as government vehicle, transportation company).

Generally the lab conducting analysis will supply or have requirements for the type of sampling containers needed. This will take into account the following:

- sturdiness
- the sample medium (liquid, solid, gas)
- possible contamination of the sample
- compatibility or chemical reactivity with the sample
- possible sample degradation by light, oxidation, etc.

When choosing sample containers, keep in mind the following additional considerations:

CONTAINER	USE
Glass	Organic analyses for sampling hazardous materials because glass is inert to most substances.
Amber glass	Prevents photo degradation; if they are not available protect samples from light.
Plastic or Teflon	Strong alkali solutions, hydrofluoric acid and mercury samples.
Teflon or glass	Organic samples and media.
High-density polyethylene (HDPE)	Metals (except mercury) for most inorganic samples and media.
Glass, polyethylene, or polypropylene	Bioassay samples.

CAUTION: Plastic containers (polyethylene, polypropylene, polycarbonate, polyvinyl chloride, or polymethylpentene) must not be used for samples intended for organic analyses, such as petroleum products, PCBs, pesticides, and wood preservatives. Plastics will leach out phthalates, which will interfere with the analyses.

CONTAINER LIDS AND LID LINERS

It is easy to take lids and lid liners for granted, but inappropriate lids or lid liners can cause serious problems. Lid liners made of paper or cardboard are a potential source of contamination.

Lids should be lined with Teflon or Teflon-coated material. In certain cases, polyethylene liners may be acceptable. Lids should be of the screw on variety and form a leakproof seal.

Heat-treated aluminum foil is sometimes used to cover the mouth of a container before the lid is screwed on to avoid contamination from a plastic lid, particularly for sampling petroleum products.

Part 4.1: Tables – Containers, Preservation and Holding Time has detailed information on container use and holding times.

1.5.2 EQUIPMENT FOR LEGAL SAMPLES

From laboratories you can obtain bottles which are suitable for legal analysis without further preparation, as long as continuity is maintained from the time of receipt of the sample bottles. Cleaned legal sample bottles should be kept in a secure area that has restricted access.



Commercial suppliers can also provide containers to meet United States Environmental Protection Agency (EPA) standards for cleanliness. The bottles are cleaned according to EPA protocols for the parameter or group of parameters to be analyzed. A certificate of analysis is available for each batch of sample bottles for an additional cost; the suppliers also retain representative bottles of each batch in case the cleanliness of the containers is questioned. Labels and seals are usually included with the containers.

Other equipment for legal sampling includes:

- permanent markers and/or marking scribe (e.g., diamond pen) for etching initials and other identification directly onto sample containers
- labels specifically designed for legal purposes
- custody seals or sealing tape
- Teflon tape for leakproofing (petroleum product sampling)
- sealable plastic bags
- lockable storage box
- notebook

1.5.3 DATA QUALITY

Data quality objectives (DQOs) define the degree of uncertainty or error that can be tolerated in the data. DQOs may be qualitative or quantitative. Qualitative DQOs are specific descriptions of actions to be taken if a response does not meet the DQO. For example, what action will be taken if QC samples are found to be contaminated?

Possible solutions include:

- discarding the data
- tracking down the problem and re-sampling
- subtracting the contaminated amount from the data

Quantitative DQOs, on the other hand, involve specific quantitative terms such as standard deviations, relative standard deviations, percent recovery, relative percent difference, and concentration. For example, if the desired lowest limit of detection cannot be met, what actions should be taken?

Possible solutions include:

- accepting a higher detection level
- compositing several samples to obtain one large sample
- trying a different analytical method
- resampling the same site or a different site and taking larger samples

1.5.4 QUALITY ASSURANCE AND QUALITY CONTROL (QA/QC)

Quality assurance (QA) is the overall program designed to ensure that the sample data meets data quality objectives (DQOs). This program includes, sampling plan design, employee training, equipment maintenance and calibration procedures, quality control, corrective action plans, performance audits, data assessments, validation, storage, management and reporting.

Quality control (QC) is the system of guidelines, procedures and practices designed to regulate and control the quality of products and services, ensuring that they meet pre-established performance criteria and standards. This encompasses sample blanks, replicates, splits, equipment calibration standards, sample container size, quality, use and preservative amount. Quality control is part of the overall quality assurance program.

Quality assessment is a system review that determines whether the quality assurance and quality control programs are being properly carried out. It involves evaluating and auditing the policies, guidelines, procedures, practices and results to assess the overall effectiveness of the QA/QC program.

QC is one part of QA. QC consists of internal (technical) activities such as the use of QC samples, to control and assess the quality of the measurements, while QA is the management system that ensures an effective QC system is in place and working as intended. (L.H. Keith, 1997. *Environment Sampling and Analysis: A Practical Guide*.)

WHY QA/QC?

Sampling operations may cause systematic and/or random errors in the sample results. Systematic errors may be caused by poor sampling practices or equipment design failures and are usually constant. Random errors are considered to be unavoidable and unpredictable errors. An effective QA/QC program will help to identify and control the frequency of such errors.

Field quality control samples such as travel, field and equipment blanks, split samples and replicate samples are designed to help identify potential problems. Travel, field and equipment blanks may be used to identify sample contamination at different points in the sampling procedure. This contamination could be either systematic or random. Replicate and split sample data may be used to calculate sampling precision. These systematic errors are usually expressed as a standard deviation. Quality control samples should be handled exactly the same as regular samples, using identical sampling devices, sampling protocols, sample containers, shipping procedures and preservation techniques.

1.5.5 FIELD QC SAMPLES

The number of QC samples depends on the objectives of the sampling program. Often the laboratory or the established analytical protocols dictate the types and number of samples needed. When preparing QC samples always use deionized (DI) water, as defined in the latest edition of Standard Methods for the Examination of Water and Wastewater for Reagent-Grade Water, Type I.

TRAVEL BLANKS

A travel blank (trip blank, transport blank) is used to check for background contamination, contamination from transport and handling, or both. Suggested Procedure:

- Before setting out, fill the appropriate containers with DI water. Add appropriate preservative as required by the protocol for the analyte of interest.
- Label blank containers clearly, indicating that these are blanks. Place samples among the empty sample containers. Keep them at 4°C, or at the same temperature the samples will be kept at.
- Do NOT open the blanks in the field. Travel blanks are transported to the field with the regular sample containers unopened and then return together with the field samples to the laboratory unopened.

NOTE: If you do not have access to lab facilities and cannot prepare your own travel blanks, ask the laboratory to provide them.

FIELD BLANKS

A field blank is exposed to the same field and travel conditions as the samples of interest and corrects for ambient conditions. You will need to prepare at least one field blank per day for each facility being sampled.

SUGGESTED PROCEDURE

- Transport 2 L - 5 L of DI water in a clean, appropriate container (see list of appropriate containers, Part 4.1: Tables – Containers, Preservation and Holding Time) to the field. During sample collection, prepare a field blank by filling an appropriate container with DI water, using identical procedures as used for normal field samples. This process will expose the DI water to the same environmental conditions as the field samples.
- Treat the blank(s) exactly as the samples are treated, using the same preservative (if any) and pack the blank with the samples to expose it to the same conditions during transport and shipping. Label and send the blanks to the lab with the field samples.

NOTE: If conditions vary significantly between sampling points at a facility, or if the sampling points are more than a kilometre apart, consider collecting extra field blanks at each sampling point.

EQUIPMENT BLANKS

Equipment blanks (rinsate blanks, instrument blanks) are a sample of DI water or solvent used to rinse the equipment. It is essential to take an equipment blank when sampling equipment contains materials that might contaminate the sample (e.g., plastic tubing or metal parts). Rinsate blanks test how well the sampling equipment has been decontaminated between sampling. Check with the laboratory or with experienced personnel to determine if you need to prepare these blanks.

SUGGESTED PROCEDURE

- Collect a sample of the final equipment rinse water or solvent as an equipment blank after the equipment has been decontaminated and before the next sampling session.

15.6 ADDITIONAL QC SAMPLES

You may be asked to include additional QC samples. These will be required less frequently, but you may be responsible for providing them.

MATERIAL BLANKS are samples of construction materials such as those used in groundwater wells, pumps and flow testing. These samples can be used to test for experimental artifacts from these materials.

FIELD SPIKE is a field sample to which a known amount of the analyte of interest is added during field sampling. Spikes are used to identify field, transportation and matrix effects. It is important that field spike samples be prepared by experienced personnel so that interpretation of analytical results is not complicated by human error.

SPLIT SAMPLES are two or more samples taken from the same initial sample. Samples may be split in the field or in the lab. It is recommended that laboratory personnel be consulted for advice on splitting samples. For example, splitting samples for organic analysis may result in subsamples that are not identical because organics often adsorb onto container walls. In cases such as this it is best to split samples immediately at the sampling site.

FIELD REPLICATE SAMPLES are two or more separate samples taken at approximately the same time at the same sampling site. These samples are used to calculate sampling precision. Laboratory in-house replicates are replicates generated by the testing laboratory and are used to calculate test method precision.

After regulatory sampling, an inspector may be requested to provide a split sample to the facility from which samples were collected. As a general rule, samples collected for compliance monitoring should not be split.

NOTE: Legal samples should not be split under any circumstances. The preferred alternative is to request facility personnel to collect their own sample.

In some situations, however, inspectors may request the facility owner or operator to provide a split sample from the facility's sampler, where it is not possible to collect a separate sample. In other situations, an inspector may be interested in obtaining comparative analysis for particular parameters, so splitting a sample may be necessary. When splitting samples for analysis comparison both samples must be treated the same with respect to pre-analysis holding time and temperature.

15.7 SOURCES OF CONTAMINATION

Possible contamination is always a concern when collecting samples. Cleanliness is a high priority, since a contaminated sample is useless. Contamination between containers, tools and equipment could become an issue during legal proceedings.

When preserving samples with concentrated acids, cross contamination from acid vapours can significantly alter the pH of an open sample designated for pH analysis, therefore ensure that those samples that do not require the addition of preservative are kept tightly capped and do not come in contact with acid vapours from your gloves or other samples.

Sample containers and clean sampling equipment must never be stored near solvents, gasoline, or other volatile substances that might cause contamination. If possible, to minimize contamination, do not fill the gas tank of your vehicle on the day of sampling until after all samples are collected.

Disposable gloves should be worn at all times when handling preservatives, sample containers and sampling equipment. Do not touch the inside of the sample container, the sample or the chemical preservatives with your gloves.

Samples may also be contaminated by:

- inappropriate containers or equipment
- containers that have not been properly cleaned
- dirty container caps
- loosely or improperly capped containers
- contaminated preservatives
- cross-contamination introduced by sampling equipment
- exposure to open air, which may contain various vapours
- sloppy sampling techniques

METAL ANALYSIS

- Do not use metal sampling equipment or hand tools.
- Hand tools such as spatulas, pipettes, trowels and the like should be wrapped only in plastic.

ORGANIC ANALYSIS

- Do not use plastic equipment (use Teflon as much as possible).
- Hand tools such as spatulas, pipettes, trowels and the like should be wrapped in cleaned or heat-treated aluminum foil.

CLEANLINESS

- Keep equipment in good repair and clean during transportation.
- Use non-porous material such as stainless steel chain as an attachment to sampling devices, it can be properly decontaminated. Only use rope if you have enough to use a fresh length each time, used rope can transfer contaminants between sites.
- Do not sample downstream of bridges, abutments, boats, etc.
- Do not put samples in containers whose history is unknown.
- Keep containers tightly sealed and store sample containers in clean areas.
- Do not smoke while sampling. Tobacco smoke contains ammonia, oxides of nitrogen and other contaminants. If water samples are left loosely capped in a vehicle filled with tobacco smoke, these samples may be contaminated through diffusion.
- Decontaminate equipment between sampling sites according to protocol.
- Sampling equipment should remain in the wrapping material until it is used in the field.
- Some single use equipment, such as bags, spoons, etc., can be purchased from laboratory companies as certified clean.

TUBING

- All lengths of polyethylene, surgical tubing and polyvinyl chloride (PVC) tubing should be as short as possible and where feasible replaced with Teflon tubing. Tubing should either be thoroughly cleaned or replaced between sites; or be dedicated to a specific site to avoid cross-contamination. If required, take equipment blanks.
- Polyethylene and other kinds of plastic tubing can leach out phenolic compounds and phthalates, which interfere with some organic analyses.

1.5.8 CLEANING FIELD EQUIPMENT

Always clean equipment parts that come into contact with the sample (water, sediment, sludge, effluent) to avoid cross-contamination.

BEFORE SAMPLING

Whenever possible clean all equipment before going into the field.

SUGGESTED CLEANING METHOD

- Wash equipment with phosphate-free laboratory grade detergent and hot water, using a brush on all accessible surfaces to remove all visible particulate matter and other residue.
- (Optional) Use a steam or high-pressure water washer to remove any dirt or residue.
- Follow approved laboratory cleaning methodology for the analyte in question.

IN THE FIELD

- Scrub equipment with detergent and remove all visible particulate matter and other residues. Rinse several times with tap water, then three times with DI water to remove the detergent.
- Take an equipment blank.

15.9 CLEANING CONTAINERS

Sample containers may be purchased from various suppliers or obtained from the laboratory. Containers must be properly cleaned. The cleaning procedure is dictated by the specific analysis to be performed on the sample. There are several commercial suppliers of pre-cleaned sampling containers that are suitable for use without further cleaning, provided they have been cleaned using the appropriate protocol.

SUGGESTED CLEANING METHOD

- Wash sample containers with phosphate-free lab detergent; scrub all surfaces with a brush kept for this purpose.
- Rinse containers thoroughly, first with tap water and finally with DI water.
- Specific container preparation requirements in addition to the normal cleaning procedures:

ANALYSES	CLEANING METHOD
METAL	Soak containers in 10% - 20% nitric acid and rinse at least three times with DI water before taking them into the field.
ORGANIC	Clean glass jars and bottles as per lab protocol or use lab-prepared containers. Be aware that cross-contamination with organic solvents or substances can be a serious problem; this is why heat treatment of containers and equipment is frequently recommended.
BACTERIAL MICROBIOLOGICAL	Sterile containers are usually available from the laboratory. If not, sterilize bottles and equipment for microbiological sampling, or for any samples requiring sterile conditions, according to Standard Methods for the Examination of Water and Wastewater, Washing and Sterilization 9040 (American Public Health Association).
BIOASSAYS	Plastic containers or plastic liners; steam-clean containers or wash them with soap, and rinse them five times with tap water; rinse containers three times with the sample to be collected.

1.5.10 SAFETY CONSIDERATIONS

Build safety considerations into the planning process from the start. Consider potential safety concerns related to sampling, such as the need for:

- personal protective equipment (PPE)
- life jackets for sampling on or near water
- fall protection
- specialized air monitoring equipment for hazardous environments
- specialized equipment for handling potentially harmful preservatives

Make yourself thoroughly familiar with **THE INSPECTOR'S SAFETY GUIDE** and follow its directions for safe sampling. Always be alert to the possibility of danger, especially in dealing with unknown sites, situations or possible contaminants.

You should only take the amount of preservatives necessary in order to avoid having to deal with excess chemicals once you have completed your sampling. The extra preservative will either have to be disposed of properly or returned to the laboratory.

If you require corrosive or oxidative reagents for your sampling expedition, ensure that they are stored in containers that are secure and non-reactive with appropriate lids.

If nitric acid is among your reagents, ensure that the cap to the container or dispenser is Teflon-lined.

CAUTION: A number of reagents – concentrated acids and alkalis, potassium dichromate and other chemicals – may be corrosive or strong oxidants. Never handle these compounds without wearing protective gloves and eyewear.

Never add water to strong acid; it may boil up. Dissolving any concentrated chemical (sodium hydroxide pellets, for example) may generate heat; handle vessels with care. Concentrated nitric acid fumes freely.

Take only the amount of reagent you need to complete the work in the field.

2.0 FIELD SAMPLING TESTS AND EQUIPMENT PROTOCOLS

2.0 FIELD SAMPLING TESTS AND EQUIPMENT PROTOCOLS

2.1 STANDARD FIELD TESTS

2.1.1 FIELD TESTS

2.1.2 FIELD EQUIPMENT CARE AND MAINTENANCE

2.1.3 FIELD MEASUREMENT OF PH

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2.4.12 SAMPLING TRIER

2.4.13 SCOOPS, TROWELS AND SHOVELS

2.4.14 THIN-WALLED CORER (PUSH/SHELBY TUBE)

2.1 STANDARD FIELD TESTS

2.1.1 FIELD TESTS

The most common field test parameters for water sampling are pH, conductivity, temperature and dissolved oxygen. Other parameters may include total dissolved solids, specific ions and turbidity. Parameters chosen will depend on the survey objectives.

Temperature and pH are two important parameters to measure in the field, since the values can change once samples have been collected. The field measurements can be compared with those obtained in the laboratory to determine if any significant changes or unacceptable deterioration in samples has occurred during transport.

Dissolved oxygen is a useful parameter in an environmental emergency or spill situation. It can help determine why a fish kill may have occurred. It may also indicate if oxygen concentrations are being affected by the presence of a particular substance or if low oxygen values could result in a possible fish kill.

2.1.2 FIELD EQUIPMENT CARE AND MAINTENANCE

Field instruments are usually battery powered and may require special batteries not easily available in remote locations. Rechargeable nickel-cadmium (Ni-Cad) batteries may be affected by prolonged storage periods. Ni-Cad batteries can develop a memory loss if not cycled occasionally. During storage, you should try to discharge and recharge these batteries occasionally to keep them in good condition. Also, some pH meters may continue to use battery power to maintain their calibration, even though the power is off, so it is a good idea to remove the batteries from such units during extended storage periods.

Before leaving on your sampling trip, make sure that you understand the field equipment's operation and maintenance requirements and that you have included any necessary spare parts, calibration standards and batteries. Meters should be calibrated prior to departure for the field as this will ensure that the meters are operating properly.

In the field:

Ensure that equipment has been properly calibrated or standardized according to the manufacturer's recommendations. The frequency of calibration or standardization will vary. Document your calibrations including expiration dates of buffer solutions and any meter adjustments made.

Ensure that equipment is safe for field conditions. Protect equipment from temperature extremes. If equipment is left in a vehicle, solutions may freeze in the cold or deteriorate from excess heat. Protect equipment from physical damage and be aware of the proper care and handling of each instrument.

In certain situations, samplers may be required to conduct air monitoring to determine if combustible vapours or specific air contaminants are present, particularly during environmental emergencies. Equipment for this includes explosimeters and various portable analyzers and detectors. Since this type of field equipment is quite variable and specialized, its use will not be covered in this manual.

The manufacturer's instructions should be consulted. Some advice may be obtained from Environment Canada's Emergency Sciences Division, Environmental Technology Centre, 335 River Road, South Gloucester, Ontario, KIA 0H3.

Never change batteries under explosive conditions. Dispose of Ni-Cad batteries as hazardous waste.

2.1.3 FIELD MEASUREMENT OF pH

There are many types of pH meters, but their operating principles are all the same. Using the pH meter is a two-stage operation: the meter is calibrated against a buffer solution of known pH value and then the sample's pH is checked.

To calibrate a pH meter follow the manufacturer's recommendations. If the meter does not automatically adjust to temperature, check the temperature of both solutions with a thermometer and record the results. The pH buffer solution may only be used once for calibration and should be discarded since there is flow through and liquid exchange between the pH probe and the buffer solution.

The buffer solution used as a calibration standard should be in the same pH range as the sample to be tested. If you are unsure of the sample's range, check it with pH paper. Buffer solutions have a limited shelf life, remember to check the expiry date before taking the buffer solution into the field. All buffer solutions and pH paper must be stored in a clean, dry environment.

Remember to rinse the probe with DI water after checking with buffer solution.

In many pen-type pH meters, the pen tip is susceptible to drying out. If the tip dries, the meter will not respond. Check to see if your pH meter tip needs to be kept immersed in solution and monitor this regularly.

2.1.4 FIELD MEASUREMENT OF WATER TEMPERATURE

Collect the water sample in a 125 mL wide-mouth bottle. Stand the thermometer in the sample, ensuring that the top of the liquid is level with the thermometer immersion line. Allow the thermometer to equilibrate for at least 3 minutes. Read the water temperature by holding the bottle and the thermometer at eye level and keeping the bulb of the thermometer submersed in the sample and ensuring that the thermometer is immersed to the thermometer immersion line. Record the temperature to the nearest 0.5°C. Use only calibrated thermometers. Thermometers should be checked against a standard thermometer annually for calibration accuracy. Do not use a thermometer that has a break in the enclosed liquid or if there is liquid in the expansion chamber at the top of the thermometer. Avoid using mercury thermometers. These situations indicate that the thermometer is no longer calibrated. Temperature can also be measured in situ.

SUGGESTED PROCEDURE

- Make sure the instrument is properly calibrated according to the manufacturer's recommendations and specifications. Try to use standard solutions with specific conductance closest to the values expected in the field.
- Do not use the same sample that has been used to measure pH because the pH electrode changes the conductivity of the sample.
- Rinse the sample container three times and the probe several times with the sample water.
- Make sure the probe does not touch the sides or bottom of the sample container.
- Take the reading, then repeat the procedure with fresh samples until reproducible readings are obtained that are within $\pm 5\%$ of each other.

2.1.5 DYE TESTING

Studies using dye for flow tracing can vary in sophistication, ranging from dumping a few millilitres of dye into a storm sewer manhole and observing where the discharge emerges in a nearby watercourse, to in-ground water studies, collecting dyes injected at known rates, up gradient, onto absorption media that are extracted and measured by spectrophotometry.

For more information, see Aley, Thomas, 1991, “The Water Tracer’s Cookbook and Related Groundwater Tracing Information”. Ozark Underground Laboratory, Protom, Missouri.

For most inspection purposes, dyes are used to verify or locate the final discharge point of spills or effluents that go through a closed or otherwise unobservable path (e.g., culverts, storm drains, drainage tiles, piping) before discharging to the environment.

Dyes may be suitable for either salt or fresh water, depending on their solubility. Intracid Rhodamine WT and Fluorescein (xanthene dyes) are water-soluble dyes commercially available for dye testing.

- Intracid Rhodamine WT is available as a dark red liquid, in a 20% solution. It turns water a bright, fluorescent orange colour.
- Fluorescein (an orange crystalline powder) turns water a fluorescent yellow-green colour.

Of the two, Fluorescein is the dye of choice for tracing enclosed piping, as it is rapidly destroyed by ultraviolet (UV) light.

Intracid Rhodamine WT is less susceptible to UV, less readily absorbed by fine particulate matter and is less toxic which makes Intracid Rhonamine WT the dye of choice for determining effluent flows using fluorescence.

DYE CALCULATION

- Fluorescein is visible to the trained eye at 10 ppb and to the untrained eye at 100 ppb; therefore aim for a concentration of 1 000 ppb or more. This is based on a commercial dye strength of 35% - 37% dye/powder.
- Fish toxicity of the two dyes (determined by 96h LC₅₀ rainbow trout) is 320 000 ppb for Intracid Rhodamine WT and 1 372 to 3 433 ppb for Fluorescein.

To calculate the amount of dye to be added, use the following formula. You will have to estimate the variables. $V_d = (QL/V) C_p$

V_d = volume (mL) of dye

Q = discharge rate (m³/s)

L = length of flow path (km)

V = velocity (m/s)

C_p = concentration (ppb) at terminal site (= 1 000 ppb)

In using dyes, remember:

- Once wet, dye will stain everything it touches and is extremely difficult to remove from clothing, hands, equipment, etc.
- Only conduct dye testing after obtaining all other samples.
- Inform emergency response agencies and local water authorities that you are conducting dye tests, as the resulting fluorescent waters will lead to many enquiries.
- Any equipment used to handle the dye will have to be bagged and disposed of since cleaning it is almost impossible.
- Low concentrations of fluorescein dye exposed to sunlight will be visible for an hour or more.

SUGGESTED PROCEDURE

1. Position personnel at observation points along the watercourse to record the progress of the dye slug. This includes removing manhole covers and inspection grates to allow visual inspection.
2. Introduce the dye directly into the watercourse, gently flushing it in with a stream of water. Record the time at which the dye was introduced and the times at which it appears at the observation points. If possible photograph the dye as it appears at each point.

2.1.6 CLARITY

Water clarity is usually measured with a Secchi Disc.

SUGGESTED PROCEDURE

1. Lower the Secchi Disc into the water to the depth where the pattern is no longer visible. Lower the disc further down and start pulling up until the pattern reappears and becomes visible. Note the depth at which the pattern becomes visible. The extinction depth is calculated based on two consecutive readings to the nearest 0.1 m.
2. Record the atmospheric and water conditions in your notebook. Avoid taking readings during dawn and at dusk.

2.1.7 DISSOLVED OXYGEN



Dissolved oxygen can be measured either by chemical titration or by the more commonly used oxygen-sensitive membrane electrode.

Follow the manufacturer's instructions for transportation, storage, calibration and measurement of the dissolved oxygen meter and probe. Ensure the probe is equilibrated before each reading is taken. The membrane must be changed if deterioration is noticed and readings become unstable. Watch for trapped air bubbles under the membrane and discoloration of the internal white electrode.

2.2 EMERGENCY RESPONSE

2.2.1 SAMPLING SPILLS

Sampling in the event of a spill requires common sense and good judgment. The primary objective is to collect enough unaltered material for chemical identification (fingerprinting) and subsequent linkage to a spill source.

Two fundamental principles apply to sampling spills: sample as soon as possible, before the spilled material can be dispersed or degraded; and treat all samples as legal samples. Using a legal sampling approach from the outset is both cost and quality effective and eliminates the need for search warrants if future samples are required. Moreover, the spill may dissipate or weather before you can return for legal samples at a later date.

Some general considerations:

- Analytes or samples most likely to disappear or deteriorate should be collected first.
- For large spills or for a spill involving more than one substance, obtain several samples from different locations to ensure that the spilled substance is properly represented.
- Samples should, if possible, follow and show the course of the spill. Samples should be collected above and below the point source of the discharge, to demonstrate that the contamination did not originate from another source.
- Collect a sample from the discharge source, if known (e.g., tank, container, pipeline, truck) to allow the laboratory to match the source with other samples collected.
- In spill incidents, the primary aim is qualitative: the question is more likely to be what rather than how much.
- In chronic pollution, the interest is more quantitative: the question is more apt to be how much rather than what.

2.2.2 SPILLED PETROLEUM PRODUCTS

Oils and waste oils may contain various contaminants so it is wise to treat them as hazardous materials. Appropriate safety precautions and careful handling should be followed at all times. When sampling transformer oils, you should determine if the oil contains PCBs. When in doubt, assume that it does and follow the procedures given for PCB sampling in this manual. Further information can be found in Parts 3 and 4.

The main sources from which oil samples may be collected are oils on water, from shorelines (oil on pebbles and stones, in sand or on debris), from contaminated wildlife and the pure oils from storage or dispensing devices.

- Protective gloves must be worn at all times. It is advisable for prevention of cross contamination and for health and safety reasons to wear two pairs of disposable gloves when taking samples. The outer pair of gloves is to be changed after each sample and the inner pair of gloves is to be changed any time they become soiled.
- Oil on skin should be removed as soon as possible by scrubbing with soap and warm water.
- Collect oil samples with as little water as possible. This will help reduce chemical, physical or biological alterations of the oil when in prolonged contact with water between the time of sampling and analysis.
- Do not fill jars completely. Temperature change of the sample could cause expansion and breakage of sample container.
- Whenever possible, avoid collecting oil samples containing organic material such as marine vegetation, grass, bits of wood, etc.
- At no time should plastic material come in contact with oil.
- After sample collection, store in a cool and dark place in order to reduce evaporative changes or microbial degradation of oil samples.

Take commonsense steps to ensure that samples do not become cross contaminated with oils from collection tools or from other samples.

When sampling petroleum products, use amber glass bottles, 125 mL - 250 mL, with Teflon-lined lids whenever possible. For wide-mouth jars, use Teflon or heat-treated aluminum foil as a seal between jar and lid.

The lab requires a minimum oil layer of 3 mm in a 125 mL glass bottle to run the full range of analytical tests. Collecting this volume can be difficult when the spill forms a thin surface film or sheen.

SUGGESTED PROCEDURE

- To concentrate the sample to the required thickness, tilt the lip of the sampling bottle just below the water surface and skim off the oily layer. If this yields too thin a layer, cap the bottle and invert it. Allow the oil to rise to the surface; then loosen the cap just enough to let the aqueous under layer run out of the bottle. Tighten the cap, turn the bottle right-side up and repeat the process until enough oil has been collected.
- In some cases you may have to resort to collecting a sample using absorbent material. Place an absorbent pad on top of the oil film and slowly turn it in a circular motion to collect the oil layer. Transfer the pad to a clean glass bottle and seal it. Include a piece of unused material in a separate jar for use as a blank.
- In emergency cases mason jars can be used in spill sampling. When sampling light petroleum products (e.g., gasoline, kerosene, diesel, furnace oil, jet fuel), line the lid with heat-treated aluminum foil to prevent the oil from dissolving the lid liner. If using a mason jar, include a clean unused jar as a blank.
- To clean the outside of the jar, wipe it with paper towels. Do not rinse or immerse the jar.
- Liquids in small dispersed pools, in globules or in difficult to reach areas may require sampling with a disposable glass or plastic pipette and bulb.
- For oil on a solid surface, gently sweep the surface with a piece of oil-absorbent material and place material into a sample jar. Include a piece of unused material in a separate jar for use as a blank.

TABLE 2.2.2

GUIDE FOR ESTIMATING THE SIZE OF AN OIL SLICK ON WATER

STANDARD TERM	APPEARANCE	OIL (L/KM ²)
BARELY VISIBLE	barely visible under most light conditions	44
SILVERY	visible as a silvery sheen on surface water	88
SLIGHTLY COLOURED	first traces of colour visible	175
BRIGHTLY COLOURED	bright bands of colour visible	330
DULL	colours begin to turn dull brown	1 170
DARK	much darker brown	2 337

NOTE: Each 2.54 cm thickness of oil equals 21.42 L/m² or approximately 30 500 000 L/km².

2.2.3 CHEMICAL CATEGORIZATION KITS AND STRIPS

Accidents and spills often involve unknown substances. In planning an early and efficient response to the emergency, one necessary first step is to determine what compounds or chemicals you are dealing with.

Hazardous chemical categorization kits use various reagents and tests to determine what the spilled product might be. These kits have improved over the years and are now more user friendly. They do tend to be very expensive and require some maintenance.

Chemical categorization strips are inexpensive, disposable and have a two-year shelf life.

They can be used to determine if the product:

- is acid or alkaline
- is an oxidizer
- contains fluorides, iodine, chlorine, or bromine
- involves organic solvents

Kits also exist to determine the presence of PCBs in transformer oil or soil. These kits are relatively inexpensive and easy to use. They are disposable and have a two-year shelf life.

2.3 SAMPLING TECHNIQUES

2.3.1 GENERAL EQUIPMENT RULES

Sampling equipment must be constructed of material compatible with matrix and target analyte. The wrong equipment or container will cause contamination.

RULES

1. Equipment should be stainless steel, not painted or plated.
2. For organic parameters, Teflon or medical grade silicone tubing should be used.
3. For microbiological samples, sterile bottles and aseptic techniques must be used.
For soil samples, use a sterile spoon to scrape top 1 inch of soil into pre-sterilized container.
4. Components of sampling devices (gaskets, seals) must also be evaluated and checked for analyte interaction (adsorption).
5. Use DI water to rinse sampling equipment prior to use.
6. Use only new sampling containers and lids. Confirm container type and size for sample volume required.
7. Obtain from laboratory a certificate of container cleanliness.
8. For metals, use a disposable field filtration kit if dissolved constituents are to be determined or submit unfiltered, unpreserved sample to the lab and indicate lab

filtration is required for dissolved metals.

9. Have a scribe, permanent marking pen and sealing tape or legal seal/label.



2.3.2 LIQUID SAMPLE BEHAVIOUR

Liquid samples are most commonly collected from surface waters (oceans, rivers, lakes, impoundments, run-offs, etc.), industrial locations, wells and springs. Water quality, in some situations, can vary considerably both in space and time. The behaviour of substances in water along with other conditions will influence the sampling methods.

FRESH WATER

- Organic matter may be suspended, but will also form strata in smoothly flowing channels.
- Organic compounds may adsorb to particulate matter in the water and cause it to sink to the bottom.
- Oils, grease and other petroleum products will float on the surface.
- Most halogenated organic compounds and PCBs are heavier than water and will sink.
- In lakes shallower than 5 m, wind action usually causes mixing, so neither chemical nor thermal stratification is likely.
- Deep rivers and lakes can exhibit chemical stratification, with or without accompanying thermal stratification.
- Stratification may occur where two streams merge.
- Incomplete mixing of substances may occur due to lack of turbulence, insufficient travel time from the point of discharge, or because of some of the factors previously outlined.
- Seasonal influences (thermocline, etc.).

For substances that dissolve in water and in cases where there is no concern about loss of the analyte due to aeration or volatilization, samples should be collected at a point of thorough mixing, where turbulence is high (e.g., downstream of a dam, flume, weir, rapids, or waterfall). If loss of volatile compounds is a concern, the sample should be collected from an area that has less turbulence.

Stratification is also a problem when sampling ocean water and large inland lakes. Various substances may be stratified at different depths and the composition of inshore water usually differs greatly from that offshore. Estuarine sampling is even more complex because stratification occurs in rivers unevenly.

2.3.3 SAMPLING TECHNIQUES AND DEVICES

Select materials that can be easily cleaned or decontaminated if you plan to use a sampling device that must be lowered into the substance to be sampled. Depending on the substance of interest, use of rope or string to collect samples should be avoided to prevent possible contamination, since rope and string are both very difficult to clean properly. Use stainless steel chain instead, or use a new piece of rope or string for each sample.

For general information on rinsing sample containers and equipment, see Part 1.5.7 - 1.5.9 and for specific protocol information check Part 4: Sampling Protocols. For example, some pesticides and halogenated compounds adsorb strongly to glass; when collecting these samples, do not rinse the bottle beforehand.

Be aware that rinsing the container will cause some mixing and disturbance of the water. If it is important to take your sample with minimal disturbance, you may wish to rinse the container with water taken a metre or two downstream from the sampling point. As a general rule you should avoid rinsing when it would significantly disturb the environment.

- If pre-cleaned bottles are not available, then rinse container three times with DI water or sample, exceptions are: sampling for any type of residue tests, oil and grease tests.
- For organics, do not rinse containers.

SURFACE SAMPLING BY HAND

Samples taken from shallow depths (less than 1 m - 2 m) can be collected by hand or by using a sampling pole (telescoping if required).

When sampling surface water from well-mixed rivers and unstratified lakes, you may take samples directly into handheld sample bottles. Collect water from below the surface to avoid skimming the surface film.

1. In a river, stand perpendicular to flow facing upstream. In a lake, wade beyond where sediment is affected by wave action.
2. Remove the lid ensuring not to touch inside of lid or mouth of sample container; or use a bucket, ensuring you do not touch the inside of the bucket.
3. Grasp bottle or bucket at the base with one hand and plunge bottle mouth down into the water or effluent.
4. Position the mouth of the bottle or bucket into the current, away from the hand of the collector and the sampling platform or boat making sure that the hand is always downstream.
5. Sampling depth should be 15 cm - 30 cm (6 in - 12 in) below the water surface.
6. If the water is static, an artificial current may be created, by moving the bottle or bucket horizontally in the direction it is pointed and away from the sampler.
7. For bottles, tip the bottle slightly upwards to allow air to exit and the bottle to fill; or remove the lid of the sample container and fill from bucket (ensuring not to touch inside of lid or mouth of sample container).
8. After removal of the bottle from the stream, you may need to pour out a small portion of the sample to allow an air space of around 2.5 cm - 5 cm for proper mixing before analysis.
9. Seal and label the bottle and place in cooler.

For bacteriological samples, use same procedure, except use 500 mL sterile bottle

Once you have taken the sample, if required, transfer it into the appropriate container, cap the container and if necessary, wipe down the outside of the container with a paper towel. To avoid introducing contaminants into the container, never immerse or rinse a sample bottle in water to clean it; use a clean, wet paper towel.

SAMPLING FROM GREATER DEPTHS AND/OR STRUCTURES

If you plan to collect samples from greater depths, some alternative sampling devices include:

- Van Dorn sampler (available in either horizontal or vertical configuration)
- Kemmerer sampler
- peristaltic pump with appropriate length of tubing (Teflon or plastic, depending on analyte)
- weighted sampler (also known as a sampling iron)

Although sampling from bridges or other structures has some obvious advantages, there are also potential contamination problems. Most structures are made of metal, concrete, or treated timber which can cause potential contamination. Always sample upstream of the structure. Make sure that there is no dirt on the sampler line and that the line's movement does not cause dust or dirt to fall into the samples as you lower and raise the sampler. General procedures include:

- The use of sufficient line to allow the sampling device to reach the required depth. The other end of the line should be secured to a permanent fixture.
- The sampling device can be weighted to keep it upright in the water, to allow accurate estimation of the depth of sample.
- When sampling from a boat or float aircraft, take the sample upstream from the engine to minimize the chance of contamination from oil.
- Avoid touching the bottom of the river or lake with the sampling device to avoid stirring up sediment.

2.3.4 SAMPLE COLLECTION METHODS



GRAB SAMPLES

A grab sample is a single, discrete sample taken from a specific spot at a specific time - a snapshot in both time and space. Theoretically, all samples can be taken as grab samples, but this is the least representative of all sampling methods. Grab sampling is a valid and often used sampling technique in many situations: regulatory sampling of industrial effluent, surface water sampling, spill situations and collecting product samples from tanks. In many cases it is the only technique available to a sampler.

Grab samples are collected over a short time, making them less expensive than composite samples, they also present fewer problems in maintaining sample continuity in legal sampling situations. In some cases you may want to collect several grab sam-

ples over a predetermined time for individual analysis or to form one composite sample, which would be more representative than a single sample.

Two types of grab samples can be collected: discrete and depth-integrated. A discrete grab sample is taken at a selected location or depth, at a single point in time. A depth-integrated grab sample is collected over a predetermined part or the entire depth of the water column, at a single point in time.

Grab samples are best collected directly in a sample container. They can be collected by hand or by using simple field equipment such as buckets or other containers. Typical grab sample equipment is a sampling pole (telescoping, if necessary) to which a sample container has been fastened.



COMPOSITE SAMPLING

A composite sample is a combination of a number of smaller samples collected over a period of time and/or locations. The objective is to produce an average sample composition when pollutant concentrations may vary over time. Compositing is useful when individual sample sizes may be too small for analysis. Compositing is also used to reduce the cost of analyzing a large number of samples when the frequency of individual samples containing the analytes of interest is low.

The alternative is to take a number of grab samples, analyze them separately and then average the results.

Compositing is simpler, easier and less expensive. The trade off is that compositing gives no information on variance (between samples, or over time) and may therefore mask real differences.

Composite sampling has other limitations such as:

- Information regarding analyte relationships in individual samples will be lost.
- Compositing may dilute an analyte to a level below the detection limit, producing a false negative.
- Grab sampling may be more cost-effective.
- If compositing reduces the number of samples collected below the required statistical need of the DQOs, then those objectives will be compromised.

2.3.5 TIME-PROPORTIONAL AND FLOW-PROPORTIONAL SAMPLING

There are two main types of composite samples in liquid sampling; time-proportional (sequential-proportional) and flow-proportional.

In time-proportional sampling, samples of equal volume are collected at regular intervals. Flow-proportional composites are obtained by collecting samples in proportion to the flow rate. Although this can be done manually, it is usually done using an automatic sampler hooked up to a flow meter. The flow meter sends a signal to the automatic sampler when a predetermined number of flow pulses have been sensed by the meter.

Flow-proportional samples can be collected by:

- continuous sampling at a rate proportional to the flow
- collecting equal volumes of liquid at time intervals that are inversely proportional to the flow rate
- collecting samples proportional to the flow at regular time intervals

See Table 2.3.5 for examples of how to calculate flow-proportional sample volumes. To collect flow-proportional composites, you must know or be able to approximate the flow rate. If this information is not available, you may have to determine flow rate yourself.

TABLE 2.3.5

FLOW-PROPORTIONED COMPOSITE FROM GRAB SAMPLES

SAMPLE NUMBER	FLOW RATE M ³ /MIN:	AMOUNT COLLECTED AT 3 H INTERVALS (mLs):	AMOUNT REQUIRED FOR 1 LITRE (mLs):
1	30	500	142
2	28	500	133
3	32	500	152
4	25	500	118
5	24	500	114
6	26	500	123
7	22	500	104
8	24	500	114
Average:	26.4	Total:	1 000

mLs required = (flow rate at sampling time / average flow rate) x (total sample size required / number of samples)

For sample 1: $(30/26.4) \times (1\,000/8) = 142$

2.3.6 FLOW MEASUREMENT



The choice of flow monitoring equipment or technique depends on the circumstances. The type of flow monitoring equipment to take into the field should be considered in preparing for the site visit. Information on measuring flows is available in “Measurement of Liquid Flow in Open Channels” or “Measurement of Fluid Flow in Closed Conduits,” both prepared by TC 113 Committee of the International Organization for Standardization (ISO).

MEASURING FLOW IN PERMANENT STRUCTURES

Hydraulic structures, built into the channel, are the most commonly used ways for measuring the rate of flow in an open channel. The structure must be designed so that the rate of flow through or over has a known relationship with the liquid level. This known relationship allows the flow rate through the open channel to be derived from a single measurement of the liquid level.

Flows can also be measured in closed pipes or conduits. One device commonly used for this is a magnetic flow meter. Industrial facilities often have their own flow-measurement devices, such as the magnetic meter, Venturi meter and Pitot tube. Verify that secondary instruments (e.g., totalizer, recorders) are properly maintained and operative and that flow records are being kept on file.

All field personnel are advised to gather literature and manufacturer’s information from the facility during their inspection (e.g., pulp and paper mill) and kept for reference.

When using a flow meter remember to check the accuracy of the primary element, the integrater totalizer.

OPEN CHANNELS

The hydraulic structures used to measure flow in open channels are called primary measuring devices. There are two types: weirs and flumes. Many industrial facilities use weirs, flumes, or both, together with secondary measuring devices that convert liquid height into units such as gallons per day (gal/d) or cubic metres per day (m^3/d). These devices also provide records in the form of strip charts and/or totalizer meters.

A weir is a dam built across an open channel. The liquid flows over the weir, usually through some type of opening or notch. Weirs are usually classified according to the shape of the notch. The most common types are the rectangular weir, the trapezoidal (or Cipoletti) weir and the triangular (or V-notch) weir. Each type of weir has an associated equation for determining the flow rate through the weir, based on the liquid height passing over the weir.

A flume is a specially shaped section of an open channel in which the area, or the slope, or both, is/are different from that of the channel. The configuration of a flume causes the liquid to flow faster through the flume. It also changes the liquid level. A flume normally consists of a converging section, a throat section and a diverging section. The flow rate through the flume is a function of the liquid level at some point or points in the flume. The liquid height is usually measured by some type of sensor. The most commonly used types of flumes are Parshall flumes and Palmer-Bowlus flumes, although there are many other types available.

PORTABLE FLOW MEASUREMENT EQUIPMENT

Whenever possible use a flow meter. Other methods allow you to determine flow rate in partially filled pipes or channels. The appropriate method for any site will depend on channel characteristics, water characteristics, flow rate, applicable measuring methods and the accuracy requirements. Some of these methods include:

- bucket and stopwatch
- velocity and cross-sectional area
- California pipe method
- weirs
- Manning equation
- current meters

Refer to Appendices for additional details.

2.4 SAMPLING EQUIPMENT PROTOCOLS

2.4.1 AUTOSAMPLERS



Autosamplers can be used in various applications, most often in sampling industrial locations, sewers, or similar locations where a representative sample over a fixed time period (e.g. 24 h) is required. Autosamplers must be mechanically and electrically suited to the environment in which they will serve and should be easily accessible for routine inspection and maintenance. The three most important characteristics of an automated sampler are its ability to obtain a representative sample, its material composition and its temperature stability.

There are various manufacturers of autosamplers. Many of these units have a digital programming capability and are versatile enough to be used in variety of situations. Some features include:

- battery or AC power capability
- rugged construction and ability to function in adverse conditions
- the ability to control sample temperature (generally an insulated base for adding ice)
- easy to use controls and programming functions
- ease of maintenance and cleaning
- the ability to collect composite or individual samples
- a purge function (flushes the intake line between samples)
- multiplex feature (ability to distribute one sample over several bottles or more than one sample per bottle)
- minimal contact of sample with materials in the sampler
- the ability when used with flow measuring equipment to allow for flow-proportional sampling
- screened and weighted intake line

- portability and versatility (for different locations)
- high-speed peristaltic pump capable of lifting a static head of about 6 m (20 ft)

Follow the manufacturer's recommendation for setting up the autosampler. The sampler should produce sufficient volume at the end of the desired sampling period (usually 24 h).

In setting up an autosampler, take the following into consideration:

- Install the sampler as close as possible to sample a free-flowing location, preferably at a point of thorough mixing.
- If using the purge function, keep the sampler above the discharge point of the tubing to ensure that the purge cycle will completely drain the intake line.
- Depending on the analytes of interest, use a stainless steel, Teflon or plastic intake screen to avoid plugging the sampling line with suspended solids. The intake should be weighted to hold it in place in the effluent stream. During sampling, it may be necessary to clean the screens periodically (depending on solid size in the sampling medium).
- The sample receiving bottle should be plastic, glass, or Teflon, depending on the analyte of interest.
- For preservation after sampling, maintain the sample temperature at 4°C using ice or a refrigeration unit. Also keep the sample in darkness to reduce any photo-degradation of the analytes of interest.
- For some samples, preservatives such as nitric acid for metal analysis may be added directly to the receiving bottle before you start sampling.
- Use minimal lengths of intake tubing to reduce contamination of the sample by the intake line. Replace the tubing with new line for each sampling site to minimize cross contamination.
- Monitor the temperature of the sample periodically during the sampling period, particularly if the sampling area is warm.
- In very cold weather use heating cables to ensure that samples do not freeze; consider wind chill when installing the equipment.
- Keep an individual maintenance log book for each autosampler.

After samples have been collected in an autosampler, transferring samples from carboys to bottles is a potential source of error. Mix the sample in the carboy thoroughly stirring with a Teflon coated bar, magnetic stirrer, solvent rinsed glass rod, or by swirling the carboy itself to ensure that results are representative. Partially fill (about 1/3) each of the sample bottles in turn, mixing the carboy contents as frequently as possible. Just before pouring, back-swirl the carboy to stop the liquid from rotating and churn it up thoroughly, since suspended solids will tend to centrifuge towards the carboy walls. Repeat this process until all bottles are filled.

2.4.2 COMPOSITE LIQUID WASTE SAMPLER (COLIWASA)



The COLIWASA is one of the most important samplers for liquid hazardous wastes. It allows representative sampling of multiphase wastes of a wide range of viscosity, volatility and solids content. Its simple design makes it easy to use and allows for the rapid collection of samples, minimizing the sample collector's exposure to potential hazards. Samples collected by a COLIWASA are depth-integrated.

COLIWASA and similar drum samplers are available in plastic, glass, or Teflon. With some, the sampling tubes may be coupled together to

extend their reach. The plastic types may be disposable and are used to sample most containerized liquid wastes except wastes that contain ketones, nitrobenzene, dimethylformamide, mesityl oxide and tetrahydrofuran. The glass or Teflon types use borosilicate glass or Teflon for the sampling tube, with Teflon parts. Glass however, is incompatible with strong alkalis or hydrofluoric acid solutions.

The main parts of the COLIWASA are the sampling tube, the closure-locking mechanism and the closure system. The sampling tube typically holds approximately 1 L of liquid and consists of a translucent pipe 1.52 m long and 4.1 cm inside diameter, usually made of polyvinyl chloride (PVC) or borosilicate glass plumbing tube. The closure locking system consists of a sharply tapered neoprene stopper, attached to a 0.95 cm outside diameter stopper rod of either PVC or Teflon. The upper end of the stopper rod is connected to the swivel of a channeled aluminum bar. The aluminum bar serves both as a T-handle and loop for the sampler's closure system.

SUGGESTED PROTOCOL

1. Put the sampler in the open position.
2. Slowly lower the sampler into the liquid so that the liquid level inside and outside the tube is the same. If the level inside the sampler tube is lower than that outside, the sampling rate will be too fast, resulting in a sample that is unrepresentative.
3. When the sampler stopper hits the bottom of the waste container, close the shut-off valve at the bottom of the sampler. Slowly withdraw the sampler from the container.
4. Place a suitable container under the sampling tube and slowly discharge the sample into the container.

2.4.3 DEPTH SAMPLER

The depth sampler is used to collect water samples or liquid waste samples. The sampler is also used for collecting water samples at different depths in lakes, rivers or lagoons. Commercially available samplers are made of a brass, PVC, or acrylic tube designed to hold various quantities.

Large stoppers or rubber cups, which act to close off the ends of the tube when the trip head mechanism is triggered, are attached to a rod that runs through the centre of the tube. The closure mechanism can be triggered by dropping a messenger weight down the sample line.

SUGGESTED PROTOCOL

1. Open the sampler by lifting the stopper-trip head assembly.
2. Lower the sampler into the liquid to the desired depth.
3. Trigger the closure of the sampler by dropping the messenger weight down the sample line.
4. Retrieve the sampler and hold it by the centre rod to prevent the accidental opening of the bottom stopper.
5. Drain sampler by holding drain valve over sample bottle.

NOTE: In swift currents where extra weight is added, make sure that the end of the rope is secure since the sampler may be quite heavy.

2.4.4 EXTENDED BOTTLE SAMPLER

The extended bottle sampler is a grab sampler designed to sample subsurface liquids to a maximum depth of 1.5 m. It has a simple mechanism that removes and replaces the sample bottle cap while being submerged.

The extended bottle sampler consists of a 1.8 m aluminum tube with a stainless steel clamp attached to the end; this can be adjusted to hold a sample jar of the desired size. The sample cap can be removed and replaced by turning the handle grip rod, which attaches to the cap by means of a screw clamp or suction cup.



SUGGESTED PROTOCOL

1. Place an uncontaminated capped bottle in the stainless steel clamp.
2. Attach the rod to the cap by means of a screw clamp or suction cup.
3. Lower the sampler into the liquid to the desired depth.
4. Turn the handle grip rod, removing the cap.
5. Allow bottle to fill and then replace the cap.
6. Raise sampler and remove bottle.

2.4.5 DIP SAMPLER

Dip samplers are used to collect liquid waste samples from disposal ponds, pits, lagoons and effluent streams. Their purpose is to isolate you from possible danger. The sampler consists of an adjustable clamp (to hold a sample container) which is attached to the end of a handle of some sort. Commercial dip samplers are available but if you have to improvise, here are some possible handles:

- golf ball retriever
- 2 or 3 piece telescoping aluminum tube
- paint roller extension pole

Whatever the handle is, it should be convenient to use and be easy to decontaminate. Telescoping tubes or paint roller extension poles are often available at hardware or swimming pool supply stores.

SUGGESTED PROTOCOL

1. Assemble the sampler, ensure all parts are solidly secured.
2. Rinse sampling clamp and container three times with water from the sample source.
3. Slowly submerge the container with minimal surface disturbance.
4. Allow sample stream to flow gently into container with minimal splashing.
5. Repeat collection if necessary. When container is fully filled, cap tightly.
6. Remove container and store sampler head (clamp) in plastic bags for subsequent decontamination.

2.4.6 MULTIPARAMETER WATER QUALITY INSTRUMENTS



Multiparameter instruments for immediate, on-site measurement of water quality are available from several manufacturers. Typically, these instruments will measure temperature, pH, conductance, oxidation/reduction potential, dissolved oxygen, turbidity and depth. These instruments come in a waterproof housing and can be immersed in natural watercourses and reasonably slow-moving effluent streams. All provide for data logging and computer interface and can be left unattended to

operate. Most have electrochemical sensors that are specific to each parameter and these sensors must, of course, be individually calibrated against reference solutions.

Consult the manufacturer's manual for applicable operating procedures, calibration and upkeep for each instrument.

2.4.7 OPEN TUBE SAMPLER (SAMPLE THIEF)

This sampler is basically a hollow glass or rigid plastic tube that can be of any length. It is used to sample most containerized liquid wastes. The glass open tube sampler is used to sample all kinds of liquid wastes that cannot be sampled with the plastic open tube sampler. Plastic must be used for strong alkalis and hydrofluoric acid solutions.

SUGGESTED PROTOCOL

1. Insert the sampler into the material to be sampled to the depth desired.
2. Place your gloved thumb securely over open end of tube, pressing down firmly to seal the tube and carefully withdraw the sampler.
3. Insert the lower end of the sampler into a sample container and ease up the pressure of your thumb to allow sample to drain in slowly without splashing. You may stop the flow at any time by increasing the pressure of your thumb.

2.4.8 VACUUM SAMPLER (VACSAM)

The VACSAM system is a liquid waste sampling device that uses a sample tube attached to a vacuum pump. When the pump is operated, the sample is transferred from its original container to a sample container. Vacuums are particularly useful for sampling hazardous liquid wastes.

SUGGESTED PROTOCOL

1. Assemble VACSAM sampler, attaching tubing to pump and sample bottle.
2. Pump desired volume directly from container into laboratory cleaned sample bottle.

2.4.9 WEIGHTED BOTTLE SAMPLER

The weighted bottle sampler can be used to sample liquids in storage tanks, wells, sumps, or other reservoirs that cannot be adequately sampled with another device. The weighted bottle sampler can either be fabricated or purchased.



The outside of the bottle is exposed to the waste and must therefore be properly decontaminated after use.

SUGGESTED PROTOCOL

1. Lower the sampling device to the required predetermined depth.
2. Pull out the bottle stopper with a sharp jerk on the sampler line and allow the bottle to fill completely.
3. Retrieve sampler.
4. Transfer sample into laboratory cleaned sample bottles.
5. Clean the sampler and line carefully.

2.4.10 GRAIN SAMPLER

The grain sampler is used for sampling powder, granular waste or materials in bags, fiber drum, sacks, or similar containers. This sampler is most useful when the solids are no greater than 0.6 cm in diameter.

This sampler generally consists of two slotted telescoping tubes, usually made of brass or stainless steel. The outer tube has a conical pointed tip that permits the sampler to penetrate the material being sampled. The sampler is opened and closed by rotating the inner tube. Grain samplers are generally 61 cm - 100 cm long x 1.27 cm - 2.54 cm in diameter and are commercially available at laboratory supply houses.

SUGGESTED PROTOCOL

1. With the sampler in the closed position, insert it into the granular or powdered material or waste being sampled. Insert from a point near a top edge or corner, through the centre, to a point diagonally opposite the point of entry.
2. Rotate the inner tube of the sampler into the open position.
3. Rotate the sampler a few times to allow materials to enter the open slots.
4. Close the sampler and withdraw it from the material being sampled.
5. Lay the sampler flat, with the slots facing upward.
6. Rotate the outer tube and slide it free from the inner tube.
7. Transfer sample into sample containers.

2.4.11 DREDGE/GRAB SAMPLERS



Dredge/grab samplers are used to sample shallow sludge and sediment, from silts to granular materials. Maximum penetration depth is 8 cm - 10 cm. Samples cannot be collected undisturbed.

The dredge is a clamshell-type scoop activated by a counter-lever system. It has a screened or open top, allowing water to flow through freely when the dredge is in the open position during descent.

Dredges are usually constructed of galvanized steel, brass, or stainless steel. Various sizes are available. Larger versions may require a winch.

Take care in operating the dredge. Once the sampler is latched open, its jaws can snap shut like a bear trap, with enough force to cause serious physical injury.

SUGGESTED PROTOCOL

1. Attach the dredge to the required length of sample line.
2. Secure the free end of the sample line to prevent accidental loss of the sampler.
3. Open the sampler jaws until they are latched. Support the sampler by its lift line or the sampler will be tripped and the jaws will close.
4. Slowly lower the sampler to the bottom and allow the sample line to slacken. When the line slackens, the latch releases, allowing the clamshell to close when the sampler is retrieved.
5. Slowly raise the sampler clear of the surface and open the clamshell into a sample tray.
6. Thoroughly decontaminate the sampler with water and appropriate solvent before next use.

2.4.12 SAMPLING TRIER

Sampling triers are used to sample moist or sticky compressed solids or soil. A typical sampling trier is a tube about 61 cm - 102 cm long and 1.27 cm - 2.54 cm in diameter, with a slot that extends almost its entire length. The tip and edges of the tube slot are sharpened to allow the trier to cut a core when rotated in a solid material. Sampling triers are usually made of stainless steel with wooden handles. Extracting the sample from the sampler, however, can be difficult.

SUGGESTED PROTOCOL

1. Insert the trier into the solid material at an angle up to 45° from the horizontal to minimize spillage from the sampler. Tilt the sample container if necessary.
2. Rotate the trier once or twice to cut a core of material.
3. Slowly withdraw the trier, making sure that the slot is facing upward.
4. Transfer the sample into a suitable container using a spatula or brush.

2.4.13 SCOOPS, TROWELS AND SHOVELS

Soils can be sampled at or below the surface, depending on the type of information required. There are several different types of samplers that can be used to collect a soil sample.

Scoops and trowels come in different sizes and makes. Many are coated with chrome paint which may peel off and contaminate the sample. Stainless steel scoops are preferred. A trowel can be bought from a hardware store; the scoop can be bought from laboratory supply houses. An alternative sampler for small samples is a stainless steel tablespoon. Shovels are generally small, collapsible models. Include a dustpan for gathering loose or particulate solids with this equipment.

SUGGESTED PROTOCOL

1. Collect small, equal portions of sample from the surface or near the surface at specified intervals.
2. Transfer sample into laboratory cleaned sample bottles. For organic analyses, cover the mouth of the container with aluminum foil before capping tightly.
3. No preservative is generally required. Keep samples at 4°C or lower.

2.4.14 THIN-WALLED CORER (PUSH/SHELBY TUBE)

Thin-walled corers or Shelby Tubes can be used manually or with power equipment to obtain undisturbed soil profiles or sediment and sludge samples. They can also be used to collect samples through shallow overlying liquids. Samples may be extruded from the tubes for examination; alternatively, if tube liners are used, the samples may be sealed and sent directly to the laboratory in the tube liner.

Push tubes used manually are straight tubes generally 5.1 cm in diameter or less and of varying length. Larger diameter push tubes require the use of power equipment. A tapered nosepiece acts as the cutting edge of the tube. These samplers are generally constructed of chrome-plated or stainless steel and most can be adapted to hold brass or polycarbonate plastic liners.

Some thin-wall corers have a handle to make it easier to drive the corer into the matrix. Corers may also have a check valve on top to prevent wash-out during retrieval through an overlying water layer. Samples from greater depths can be obtained by first auguring a hole to the desired depth and then attaching a push tube to a sampling head connected to the correct length of push rod extension.

SUGGESTED PROTOCOL

1. Push the corer into the material to be sampled with a smooth continuous motion until the tube is full.
2. Twist the corer, then pull to withdraw.
3. Remove the nosepiece (if removable) and push the sample into the container or tray.
4. Thoroughly decontaminate the sampler by washing with water and appropriate solvent.

3.0 SAMPLING MEDIA BACKGROUND

3.1 LIQUIDS

3.1.1 SAMPLE COLLECTION SAFETY

3.1.2 SAMPLING FOR TOXICITY TESTING (BIOASSAY PROTOCOL)

3.1.3 GROUNDWATER SAMPLING

3.1.4 SEDIMENT WATER

3.1.5 WATER SAMPLES FOR METAL ANALYSIS

3.1.6 PETROLEUM PRODUCTS

3.1.7 POLYCHLORINATED BIPHENYLS (PCBS) IN TRANSFORMERS

3.1.8 OTHER LIQUIDS

3.2 SOLIDS

3.2.1 SAMPLING SOLID SURFACES

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3.2.4 DETERGENTS

3.3 GASES

3.3.1 AIR SAMPLING

3.3.2 INSTRUMENTS USED IN AIR SAMPLING

3.4 BIOTA

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3.4.2 SAMPLING WILDLIFE

3.4.3 FARM INSPECTIONS

3.5 HAZARDOUS WASTE

3.5.1 SOURCES OF HAZARDOUS WASTE

3.5.2 SAMPLING HAZARDOUS WASTES

3.1 LIQUIDS

3.1.1 SAMPLE COLLECTION SAFETY



Review THE INSPECTOR'S SAFETY GUIDE and task hazard analyses (THAs) for detailed procedures regarding sampling streamside, by boat and in confined spaces.

Prior to sampling on ice, contact Environment Canada's Water Survey of Canada office for safety requirements and training for working on ice. Water Survey of Canada offices are located within Environment Canada regional offices or via Environment Canada's web site.

Important highlights include:

STREAMSIDE

- If you need to wade into the water to collect a sample, another person must be present and you must wear a flotation device (life jacket). If you have to wade into the water to take a sample from larger streams or rivers, wear a lifeline as well as the life jacket.
- If the water is too deep or fast-moving for wading, collect samples from a boat or from shore. Do not take any risks. See THAs in THE INSPECTOR'S SAFETY GUIDE for limits for safety depth and current speed.
- If you are sampling from shore, be sure to maintain a safe footing and a solidly balanced position.

BOATING

- Be familiar with basic boating safety rules and follow all Transport Canada boating regulations. Be aware that the federal requirements for the operation of non-pleasure craft are: Small Vessel Operator Proficiency (SVOP) and the Marine Emergency Duties 03 (MED03). Please contact your local Coast Guard office to clarify your requirements.
- Always wear your flotation device while on the water.
- If possible, turn the boat engine off before collecting water samples. Samples should be collected as far away from the motor as possible and upstream of it, to avoid fuel/oil contamination.

CONFINED SPACES

NOTE: These procedures are general guidelines and do not supercede governing regulations. They must be augmented with specific procedures for each designated confined space.

- Under normal circumstances, samplers should not have to enter confined spaces. Samples can usually be obtained using sampling devices such as sampler poles, buckets and chains.
- No person shall enter a confined space or act as an assistant during an entry without proof of current Confined Space Entry training.
- If you have to enter a confined space such as a manhole to collect a sample, another person must be present and all safety issues addressed as outlined in THE INSPECTOR'S SAFETY GUIDE.
- Confined spaces usually encountered are manholes, sanitary sewage stations, sumps, holes in tanks, culverts, deep ditches, between hulls of ship and barges.
- Be cautious of running water, explosive gases and toxic fumes.

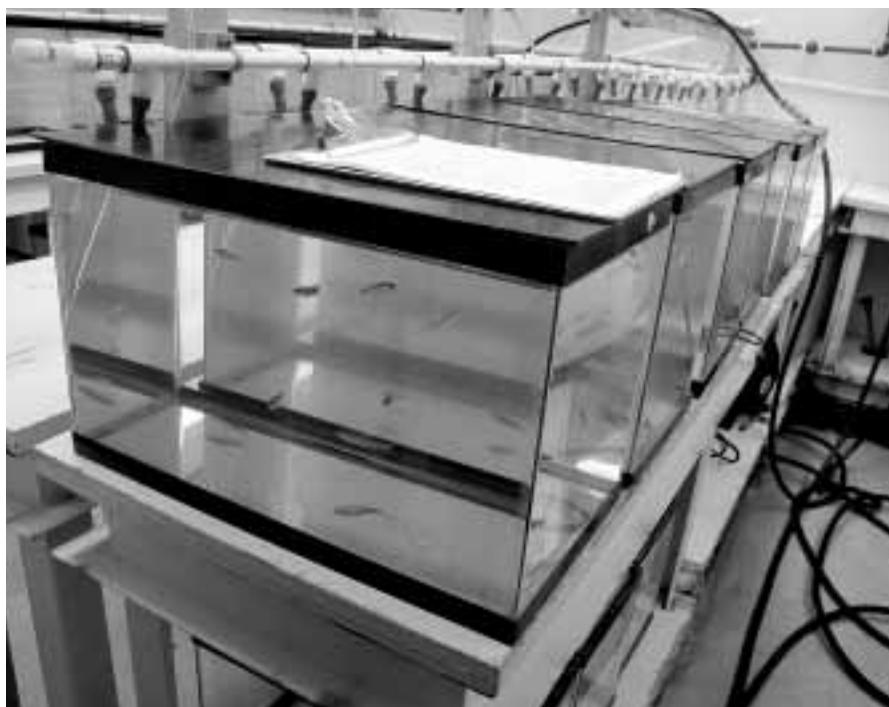
3.1.2 SAMPLING FOR TOXICITY TESTING (BIOASSAY PROTOCOL)

SUGGESTED PROCEDURES

- Samples for toxicity testing (bioassay protocol) may be collected as grabs or composites.
- Sample-volume requirements depend on fish size and numbers per test solution, loading density requirements, test concentrations and the use of replicates.
- For single concentration tests (LT_{50}), sample volumes of 25 L - 50 L or more are normally required.

OR

- For multi concentration tests (LC_{50}), sample volumes of 50 L - 100 L are normally required.



- Containers for storage and transportation of samples must be made of nontoxic material (e.g., polyethylene or polypropylene carboys or pails). The containers must be new or thoroughly cleaned and dried and should be rinsed with clean water, then with the sample to be collected. They should be filled with sample to exclude air and then sealed.
- Samples must be kept from freezing. During transport, samples should be kept in the dark and at a temperature between 1°C - 8°C. Upon receipt of sample(s) at the laboratory, the temperature of the effluent in each sample container should be measured and recorded (infrared thermometer is best used to minimize possibility of cross-contamination). That portion of each sample to be used in the toxicity test must be adjusted to +/- 15°C before the toxicity test can be started.
- Samples for *Daphnia magna* testing should be collected in plastic 1 L bottles.
- Testing of samples should commence as soon as possible after collection, refer to Environment Canada Reference Method EPS-1/RM/13. The test should begin within three days and no later than five days after termination of sampling.
- The contents of each sample container must be agitated thoroughly just before pouring aliquots to prepare solutions. Sub-samples (i.e., aliquots of a sample divided between two or more containers) must be combined.

3.1.3 GROUNDWATER SAMPLING

Groundwater sampling is conducted via wells which have been previously installed by either a Environment Canada specialist or consultant who has used a hydrogeological investigation to determine the location and depth of the monitoring well.

SUGGESTED PROCEDURES

- The monitoring well must be constructed of material that is compatible with the analytes being sampled in the groundwater. The monitoring well incorporates a protective casing which is screened and equipped with a filter to collect sediment free groundwater.
- Once drilled, the monitoring well installation must continue until the water sample collected is free of any materials that may be introduced during the well construction, and until the temperature, conductivity and pH have stabilized and the water is free of suspended solids.
- Purge the water held in the monitoring well casing prior to sampling - the well diameter will determine the volume of water required to be purged.
- Sampling equipment must be decontaminated between sampling events and bore holes.
- Parameters selected for sampling are site specific and frequency of sampling will depend on the conditions that influence the groundwater flow and movement of contaminants. Regular monitoring of field parameters will indicate the changes of water quality as it occurs and may trigger more extensive testing.

3.1.4 SEDIMENT WATER

Sediment water samples can be interstitial water, collected in the field, or pore water collected through centrifugation later in the lab. The two types of samples are used for different purposes.

Small quantities of interstitial water are often sufficient for geochemical investigations to evaluate sediment chemistry, however it is difficult to retrieve sufficient volumes of interstitial water for biological testing. Interstitial water is mostly used for academic studies, for example, to detect changes in water chemistry due to changes in temperature, pressure and exposure to oxygen, or to determine metal and major ion concentrations compared to pore water collected by centrifugation and filtration.

Pore water is collected by centrifuging sediment at 11 000 rpm. Centrifugation should be completed as soon as possible, within 24 h of sampling. For routine toxicity tests, pore water should be analyzed as soon as possible; it should be stored at 4°C no longer than 72 h and preferably not longer than 48 h.

3.1.5 WATER SAMPLES FOR METAL ANALYSIS

Decide if your water sample for metal analysis is to be determined as dissolved, extractable or total, before collecting. This decision will determine whether the sample is field filtered, if it is to be acidified with or without filtration and what type of digestion will be required during analysis.

NOTE: Consult the laboratory about the field procedures you should use.

SUGGESTED PROCEDURES

- Nitric acid (HNO_3) is used as a preservative for metal samples. This is a colorless or slightly yellow liquid with an acrid odour; it is extremely corrosive and, in concentrated form, fumes freely. Nitric acid keeps metal ions in solution from being adsorbed onto the container's walls and prevents bacterial growth. The preservative is prepared by diluting 70% nitric acid 1:1 with DI water to make a 35% solution. For mercury analysis, use potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) and nitric acid as preservatives.



- Dissolved samples are field filtered through $0.45\ \mu$ filters into 250 mL acid washed polyethylene bottles and are preserved with nitric acid.
- Dissolved, extractable and total recoverable sample fractions are acidified to $\text{pH} < 1.5$ with trace metal grade nitric acid to prevent the metals from adsorbing to the container material or precipitating out.

3.1.6 PETROLEUM PRODUCTS



Petroleum products are hydrocarbon-based materials derived from crude oil, natural gas and synthetic oils. Many petroleum products are classified as dangerous goods and have to be properly handled. Ensure that proper safety precautions are followed.

Samples may be obtained from bulk tanks, piping systems, rail tank cars, tanker trucks, tote tanks, or drums by means of sampling valves, dispensing nozzles, loading and sampling hatches and bung holes.

Generally the techniques used in sampling different types of petroleum products are the same. The exceptions are:

- Very heavy lubricating oils, which are so viscous that normal liquid sampling techniques are difficult.
- Products under pressure, such as liquified petroleum products, require specialized techniques.

WARNING!

Many petroleum products (gasoline is a good example) release vapours that will build up in confined areas. These vapours are potentially explosive. Sample unknowns only in well ventilated areas.

SUGGESTED PROCEDURES

- Wear flash-resistant coveralls such as Nomex.
- Ensure that environmental conditions are safe; no lightning storm in area, there is nothing nearby that could ignite the fumes and that no one in the area is smoking.
- Equipment is safe and electrically grounded, to prevent static build-up. See grounding procedures in THE INSPECTOR'S SAFETY GUIDE.
- That the area is well ventilated.
- Sampling bottles and equipment cannot be degraded by petroleum solvents.
- Label sample bottles before sampling; labeling and etching is difficult on a bottle coated with petroleum, do not write directly on bottle, ink may wash off.
- Use dedicated containers (including coolers) and sampling equipment; do not use these for any other sampling.



CLEANING OF SAMPLE CONTAINERS

If not using pre-cleaned containers, wash each container with a warm detergent mixture followed by 6 hot water rinses, rinse twice with DI water and then twice with pesticide grade acetone and pesticide grade hexane. Retain solvent washings for appropriate waste disposal. Allow the jars to air dry thoroughly. Record cleaning method in your notebook.

CAUTION: Mason jars lined with aluminum foil lids have been noted to leak. They should only be used if the 120 mL Teflon lined containers are not available

CAUTION: HDPE containers and plastic bags must not be used for sample collection since plastics may dissolve in oils or permit volatile hydrocarbons in oils to diffuse through the container walls. In addition, plastizers used in plastics can contaminate samples and interfere with subsequent oil match analysis.

SAMPLING STORAGE TANKS

- Large storage tanks (particularly at refineries) may be equipped with sample taps that draw the product from several levels in the tank, to compensate for stratification. Ask company personnel to operate these taps whenever possible.
- Tanks without taps are usually sampled through access ports in the tank roofs.

SUGGESTED PROCEDURES

- Flush tap lines to ensure that fresh product is sampled.
- If you want to collect a composite sample, take equal portions from each tap directly into one sample bottle to form the composite.
- Deposits of metals and other additives often accumulate on the spout; ensure that none of these deposits drops into the bottle.
- Remember that while the bottles are clean, the bottle holder itself may carry contaminants.
- Lower the bottle in a weighted metal bottle holder, into the tank by means of a steel wire. Keep the wire in contact with the lip of the port to prevent static buildup.
- A second wire should be attached to the stopper in the sample bottle. At the correct depth, tug sharply on the wire to dislodge the stopper from the mouth of the bottle.
- For a depth-integrated sample, draw the apparatus up at a constant rate through the tank, allowing the bottle to fill gradually so that all levels are equally sampled. The bottle must be partially empty when it reaches the top.
- For a sample from a particular depth, allow the bottle to remain at the predetermined depth until it is full, then raise the bottle to the surface. You may want to repeat this procedure if several samples from different depths in the tank are needed.

TANK TRUCKS

Tank trucks should be sampled through top-loading hatches, never through bottom valves. Unless there is reason to believe otherwise, assume that the loading process and the churning of the fuel during transport will ensure that the load is well mixed.

SUGGESTED PROCEDURES

- Ensure the truck is grounded following the grounding procedures in THE INSPECTOR'S SAFETY GUIDE.
- Use fall protection according to THE INSPECTOR'S SAFETY GUIDE.
- Ask the driver to open the hatches. Samplers should avoid operating company equipment.
- Sample by dipping a clean sample bottle into the tank by means of a pole or line.
- To avoid static buildup, keep the pole or line in contact with the lip of the tank.

DRUMS

Drums are usually sampled using a sample thief (drum sampler). A sample thief extracts a column of product from the drum, ensuring proportional sample from every liquid level. A sample thief can be a rigid tube made of glass, Teflon, or polyethylene; commercial drum samplers are also available. The sample thief should be longer than the drum to be sampled.

If the drums contain flammable product, ensure that each drum has been properly grounded before collecting samples.

SUGGESTED PROCEDURES

- Ensure the sample bottle has sufficient volume to hold the contents of the sampler.
- Insert the drum sampler through the bung hole straight to the bottom of the drum.
- For viscous samples, lower the sampling tube slowly.
- Stopper the top end with your (gloved) thumb or close the valve if tube is equipped with one.
- Pull the tube smoothly out; insert the other end in the sample bottle and allow the sample to drain out.

Drums may be sampled using transformer pipettes if the contents of the drum are known to be well mixed.

GASOLINE AND DIESEL FUELS

Gasoline samples, which are collected at retail outlets, bulk stations, or directly from drums for lead and phosphorus analysis, are usually taken in acid-washed, 125 mL amber glass bottles with Teflon-lined caps.

SUGGESTED PROCEDURES

- Collect samples directly into the bottle.

OR

- Take samples into a small CSA approved jerry can and transfer a portion to the amber glass container.
- Do not fill the glass containers completely. Allow 1 cm headspace for expansion.
- Put samples in well sealed containers and keep them at 4°C (generally in a cooler on with freezer packs) to minimize the escape of vapours.
- Do not keep coolers in the passenger compartment when transporting samples. If this is impractical, keep the vehicle's windows open.
- Legal samples should be collected directly into the bottle.
- Take great care not to overflow the bottle and keep absorbent pads on hand to soak up any spills. Wear protective gloves and eyewear.

For additional information on shipping gasoline samples or transporting them in your vehicle, refer to 4.3.2: Dangerous Goods.

There are some exemptions under the Transportation of Dangerous Goods Regulations (TDGR) that may apply, but since each situation will differ, samplers should consult with the TDGR. When transporting gasoline samples, it is recommended that each package contain no more than 5 L. This is the maximum net quantity allowed per package in a passenger road vehicle, under Schedule II, Part II of the TDGR, unless the gasoline is carried in an "open vehicle" (see s. 2.31 of the TDGR). If jerry cans are used for sampling, they should be secured in the vehicle.

CONTAMINATED FUELS

Contaminated fuels are tested for metals, PCBs and halogenated organics. Sampling techniques are the same as the ones used for general petroleum products.

Gasoline is volatile at ambient temperatures. The liquid is highly flammable and mixtures of gas vapours with air are explosive. Direct skin contact and inhalation of vapours are a health hazard.

3.1.7 POLYCHLORINATED BIPHENYLS (PCBs) IN TRANSFORMERS

In general, samplers should not collect polychlorinated biphenyls (PCBs) samples from transformers if company or utility personnel are available to do so. If you must collect a sample, exercise extreme care.

WARNING!

- Ensure that the transformer is off line and de-energized by a qualified electrician or other responsible person.
- Be aware that a substantial electrical charge will persist for some time after the transformer is off-line. Allow time for this charge to dissipate.
- Be careful of other energized electrical equipment nearby. There should be at least 3 m between transformers. Electricity can arc at least a meter from nearby energized equipment, such as other transformers, bus bars, etc.
- Be careful when working in tight areas; do not accidentally come into contact with other live equipment.

If these conditions cannot be met, do not attempt to sample the transformer.

Because transformers are often located in secured, out-of-the-way locations, access may present a problem. For pole mounted transformers, a power-operated scissor lift or cherry picker may be needed. In other cases, the transformer may be in an underground cell.



Take measures to prevent spills; to contain spills if they occur use plastic sheeting and absorbent pads. Once the power source to the transformer is cut and spill control measures are in place, the cover of the transformer can be removed using hand tools.

A sample of the dielectric fluid is most efficiently obtained using a disposable glass COLIWASA or similar sampling device. To obtain a representative sample:

- Lower the COLIWASA or other sampler into the fluid slowly, allowing the levels of the fluid inside and outside the sampler to remain the same.
- When the sampler reaches the bottom of the transformer, close it.
- Pull the sampler out, wiping the outside dry with a disposable absorbent pad.
- Transfer the sample directly into the sample bottle.
- Drum the sampler, along with protective clothing, sheeting and absorbent pads and dispose of them at a predetermined approved location.

Storage of PCB Materials, PCB Waste Export Regulations:

- Samples of chlorobiphenyls are taken from transformers, capacitors, light ballasts, paint pigments, oils and other materials that may contain PCBs. If the equipment or machinery is small (such as small capacitors or light ballasts) the equipment may be sent to the laboratory intact; otherwise try to obtain a subsample of the oil or solid contained in the equipment.

WARNING!

It is best not to use the transformer drain valve for sample collection for two reasons. The valve may be rusty and break off or become jammed in the open position, allowing the dielectric fluid to spill. The transformer contents may have stratified. Since PCBs are heavier than other insulating oils, samples obtained from the valve near the bottom of the transformer might reveal higher than representative PCB concentrations.

3.1.8 OTHER LIQUIDS



Samples of volatile organics are usually taken in 40 mL amber vials with Teflon-lined septum caps. The septum cap allows the laboratory to insert a syringe into the vial and obtain the volatiles without removing the cap. The vial should be filled to the point of overflow at the mouth of the vial. This provides excess sample that is squeezed out as the sample is capped and ensures that there is no air headspace. Do not try to composite samples for volatile compounds; the analyte of interest may evaporate during the mixing process.

Oil and grease samples are usually not composite, as the oil and grease tends to remain on contact surfaces such as sampling equipment and transfer containers. Composite samples are usually done in the laboratory, since the individual sample containers can be extracted to remove any residual oil and grease. Wherever possible, laboratory containers should be submerged directly in the waste using an extension pole while avoiding floating solids.

Liquids subject to Ozone Depleting Substances (ODS) Regulation are not usually found in large containers, but rather in small retail products. Small retail products are sent to the laboratory for analysis. Some products may come with a dispenser, if so, they should be purchased for use by the lab. If you are required to sample large containers, you will require specialized expertise.

3.2 SOLIDS

This section presents methods for taking samples from solid surfaces, as well as techniques and suggestions for sampling sediments, soils and detergents. Samplers should also refer to the Environment Canada report "Guidance Document on Collection and Preparation of Sediments for Physiochemical Characterization and Biological Testing," Report EPS 1/RM/29, 1994.

3.2.1 SAMPLING SOLID SURFACES

SAMPLING TECHNIQUES FOR SOLID SURFACES

The three sampling techniques described in this section all have the following in common:

- Pre-measure a 25 cm x 25 cm area for sampling.
- Use disposable protective gloves and change them before handling clean sampling equipment or containers.
- Use wide-mouth sampling bottles.

In choosing sampling points for these techniques, take into consideration the site history, manufacturing processes, obvious contamination and available surface area.

WIPE SAMPLES

Wipe samples are used to monitor superficial contamination (e.g., PCBs, dioxins) on non-porous surfaces such as metal and glass. Suggested sampling points include process vessels, ventilation ducts and fans, exposed beams, window panes, etc.

To collect a wipe sample, use:

- sterile wrapped gauze pads, 7.6 cm x 7.6 cm
- an appropriate solvent

The use of filter paper for wipe sampling is not recommended. The solvent of choice will depend on both the analytes of interest and the surface being sampled. Generally pesticide-grade hexane is the preferred solvent. Occasionally, however, the desired samples must be taken from painted or waxed surfaces and since hexane may degrade the finish or pick up interfering substances, an alternate solvent should be used. In this case, alcohol or DI water may be acceptable substitutes, depending on the desired analyte's volatility.

SUGGESTED PROCEDURES

- Create a wipe blank by wearing gloves, wet a gauze pad with a measured amount (approximately 15 mL - 20 mL per sampling pad) of solvent and put the pad directly into a sample container and cap container.
- Take a sample by soaking a second gauze pad in a measured amount of solvent.
- Stroke the entire pre-measured area twice firmly with the pad: once in one direction, once at right angles to the first direction.
- Put the pad in the second sample container and cap the container.
- Label both containers.

CHIP SAMPLES

Chip sampling is used to monitor non-volatiles such as PCBs, tetrachlorodibenzo-para-dioxin (TCDD), or tetrachlorodibenzo furan (TCDF) on porous surfaces such as cement, brick, or wood. Suggested sampling points include floors near process vessels and storage tanks, loading dock areas, etc.

To collect a chip sample, use:

- A decontaminated chisel and hammer or electric hammer.
- A dedicated natural bristle brush and a dedicated decontaminated dust pan constructed of a pre-approved material which will not interfere with the contaminants of concern.

Locate your sampling point, measure and mark it off, then:

- Wearing a new pair of disposable gloves for each sample and using decontaminated tools, break up the pre-measured surface to be sampled. Try to avoid scattering pieces out of the sampling area boundary. Follow safety procedures (e.g., safety glasses/goggles).
- The area should be chipped to less than 6 mm and preferably to 3 mm. Record how deep chips were taken.
- Collect the chipped pieces using a dedicated decontaminated dust pan and natural bristle brush and transfer the sample directly into the bottle.

SWEEP SAMPLING

Sweep sampling is used for non-volatiles such as PCBs, TCDD and TCDF in residues found on porous (e.g., asphalt) or non-porous (e.g., metal) surfaces. Collecting dust or residue samples may help in determining the nature and amount of contamination. Suggested sampling points include floor surfaces near process vessels and storage tanks. Examples might be a linoleum floor where a solvent cannot be used or too much residue exists for a wipe sample to be easily collected, or a street gutter where contaminated sediments may have migrated and accumulated.

To collect a sweep sample, use:

- A dedicated natural bristle brush.
- A decontaminated spatula and/or a dedicated decontaminated dust pan constructed of a pre-approved material which will not interfere with the contaminants of concern.

Locate your sampling point, measure and mark it off, then:

- Wearing a new pair of disposable gloves for each sample and using decontaminated equipment, sweep all residue in the measured area to be sampled into a decontaminated or dedicated dust pan.
- Using a decontaminated or dedicated spatula, transfer the sample into the sample bottle.

3.2.2 SEDIMENT



Sediment from surface layers usually reflect recent contamination. For this reason, the top 2 cm is often the layer of interest. The usual technique is to take core samples and extract the uppermost 2 cm for analysis. In such cases, the recovered core should include the original interface between the sediment and the water, to ensure that the sampler was not inserted too far into the sediment. This topmost layer can usually be recognized by the loose, flocculant, uncompacted layer of sediment which often has traces of benthic invertebrates.

The volume of sediment required depends on the end use. Each chemical and biological test requires a specific amount of sediment. Check with the lab to determine what volume to collect and what steps to take to preserve the sample. With small samplers such as KB corers, several cores may be required to collect the necessary volume of sediment/soil for analytical purposes.

Once collected, sediments should be transferred using a clean, solvent-washed, heat-treated spoon into proper glass or polyethylene containers with Teflon or aluminum-foil lined lids. After labeling, all sediment samples should be frozen until analysis.

Some special considerations for sediment sampling:

- In core sampling, aim for a minimum penetration depth of 5 cm - 6 cm.
- Grab samples may be collected using a Ponar or Ekman dredge with a maximum 8 cm - 10 cm depth.
- When samplers require a winch, the winching system must control the rate of both the descent and ascent of the samplers.
- The sampling vessel or platform should be stationary and as stable as possible.
- Between collections thoroughly rinse the sampler with water and organic solvent if the sample is intended for organic analysis.
- Sampling equipment used to collect and handle control or reference sediments should be kept separate from those used for test sediments. If this is not possible, clean the equipment thoroughly following the proper procedures between stations.
- In situ collection of interstitial water is recommended for geochemical investigations; for routine toxicity testing, pore water should be extracted in the laboratory using centrifugation of collected sediments (see Section 3.1.4).

Sediments may contain hazardous substances. Avoid all skin contact with sediment by wearing protective clothing, gloves, boots and lab coats or aprons during sampling and sample handling.

In collecting sediment and dredge materials:

- Obtain containers for the samples from the laboratory contracted to conduct the required analyses.
- Avoid unnecessary contact with clean glassware and utensils. A clean stainless steel or Teflon spoon or scoop is ideal for transferring samples.
- Handle containers by the outside only. Do not touch the inside of the jar, or Teflon (or foil) liners and use spoons or scoops by the handles only.
- When removing a foil or Teflon liner and lid from a jar, remove as one piece and put down on a piece of foil or other clean surface while filling the jar.
- If using a grab sampler, remove the water from the surface of the grab, taking care not to disturb the contents if possible. Use the clean scoop to remove a portion of the sediment from the middle of the sampler down to the depth of the material caught in the grab. The material in the sample jar should be representative of the material sampled.

- Alternatively, you may collect the sample by removing the container lid, holding the jar by the sides and scooping the sample from the sediment without the use of other utensils. Wipe the threads of the jar clean with a paper towel before replacing the lid. Do not fill the jar more than 3/4 full, allowing plenty of room for mixing/stirring and expansion during freezing.
- When the sample is in the jar, replace the Teflon/foil liner.
- To clean the sampling spoon or grab in between samples, wipe with a paper towel to remove any solids, then rinse with water. Clean the tray in this manner as required.

3.2.3 SOIL



Surface sampling of soil usually involves collecting samples at a depth of 0 cm - 15 cm, depending on soil parameters and the nature of the investigation. One of a wide variety of tools may be used: trowel, scoop, bucket auger, soil coring device, waste pile sampler, power auger, split spoon sampler, or Shelby tube sampler. For samples at lower depths, a clean bucket auger or power auger may be needed to dig down to the

point of collection. The use of a drill rig and split spoon sampler may be necessary for samples at depths of greater than 1 m.

For biased sampling, collect samples from obviously contaminated, stained soil. For systematic samples, divide the area to be sampled into a grid, based on statistical considerations. Take at least one sample at each intersection in the grid. Alternatively, choose sampling locations in the grid using a random numbers table.

Important points during soil sample collection:

- Try to maintain the physical form and chemical composition of the sample as much as possible.
- The tool used to dig the hole must be uncontaminated; use a different, clean tool to take the sample.
- Transfer the sample to a container as quickly as possible, with no mixing, to avoid the loss of volatile fractions.

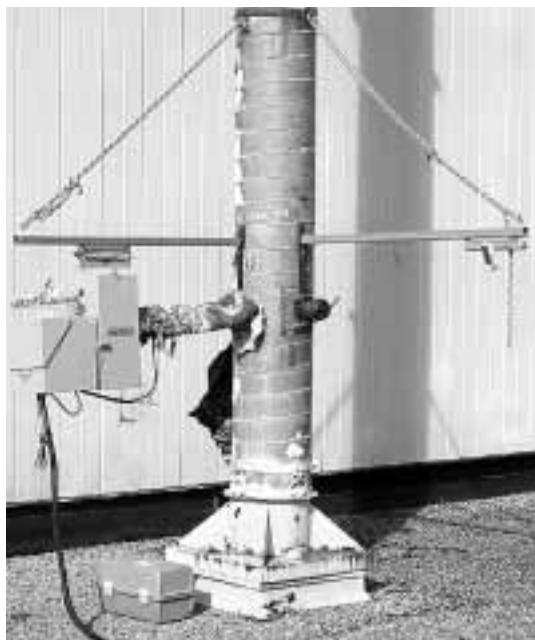
3.2.4 DETERGENTS

The Phosphorus Concentration Regulations require detergents to be tested for phosphorus as phosphorus pentoxide (P_2O_5) or elemental phosphorus (P). Samples may be collected from manufacturers, formulators or retailers in unopened boxes. The laboratory doing the testing will take subsamples as necessary. Samples may also be taken from bulk detergent storage bins or the production line at manufacturers or formulators. These are generally composite samples taken from different locations in the bins or on the line.

3.3 GASES

3.3.1 AIR SAMPLING

Sampling protocols for air sampling tend to be complex and technical. For these reasons, air sampling is usually carried out by experienced contractors or trained specialists.



Inorganic and organic gases can be sampled using grab techniques, using bags or evacuated canisters, but these must be sent to specialized labs for analysis. A knowledge of wind speed and direction, the area of sampling and other variables are all necessary for effective sampling. Meteorology and site selection play important roles in any ambient air sampling.

Sampling for inorganic pollutants (e.g., sulphur, ozone, carbon mono- or dioxide, NO_x) in ambient air generally relies on fully automated, non-portable, continuous analyzers with electronic data logging.

These analyzers use designated

U.S. EPA reference methods and must be routinely calibrated under field conditions, using span materials certified by the National Bureau of Standards. Portable analyzers include calorimetric and electronic devices.

Airborne particulate may be collected by filtration. A wide variety of instrumentation exists for filter-based sample taking and analysis. Equipment choices depend on the type and size of the particles to be collected, as well as the environment to be sampled. Settleable particles constitute a separate category and are usually collected in stationary open-top containers over a prolonged period.

Sampling for polycyclic aromatic hydrocarbons (PAH), PCBs dioxins and other toxic substances can be done with absorbents or polyurethane foam cartridges; analyses are carried out in specialized labs.

VINYL CHLORIDE AND SECONDARY LEAD SMELTER RELEASE REGULATIONS

Samplers in most regions will not be doing the actual stack sampling but will witness the sampling by other qualified persons; either plant personnel or a staff member from the Environmental Technology Centre Laboratories, 335 River Road, Ottawa, Ontario, K1A 0H3.

AIR SAMPLING TECHNIQUES

Grab sampling involves collecting a sample of air from the area of interest over a short period of time. This is usually done by means of an evacuated chamber (e.g., air sampling canister) which when opened, fills with the air to be analyzed. Closing the container makes an airtight seal. Another technique is to use a pump system to take a pressurized sample.

Sorbent sampling collects contaminants by drawing air through specific sorbent materials which trap the contaminant of interest. Different materials are used for different contaminants. The volume of air drawn through the sorbent material must be measured and recorded to determine concentration.

Particulate sampling is a filtration method; the filter in this case may be paper or glass fibre and collects airborne particles. The volume of air filtered must be measured to determine concentration.

3.3.2 INSTRUMENTS USED IN AIR SAMPLING

Since samplers will be witnessing stack sampling, here is some information on the equipment and methods in use.

When the amount and type of organic vapours are unknown, instruments such as a portable photoionization meter detector or a portable flame ionization detector, operated in a total readout mode or as a chromatography, should be used to detect organic vapours. Until specific constituents can be identified, the readout indicates total airborne substances to which the instrument is responding. When specific constituents have been identified and the instruments are calibrated correctly to measure these constituents, readings in ppm can be obtained with some degree of confidence.

Very high readings on portable photoionization meter or portable flame ionization meter may also indicate the possible displacement of oxygen or the presence of combustible vapours. The portable flame ionization meter has no measuring capacity for inorganic vapours or gases, while the portable photoionization meter's detection capacity for these vapours is limited. The meters have other limitations as well. Users should consult the operating manuals for information on what the meters can and cannot detect.

Oxygen meters are used to monitor oxygen levels in the air, usually about 20% by volume. In general, if this level falls below 19.5%, respiratory equipment will be needed. Concentrations of about 25% carry a risk of combustion. If the meter reports a drop in oxygen levels, this could reflect either the consumption of oxygen (by combustion or reaction) or an increase in the levels of some other gas. Oxygen meters are also affected by temperature variations and by high levels of carbon dioxide, which can shorten the lifespan of the oxygen sensor. VHF and UHF radio transmission signals can interfere with the instrument and produce false readings. Strong oxidizing chemicals such as ozone or chlorine can also interfere with oxygen meters.

Combustible gas indicators (CGIs) measure the concentration of a flammable gas or vapour in air. At or below the lower explosive limit (LEL) of a combustible gas or vapour, the concentration is too low to support combustion. Above the upper explosive limit (UEL), the mixture is too rich to support combustion. At concentrations between the two, the vapour will ignite if brought into contact with a spark or flame. CGIs are affected by temperature, VHF and UHF radio transmissions and oxygen deficient atmospheres. Organic lead vapours, sulphur compounds and silicone compounds will foul the instrument, acidic gases can corrode it.

Colorimetric air sampling tubes (detector tubes) provide a quick, inexpensive and fairly reliable method of sampling the atmosphere for a wide range of gases and vapours. Tubes are available for measuring over 150 different atmospheric hazards. Detector tubes are glass tubes filled with treated chemicals (reagents); they have break-off tips. During use the tip of the tube is snapped off and the tube is attached to a pump. The pump pulls a sample of the atmosphere through the tube. If the appropriate gas or vapour is present, the chemical layer in the tube will change colour. The intensity/length of the coloured band helps to indicate, at least roughly, the concentration of the gas or vapour in the atmosphere.

Variation exists between tubes made by different manufacturers and the reaction in a tube may be interfered with by chemicals similar to the one being tested for. The tubes are also affected by high humidity and have a limited shelf life.

HIGH VOLUME AIR SAMPLERS

These samplers require expertise and should only be operated by people who are technically qualified. The following section is primarily for information.

High volume air samplers consist of a vacuum motor attached to a filter and housed in a shelter that protects the equipment while allowing air to flow through freely. These samplers are used to trap suspended particulate matter (coal dust, fly ash, smoke, etc.), an index of air pollution, as one basis for ambient air monitoring.

Typically, the sampler has a filter $17.8\text{ cm} \times 23.9\text{ cm}$ or 406 cm^2 , through which air is drawn at the rate of $1.1\text{ m}^3/\text{min}$ - $1.5\text{ m}^3/\text{min}$. The filter picks up particles $25\text{ }\mu$ - $50\text{ }\mu$ in aerodynamic diameter, depending on wind speed and direction. The size of the opening to the filter and the volume of air filtered over a given time affect the size range of the particles collected. Therefore samplers should have uniform sample air inlets that allow an air flow rate of between 20 cm/seconds - 35 cm/seconds , ideally $25\text{ cm} \pm 2\text{ cm/seconds}$. Filters may be made of glass fibre or cellulose.

Glass fibre filters collect at least 99% of particles $0.30\text{ }\mu$ or larger; these filters have low resistance to air flow and little affinity for water. Samples collected on glass fibre filters are suitable for a wide variety of analyses: organic pollutants, inorganic contaminants, trace metals and several nonmetallic substances, and are excellent for monitoring gross radioactivity. Spectro-quality grade filters have low metal content and are suitable for metal analysis.

Glass fibre filters present two problems: it is not possible to sample for materials that are already present in the filters in substantial amounts; and they can take up enough water to distort final weight values. To control for the first problem, a statistically significant random sample of new filters should be analyzed to determine whether the filter blank concentration is high enough to interfere with a specific analysis. This is particularly important in making purchasing decisions. Cellulose filters, while less common than glass fibre filters, have low metal content, making them suitable for metal analyses. They do, however, have a number of drawbacks. They clog rapidly, absorb water irreversibly and produce nitrate and sulphate artifacts. These problems can be compensated for by using a control blank filter.

Handling filters requires great care. They should be:

- tagged for identification
- inspected visually for holes, tears, or other imperfections
- kept from being folded or creased
- conditioned in a controlled environment (15° - 30°C varying no more than $+ 3^\circ\text{C}$; relative humidity $< 50\%$, with no acid/basic gases) for at least 24 h before use
- weighed to the nearest milligram before installation

Read the equipment manual for the procedures for installing the filter.

OTHER PROBLEMS

- Volatile particles may be lost during sampling, shipment and storage before the filter is weighed. These losses are largely unavoidable and the filter should be reweighed as soon after conditioning as possible.
- The glass fibres themselves are alkaline and will react with acidic gases in the sample air, giving artificially high results. This effect usually happens early in the sampling period and depends on the composition of the filter and the levels of acid gas. The error introduced is probably small, but may be a problem when relatively small amounts of particulate are encountered.
- While glass fibres are comparatively insensitive to humidity changes, the collected particulate may take up water.
- Great care must be taken in handling the filter between the pre- and post-sampling weighings to avoid accidentally losing filter fibres, which would affect the results.
- Wind can deposit particulate on the filter outside the sampling period. Improper recording, power failure, or malfunctions can lead to inaccurate timing of the sampling period.
- Unless the exhaust air is directed away from the intake (at least 40 cm away), the exhaust may recirculate through the sampler, taking with it exhaust gases from the motor.

3.4 BIOTA

Biological samples are more difficult to collect than other samples, since many of the organisms collected are mobile. Biological sampling usually involves the collection of fish, although some studies rely on sampling freshwater clams, macrophytes, phytoplankton and zooplankton. The collection of biota requires specialized expertise that may be available elsewhere within the department or in other agencies, such as the Department of Fisheries and Oceans or provincial fish and wildlife authorities. You may need special permission from these agencies to use techniques such as electro-seining.

3.4.1 BACTERIAL SAMPLING

Bacterial sampling is usually carried out by provincial or federal health officials with proper training in aseptic (sterile) technique. It is extremely important that prior to bacterial sampling you have training in aseptic technique.

- Aseptic technique is necessary not only to ensure your safety when dealing with microorganisms, but also to prevent the contamination of the sample with environmental organisms.
- Contamination can come from the air and surfaces of the area that you are sampling as well as your hands or mucous.
- Never touch the sample or the inside of containers with your bare hands, or sneeze or cough into the sample.

Prior to undertaking any sampling for microorganisms, ascertain the biosafety level of the facility to determine the level of personal protective equipment (PPE) required. Biosafety levels are defined in Health Canada's Laboratory Biosafety Guidelines, 2nd Edition 1996. Biosafety levels are based on the risk group of the biological agents present

at the facility. There are four biosafety risk levels, with a level 4 rating for facilities handling the most dangerous pathogens.

- Containment level 1 PPE: includes safety footwear (never wear open-toed shoes), safety glasses and disposable surgical gloves.
- Containment level 2 PPE: add lab coat or coveralls, NIOSH-N95 disposable face mask, head cover.
- Containment level 3 and 4 PPE: contact local CFIA or Health Canada Office for inspection assistance.

Check your equipment before sampling in order to prevent contamination by the equipment.

- The packaging for disposable, sterile items should be intact.
- Reusable items should have been autoclaved beforehand and put in their proper containers.
- Sample containers should have been autoclaved beforehand and therefore sterile.
- Any contamination prior to the field sampling will result in a compromised sample.
- A field blank and travel blank need to be provided. These blanks are used as controls to check if contamination has occurred. The field blank will be opened when the samples are being taken, but nothing put in it during sampling. This will aid in determining if contamination occurred during sampling. The travel blank will remain closed throughout and will be used to check for contamination during transport, handling or both. See Part 1.5.4 - 1.5.6 for further information on QA/QC.
- All bacterial samples are treated as legal, requiring appropriate labels, markers and documentation. Therefore, take extreme care that labels are accurate and the chain-of-custody form is filled out properly.
- Know where disposable items are to be disposed of after use.
- Wipe the exterior of the sample bottle with a broad spectrum disinfectant, such as Virkon or Wescodyne.

SAMPLING LIQUID

- Ideally, the liquid should be well mixed so that the sample will be an accurate representation of the contents of the liquid.
- Do not allow the sampling equipment to touch the bottom, sides or opening of the container in order to prevent contamination.

- Take the sample from below the liquid surface, as the surface is likely to be contaminated by the air.
- Ensure that you transfer the sample to the container before wiping excess liquid from reusable sampling equipment so as to avoid sample contamination. Make sure that reusable equipment is returned to the appropriate container.

SAMPLING SOLID

- If a sealed, labeled package of the sample is available and is easily transportable, it should be taken to the lab intact.
- Take care not to send dust or debris into the air if a sample must be taken so as not to compromise your safety.
- If possible, take the sample from an unopened container or bag. Sampling equipment exists to allow a sealed bag to be punctured with minimal damage and a sample taken. The hole can be easily resealed.

SENDING SAMPLES TO THE LAB

- The sample should be secured, labeled and placed in a sealable plastic bag before being chilled with freezer packs.
- As noted above, all samples are treated as legal. Therefore, proper labeling is essential. Ensure that the chain-of-custody form is properly filled out and that the sample is secure under lock and key to prevent tampering.

It is highly unlikely that you will be asked to sample anything that contains an infectious substance as defined under the Transportation of Dangerous Goods Regulations (TDGR), so samples can be treated as any other exempt sample.

Furthermore, unless the organism in question falls under containment level 3 (with certain exceptions) or containment level 4, the infectious substance is exempt from the TDGR provided that they are properly contained. In this case, the methods of containment for the samples are the same for infectious substances as non-infectious substances:

- In a type 1B means of containment that is in compliance with CGSB-43.125; consists of a watertight primary receptacle, a watertight secondary packaging, absorbent material between the primary and secondary packaging and a strong outer packaging. type 1B packages must display the symbol "TC-125-1B" and the name and address or symbol of the manufacturer of the package. This type of packaging can be used for either infectious or non-infectious substances (other than for containment level 3 or 4 microorganisms).

- In a type 1C means of containment that is in compliance with CGSB-43.125; usually used for the transportation of biomedical waste in risk group II or III. This type of packaging can only be used with non-infectious samples and tends to vary in form. You are unlikely to use type 1C packaging.
- In a means of containment that is designed, constructed, filled, closed, secured and maintained so that under normal conditions of transport, including handling, there will be no release of the substances. This type of packaging can be used with either infectious or non-infectious substances.
- For more information, consult the TDGR.

FLOWING AND STANDING WATER

Samples can be collected either by sampling directly by holding the sterile 250 mL container in the hand, or by using a sampling device. Remove the lid without touching the inner surface of the lid or the mouth of the bottle. When sampling by hand, dip and hold the sterile container into the current away from the sampler at a depth of 15 cm - 30 cm. If there is no current, an artificial current may be created by moving the bottle horizontally away from the sampler. When removing the bottle from the water, tip the container upwards to allow for approximately 3 cm - 5 cm air above the water level so that adequate mixing can be done at the laboratory.

WATER TAP

Prior to sampling, flush the tap for approximately 2 min - 3 min. Then, remove the sample container cap and hold the container under the water tap until container is filled. Do not rinse sample bottle. Replace the cap without touching the neck of the bottle.

NOTE: Sodium thiosulphate may be added to eliminate any free chlorine in the water. Bacteriological determination should start as soon as possible after sample collection, preferably within 1 h and not more than 6 h after sample collection. Samples must be chilled with freezer packs, but not frozen, during the time between collection and filtration.

3.4.2 SAMPLING WILDLIFE

Samples from small fish species (forage fish) are pooled to provide adequate sample sizes. Larger animals may require dissection at the lab so that different tissues may be analyzed. Store samples in aluminium containers or aluminium foil (for organics analyses), plastic bags (for metal analyses) and freeze until analysis.

SAMPLING FOR FISH KILLS

Fish kills may result from natural or human activities. Natural causes include temperature change, storms, ice and snow cover, decomposition of natural materials, salinity change, spawning mortalities, parasites, bacterial and viral epidemics. Man-made causes include municipal or industrial waste discharges, agricultural run-off or activities, water-level management (such as hydroelectric dams) and chemical or other spills.

A field investigation of a fish kill consists of:

- visual observation
- collecting samples of fish, water and other biota
- physical measurements of the environment

In preparing for a field investigation, study area maps and determine the zone of fish mortality and the access to it. If surveillance of a particular body of water or area is involved, have present plans and equipment available on a standby basis. Identify waste dischargers. Contact participating laboratories regarding the number and types of samples that will be submitted, types of analyses required, method of sample shipment and date by which results are to be reported.

On all fish kill investigations, take:

- thermometer, dissolved oxygen test kit, conductivity and pH meters
- general chemical kit, biological sample gear, sample bottles and other specimen containers

SUGGESTED PROCEDURES

- Include at least one person in the investigation team with experience in investigating fish kills. Local observers may be useful guides to the area.
- Collect water samples from both polluted and unpolluted areas. As a minimum, measure temperature, pH, dissolved oxygen and conductivity. Make additional tests depending on suspected causes of the fish kill.
- Record observations on water appearance, stream flow and weather conditions.
- Colour photographs are valuable in recording conditions.

COLLECTING BIOTA IN SPILLS OR EMERGENCIES

Usually, your role is to collect water or other environmental samples, while federal or provincial wildlife authorities are responsible for collecting dead animals. If, however, circumstances require that you collect dead or dying animals, consider the following:

- Getting to the scene promptly, before the evidence has decomposed or drifted away is vital.
- In the case of fish kills, collecting dying or recently dead fish is critical, but for purposes of comparison, also collect healthy fish from an unaffected area if possible.
- Do not freeze samples for pathology. If fixatives are unavailable, place samples in plastic bags on ice and rush to the pathologist.
- Try to estimate the total number of dead and dying animals as accurately as you can.
- Birds killed by an oil spill should be tagged and put in sample bags large enough to accommodate the bird. Label them appropriately and freeze them if they are to be held for an extended period of time. Depending on the analysis required, it may only be necessary to clip oiled feathers and send them to the lab for analysis.
- If you need to take vegetation samples, collect them separately, using shears or other tools as necessary. Try not to bruise the specimens. Put them in pre-cleaned sample containers and label appropriately.

3.4.3 FARM INSPECTIONS

When conducting farm inspections you should follow bio-security procedures taken by practicing veterinarians.

Equipment or supplies recommended for staff and field vehicles:

- rubber boots that can be cleaned and then reused; or disposable
- heavy gauge garbage bags
- washable/reusable coveralls or disposable coveralls
- hair nets
- disposable or washable rubber gloves
- antibacterial waterless soap
- disinfectant in solution (e.g., Virkon)
- water for additional solution if needed
- pail or tub with lid to contain disinfectant solution
- long handled scrub brush
- paper towels
- antibacterial handy wipes

The following are basic steps to follow with a farm visit:

- Park your vehicle away from the barn area. Some farms may not allow you to drive in past a certain point, but most will not control the laneway. Park by the house.
- If going near the barn/barnyard to inspect, change into clean coveralls.
- Then put on disinfected boots (disinfect in and out).
- Although it is unlikely that samplers would be required to go into the barn, be prepared to have a shower and wear a helmet or hair net if required.
- When done, brush boots in disinfectant first, then remove coveralls.
- Some operators may provide disposable clothing and keep it for disposal.

Avoid unnecessary contact with livestock, their manure and feed. Avoid recently emptied barns. Do not touch farm pets; they are in routine contact with livestock, the barn and barnyard environment.

3.5 HAZARDOUS WASTE

3.5.1 SOURCES OF HAZARDOUS WASTE



No universally accepted standardized methods exist for collecting hazardous materials. Contaminated sites, by their nature and definition, contain concentrations of pollutants that may be harmful to health. Not all sampling episodes will need special safety consideration, but well-documented and well-designed safety protocols are often critically important.

Considerable data on chemical protective clothing is available; several databases have been published that allow extensive searches to be conducted. The US Government Centers for Disease Control and Prevention database can be found at: www.cdc.gov/niosh/database.html.

Special sample hazards may affect laboratory personnel; these should be documented and passed on to analysts so that appropriate precautions may be taken. If no special safety protocols or equipment are necessary, a statement to that effect should be included in the sampling plans and submission.

Disposal of all hazardous waste should adhere to the existing municipal by-laws or provincial regulations.

In contrast to environmental samples, waste samples generally contain high levels of contaminants. They are often collected from drums, tanks, spills and areas in the immediate vicinity of an incident. Waste samples are usually considered hazardous samples.

It is essential to distinguish between environmental and hazardous samples for the purposes of choosing sampling equipment, for personal safety precautions and for complying with transportation requirements.

WARNING!

Sampling of hazardous wastes requires specialized training and equipment. Personnel not trained in the collection of hazardous wastes must not undertake such activities.

3.5.2 SAMPLING HAZARDOUS WASTES



Before sampling begins, inventory all containers to be sampled. Record all available information concerning each container in the notebook. Include the type of container, capacity (estimated or actual) marking, label, origin, condition, etc. Take photographs to provide a permanent record.

Depending on the location and position of containers holding hazardous wastes, it may be necessary to set the containers upright or move them before sampling. Take extreme care to ensure that no rupture or leakage occurs, both to ensure your safety and to prevent spillage.

Once the container is opened:

- Insert the sampling device into the centre of the materials.
- Collect a core of material from a point diagonally opposite the point of entry.
- Retrieve the sample and immediately transfer it into the sample bottle.
- If the sampling device is disposable it may be left in the container sampled. Otherwise decontaminate the device thoroughly before collecting the next sample.

Each container should be sampled individually. Depending on the sampling objective, compositing of samples in the laboratory on a weight/weight or volume/volume basis before analysis may be permissible.

HAZARDOUS SOLIDS IN CONTAINERS

Containerized solid materials such as sludges, granular materials, or powder are usually sampled using one of the following tools:

- scoop or trowel
- waste pile sampler
- Veihmeyer sampler/corer
- sampling trier
- grain sampler
- wide-mouth sample container

LIQUID HAZARDOUS WASTES

Drums containing liquid waste may be under pressure or vacuum. A bulging drum should not be moved or sampled until the pressure can be safely relieved with a remote barrel opener.

- If possible, set containers with the opening or bung upright.
- Mark containers with an identification number for present and future reference. Enamel spray paint is often suitable for this purpose. Again, photographs of the numbered containers can prove valuable in documenting the containers' condition.
- The sampling team leader should determine which drums will be opened.
- Open container using a remote opening device or penetrating apparatus. Such equipment must be used only by an experienced operator and specific procedures for assuring health and safety must be clearly defined and carefully followed. All containers should be opened with the utmost care.

For drums, the bung opening should be loosened slowly with a non-sparking bung wrench. If the bung is badly rusted or frozen it may be necessary to use a non-sparking hydraulic penetrating device. Monitor organic vapour concentrations as vessels are opened using portable instrumentation. Record results in the notebook.

Samples may be taken using the following tools:

- COLIWASA
- open-tube sampler
- stratified sample thief
- VACSAM

4.0 SAMPLING PROTOCOLS

4.1 TABLES – CONTAINERS, PRESERVATION, HOLDING TIME

4.1.2 TOXICOLOGY

4.1.3 ORGANIC CHEMISTRY

4.1.4 BACTERIAL SAMPLING

4.1.5 TEST SELECTION BY INDUSTRY TYPE

4.2 SAMPLING PROTOCOLS

4.2.1 ADSORBABLE ORGANIC HALIDES (AOX)

4.2.2 ALKALINITY/ACIDITY

4.2.3 ANTI-SAPSTAINS

4.2.4 BACTERIA: FECAL COLIFORMS AND STREPTOCOCCI

4.2.5 BIOASSAY OR ACUTE LETHALITY TESTING (LC₅₀/LT₅₀)

4.2.6 CARBON

4.2.7 CHLORINATED PHENOLS

4.2.8 CHLORIDE, FLUORIDE AND SUPHATE

4.2.9 CYANIDE

4.2.10 DIBENZOFURANS (PCDD/PCDF), POLYCHLORINATED DIBENZO-DIOXINS AND DIBENZO-P-DIOXINS

4.2.11 GLYCOL

4.2.12 LEACHATE (TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP))

4.2.13 METALS

4.2.14 NITROGEN COMPOUNDS

4.2.15 NON-FILTERABLE RESIDUE (NFR)

- 4.2.16 OZONE-DEPLETING SUBSTANCES (ODS)
- 4.2.17 OXYGEN DEMAND
- 4.2.18 PESTICIDES
- 4.2.19 PETROLEUM PRODUCTS
- 4.2.20 PHOSPHORUS
- 4.2.21 POLYCHLORINATED BIPHENYLS (PCBS)
- 4.2.22 POLYCYCLIC AROMATIC HYDROCARBONS (PAHS)
- 4.2.23 RADIONUCLIDE – RADIUM-226 (^{226}Ra)
- 4.2.24 RESIN ACIDS
- 4.2.25 SULPHUR COMPOUNDS
- 4.2.26 SURFACTANTS (ANIONIC)
- 4.2.27 TURBIDITY
- 4.2.28 VOLATILE ORGANIC CARBON (VOC)
- 4.2.29 VOLATILE RESIDUES IN SEDIMENT
- 4.3 SAMPLE SHIPPING
 - 4.3.1 SHIPPING LEGAL SAMPLES
 - 4.3.2 DANGEROUS GOODS
 - 4.3.3 SPECIAL CASES

4.1 TABLES – CONTAINERS, PRESERVATION, HOLDING TIME

To get the best quality analytical results the correct handling of sample collection and its prompt delivery to the laboratory is crucial. The tables in this section and the protocols in the following section outline container and preservation usage. Samples should be delivered to the laboratory as soon as possible to ensure the analytical results are representative of the collection site. Two definitions you should be aware of:



HOLDING TIME - is the length of time between when the sample is collected and when the sample is analyzed or fixed (i.e. extracted out of the matrix into solvent).

TURNAROUND TIME - is the length of time it takes from the laboratory receiving the sample, to the time of issuing a result of analysis to the submitter.

In most cases, it will be critical to get the sample to the lab as soon as possible. You should always keep holding time as short as possible and submit samples promptly. Samples will be flagged in the laboratory analytical report if holding times are exceeded and the submitter will be informed.

SAMPLE PRESERVATION

Since it is difficult to know what physical, biological and chemical changes may occur during holding time, samples should be refrigerated at approximately 4°C to reduce biological activity and the rate of chemical decomposition. Chemical preservatives should be added to the sample where required to fix the analyte in question from loss or breakdown.

4.1.1 INORGANIC CHEMISTRY

Parameter	Substance	Sample Container	Preservation	Holding Time (days)+
ACIDITY, ALKALINITY	water	HDPE, 1 L++	4°C	14
AMMONIA	water	HDPE, 1 L++	4°C	5
BIOCHEMICAL OXYGEN DEMAND	water	HDPE, 1 L	4°C	3
BROMIDE, CHLORIDE, FLUORIDE, NITRATE, NITRITE, PHOSPHATE, SULPHATE, TOTAL NITROGEN	s/s/b	tissue cup, 125 mL	4°C	30
CHEMICAL OXYGEN DEMAND	water	HDPE, 250 mL	H ₂ SO ₄ < pH 2*** (at lab)	30
CHLORIDE, FLUORIDE, SULPHATE	water	HDPE, 1 L++	4°C	30
CHLORINE, RESIDUAL	water	on site test	4°C	immediately
COLOUR	water	HDPE, 1 L++	4°C	3
CONDUCTIVITY	water	HDPE, 1 L++	4°C	30
CONDUCTIVITY	s/s/b	tissue cup, 125 mL	4°C	30
CYANIDE	water	HDPE, 250 mL	field NaOH > pH 12***	14 (7 for MMER)
CYANIDE	s/s/b	tissue cup, 125 mL	4°C	30
DISSOLVED OXYGEN	water	HDPE, 1 L	none; fill to exclude air, 4°C	ASAP
HEXAVALENT CHROMIUM	water	HDPE, 250 mL	4°C	24 h
LEACHATE	water	amber glass, 1 L		7
LEACHATE	s/s/b	amber glass, 1 L		7

Parameter	Substance	Sample Container	Preservation	Holding Time (days)+
MERCURY, dissolved	water	acid washed amber glass, 100 mL	field filtered thru 0.45µ cellulose acetate filter, K ₂ Cr ₂ O ₇ < & HNO ₃ pH 2*** (at lab)	30
MERCURY - total	water	amber glass, 100 mL	K ₂ Cr ₂ O ₇ & HNO ₃ < pH 2*** (at lab)	30
MERCURY - total	s/s/b	amber glass, 180 mL or tissue cup, 125 mL	4°C	30
METALS - dissolved	water	new certified clean or acid washed HDPE, 250 mL	field filtered thru 0.45µ cellulose acetate filter, HNO ₃ < pH 2 or filter & preserve at lab (source dependent)	180
METALS, total	water	new certified clean or acid washed HDPE, 250 mL	field HNO ₃ < pH 2 or at lab (source dependent)	180
METALS, total	s/s/b	tissue cup, 125 mL	4°C	180
MOISTURE	s/s/b	tissue cup, 125 mL	4°C	30
NITRATE, NITRITE, PHOSPHATE - total, dissolved, ortho	water	HDPE, 1 L++	4°C	3
NITROGEN - total, dissolved	water	HDPE, 1 L++	4°C	5
NITROGEN, total kjeldahl	water	HDPE, 1 L++	4°C	28
NON-FILTERABLE RESIDUE - total, total dissolved, suspended also known as total suspended solids	water & paper effluent;	HDPE, 1 L++	4°C	24 h pulp 7 days other
PH	water	HDPE, 1 L++	4°C	3

Parameter	Substance	Sample Container	Preservation	Holding Time (days)+
PH	s/s/b	tissue cup, 125 mL	4°C	30
RADIONUCLIDE, RADIUM - 226	water	HDPE, 1 L		30
SULPHIDE	water	HDPE, 500 mL	field ZnAc	7
SULPHIDE	s/s/b	tissue cup, 125 mL	field ZnAc	30
TURBIDITY	water	HDPE, 1 L++	4°C	3
VOLATILE RESIDUE IN SEDIMENT	s/s/b	tissue cup, 125 mL	4°C	7

4.1.2 TOXICOLOGY

Parameter Name	Sample Container	Preservation	Holding Time (days)+
DAPHNIA (chronic 21d, chronic EC25)	20 L bioassay container	4°C	5
DAPHNIA (LC ₅₀ , LT ₅₀)	2 x 1 L HDPE	4°C	5
TROUT (LC ₅₀)	4 x 20 L bioassay containers	4°C	5
TROUT (LT ₅₀)	2 x 20 L bioassay containers	4°C	5

4.1.3 ORGANIC CHEMISTRY

Parameter	Substance	Sample Container	Preservation	Holding Time (days)+
ADSORBABLE ORGANIC HALIDES	water	amber glass, 500 mL**	field HNO ₃ < pH 2***	30
ANTI-SAPSTAINS	water	amber glass, 1 L	4°C (refer to sampling protocol)	30
ANTI-SAPSTAINS	s/s/b	amber glass, 180 mL	4°C (refer to sampling protocol)	30
BEAR BILE	water	amber glass, 180 mL**	4°C	30
BEAR BILE	s/s/b	amber glass, 180 mL**	4°C	30
CARBON - total inorganic, total organic, dissolved inorganic, dissolved organic	water	HDPE, 250 mL	HCl < pH 2, 4°C	28
CARBON, total	solid	tissue cup, 125 mL	HCl < pH 2, 4°C	28
CHLORINATED PHENOLS	water	amber glass, 1 L**	4°C	30
CHLORINATED PHENOLS	s/s/b	amber glass, 180 mL**	4°C	30
DIOXIN & FURAN	s/s/b	amber glass, 180 mL**	4°C	30
DIOXIN & FURAN	water	amber glass, 1 L**	4°C	30
FATTY ACIDS	s/s/b	amber glass, 180 mL**	4°C	30
FATTY ACIDS	water	amber glass, 1 L**	4°C	30
GLYCOLS	water	amber glass, 1 L**	4°C	7
GLYCOLS	s/s/b	amber glass, 180 mL**	4°C	7
HERBICIDES (AEH)	water	amber glass, 1 L**	4°C	7
HERBICIDES (AEH)	s/s/b	amber glass, 180 mL**	4°C	30
HYDROCARBONS	water	amber glass, 1 L**	4°C	7

Parameter	Substance	Sample Container	Preservation	Holding Time (days)+
HYDROCARBONS	s/s/b	amber glass, 180 mL*/**	4°C	14
HYDROCARBON IDENTIFICATION	water	amber glass, 1 L**	4°C	7
HYDROCARBON IDENTIFICATION	s/s/b	amber glass, 1 L**	4°C	7
HYDROCARBON, OIL & GREASE	water	amber glass, 1 L**	HCl < pH 2*** (at lab)	30
HYDROCARBON, OIL & GREASE	s/s/b	amber glass, 180 mL**	4°C	30
OZONE-DEPLETING SUBSTANCES	container	2 cans of product	4°C	7
PCBS	water	amber glass, 1 L**	4°C	30
PCBS	s/s/b	amber glass, 180 mL**	4°C	30
PESTICIDES	water	amber glass, 1 L**	4°C	7
PESTICIDES	s/s/b	amber glass, 180 mL**	4°C	30
POLYCYCLIC AROMATIC HYDROCARBONS	water	amber glass, 1 L**	4°C	7
POLYCYCLIC AROMATIC HYDROCARBONS	s/s/b	amber glass, 180 mL**	4°C	30
RESIN ACIDS	water	amber glass, 1 L**	NaOH > pH 12*** (at lab)	30
RESIN ACIDS	s/s/b	amber glass, 180 mL**	4°C	30
SUBSTITUTED PHENOLS	water	amber glass, 1 L**	4°C	14
SURFACTANTS	water	amber glass, 1 L**	4°C	30
TRIALOMETHANE	water	2 x amber glass, 40mL septum vials*	4°C	14
TRIALOMETHANE	s/s/b	amber glass, 180 mL*/**	4°C	30
VOLATILES	water	2 x amber glass, 40mL septum vials*	4°C	7
VOLATILES	s/s/b	amber glass, 180 mL*/**	4°C	14

4.1.4 BACTERIAL SAMPLING

Parameter	Sample Container	Preservation	Holding Time (days) +
FECAL COLIFORM	aseptic 250 mL container	4°C; for chlorinated samples add sodium thiosulphate	6 hours max
BOTTLE DEFINITIONS			
HDPE	HIGH DENSITY POLYETHYLENE BOTTLE		
AMBER GLASS	HEAT TREATED AMBER GLASS BOTTLE		
S/S/B	SOIL/SEDIMENT/BIOTA *NO HEADSPACE/AIR BUBBLES IN CONTAINER		

**Containers must have Teflon lined cap

***Corrosive – wear protective gloves

+Holding time is from sampling to start of analysis (or fixed)

++Only one HDPE, 1 L bottle is required for all analysis

4.1.5 TEST SELECTION BY INDUSTRY TYPE

Depending on the situation, toxicology testing could be requested for any of these industry types.

AGRICULTURAL RUNOFFS

Herbicides, NO_{2+3} , Pesticides, Phosphorous, pH

CHEMICALS & PLASTIC

Metals

COAL MINES

NFR, PAHs

CONTAMINATED SITES

BTEX, EPH, VH/VPH, Metals, PAHs

DUST SUPPRESSION OILS

PCBs

FISH FARMS

Available Phosphorous, H_2S (field), Redox (field), Sediment Grain Size, Temperature (field), Total Metals, TVR

FISH HATCHERIES

Ammonia, NFR, Total Phosphorous

FOOD PROCESSING

Ammonia

GROUNDWATER

Bromide, Chloride, Fluoride, Metals, NO_{2+3} , Pesticides, pH, Turbidity

HAZARDOUS WASTE

Metals, PCBs, Pesticides

INDUSTRIAL EFFLUENT

Acidity, Alkalinity, Ammonia, Bacteria (Total/Fecal Coli), Bioassays, (Trout/Daphnia LC_{50} & LT_{50}), BOD, Bromide, COD, Chloride, Fluoride, Metals, NFR, NO_{2+3} , TOC, Turbidity

LANDFILL LEACHATES

Mercury, NO_{2+3} , pH

LAUNDROMATS

Ammonia, Phosphorous, pH, PERC

MEAT & POULTRY

Oils & Grease, pH

MINING & METAL FINISHING EFFLUENTS

Ammonia, Cyanide, Mercury, Metals, NFR, PAHs, pH, Sulphides

MUNICIPAL EFFLUENTS

Ammonia, Bacteria (TC, FC, Strep.), BOD, Bioassay (Daphnia and Trout), COD, Conductance, Metals NO₂₊₃, Ortho-P, pH, TOC, Total-P, Turbidity

PETROLEUM PRODUCTS (REFINERY)

VH/VPH for gasolines, mineral spirits, paint thinners

EPH for diesel fuels, lubricating oils & grease, hydraulic oils

BTEX, Oil & Grease, TOC, Metals, Sulphides, Turbidity, NFR, pH, Phenols

NOTE: Test for EPH in conjunction with VH to capture the quantitative values of most petroleum products. Let lab know if need to distinguish between naturally occurring vs. petroleum HC's

PULP AND PAPER

Ammonia, BOD, Dioxin/Furans, LC50/LT50 Fish & Daphnia, Metals, NFR, pH, Resin Acids

SMELTERS

Mercury, Metals, NO₂₊₃

SURFACE WATER

Acidity, Alkalinity, Bacteria (enterococcus, E. coli, total/fecal coli), Chloride, Fluoride, NFR, Ortho-P, pH, TIC, Total-P, Turbidity

TRANSFORMERS, CAPACITOR

PCBs

WASTE OILS

EPH, Oil & Grease, PCBs, SWOG

WOOD CHIPS

Chlorinated Phenols

WOOD PRESERVING FACILITIES

Antisapstains (DDAC, IPBC, Cu-8, TCMTB), Chlorinated Phenols (penta, tetra, tri, di-chlorophenols, guaiacols, catechols), PAHs

4.2 SAMPLING PROTOCOLS

4.2.1 ADSORBABLE ORGANIC HALIDES (AOX)

LIQUID SAMPLING POINT	Paper mill effluent, including bleach water and stages of pulp bleaching process
SAMPLING TECHNIQUE	Grab or composite over 1 h
CONTAINER TYPE AND SIZE	Amber glass, 500 mL; Teflon lid liner, Teflon septum
PRESERVATION	Exclude air headspace in container; add 0.5% sodium sulphite (Na_2SO_3) if needed and 1 mL concentrated nitric acid (HNO_3); store at $< 4^\circ\text{C}$; DO NOT FREEZE
HOLDING TIME	
FINAL EFFLUENT	30 days
C AND E STAGES	2 days

PROTOCOL

1. Collect sample in an amber glass bottle.
2. Fill bottle completely to exclude headspace.
3. Prepare a traveling blank of reagent water, acidified to pH 2 with 1 mL concentrated HNO_3 .
4. If composite samples are required, collect over a 1 h period.
5. Test sample for the presence of residual chlorine as follows:
 - transfer approximately 10 mL of sample to a test tube
 - add a few crystals of potassium iodide
 - add 5 drops of a 1% soluble starch solution
 - a blue colour indicates presence of residual chlorine
6. Any residual chlorine must immediately be removed with sodium sulphite, add 1 mL of a 0.5% $\text{Na}_2\text{SO}_3/\text{L}$ sample; add more if necessary until the blue colour disappears. Use the minimum possible, since excess sodium sulphite will give erroneously low AOX results.
7. Upon collection, but after adding any sodium sulphite, adjust samples to pH 1.5 - 2 with concentrated HNO_3 (1 mL/L is sufficient).
8. Tightly cap sample; transport with freezer packs and store at $< 4^\circ\text{C}$ (do not freeze). Protect from light.

(REFERENCE METHOD EPS L/RM/16, JANUARY 1992)

4.2.2 ALKALINITY/ACIDITY

LIQUID SAMPLING POINT	Water; effluent
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	HDPE, 1 L
PRESERVATION	4°C
HOLDING TIME	14 days

PROTOCOL

1. Rinse bottle three times with water to be sampled.
2. Immerse container in water to a depth approximately 1/3 from the bottom. Avoid skimming the surface.
3. Fill container 95% full.
4. Remove from water and tightly cap sample.
5. Store at 4°C.

NOTE: Choosing the sampling depth requires careful judgment. For deep water, taking the sample 1/3 from the bottom may be impractical; sample at least 1 m from the surface. In shallower water, you may take samples 1 m from the surface and 1 m from the bottom. In pipes or open channels, take samples 1/3 of the way up from the bottom.

4.2.3 ANTI-SAPSTAINS

Wood preservatives: didecyldimethylammonium chloride (DDAC); 3-iodo-2-propynylbutyl carbamate (IPBC); copper-8 (Cu-8); and 2-(Thiocyanomethylthio) benzothiazole (TCMTB).

SAMPLING POINT	Wood preserving/treatment plant effluent; soil; sediment
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	Amber glass, 1 L; Teflon lid liner
SEDIMENTS	Wide-mouth glass jar, 180 g
PRESERVATION	4°C
DDAC AND IPBC LIQUIDS	5 mL Rexonic N25-7* + 10 mL 37% formaldehyde/L sample
DDAC AND IPBC SEDIMENT	2.5 mL Rexonic N25-7 + 5 mL 37% formaldehyde/100 g sample
CU-8	1 mL 3 N NaOH
TCMTB	No preservative
HOLDING TIME	30 days

*NOTE: Rexonic N25-7 is a trade name for alkoxypolyethylenoxyethanol.

PROTOCOL

1. No rinsing should occur, solids should be placed in a 180 mL wide-mouth glass jar.
2. Add preservative as specified in list above. Tightly cap sample.
3. Store at 4°C.

4.2.4 BACTERIA: FECAL COLIFORMS AND STREPTOCOCCI

SAMPLING POINT	Sanitary effluent; surface water; wastewater sediment; biota
SAMPLING TECHNIQUE	Grab only
CONTAINER TYPE AND SIZE	Sterile HDPE or sterile amber glass, 250 mL
PRESERVATION	Chlorinated samples add sodium thiosulphate; 4°C
HOLDING TIME	6 h

NOTE: Bacterial sampling is almost always the responsibility of health authorities. Taking samples without bacterial contamination requires special handling techniques (called aseptic technique). If you must take bacterial samples, get a health inspector or another person with training in aseptic technique to take the sample for you.

PROTOCOL

1. Obtain sterile bottles from lab. Specially treated bottles are required for samples having a chlorine residual up to 15 mg/L (e.g., sewage treatment plant effluent). These bottles should contain 0.1 mL of 10% sodium thiosulphate to dechlorinate the sample.
2. Collect sample as aseptically as possible; the sample bottle must be kept unopened until ready to be filled. Do not rinse bottle with sample. Keep open bottle neck at an angle to prevent bacteria from drifting in from above; hold bottle cap with the open side facing down. Do not pass your hand, clothing, or any object over open bottle neck. Recap as quickly as possible.
3. If sampling drinking water from a tap, run the water for 2-3 minutes before collection to clear the line; then uncaps the bottle quickly, fill and recap immediately.
4. For surface water, uncaps the bottle and quickly plunge it about 30 cm under the water. If a current is present, direct the mouth of the bottle against the current.
5. Be sure to leave ample air space in the bottle to permit mixing during analysis.
6. Keep sample at 4°C; do not freeze. Samples not kept at 4°C will not be analyzed.

4.2.5 BIOASSAY OR ACUTE LETHALITY TESTING (LC₅₀/LT₅₀)

LIQUID SAMPLING POINT	Runoff and wastewater
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	Pail, jerry can, or carboy; HDPE, 1 L, 20 L, 50 L, 100 L drums or 20 L collapsible plastic container
PRESERVATION	4°C; do not freeze; samples should be kept dark and at a temperature of 4°C during transport
HOLDING TIME	5 days

NOTE: Always call the environmental toxicology section to confirm sample requirements prior to sampling for legal analysis. Sample volume requirements depend on numbers of daphnids or fish size exposed to each test solution, loading-density requirements, test concentrations and the use of replicates. For single-concentration tests using rainbow trout, sample volumes of 25 L - 50 L are normally required. For tests to determine an LC₅₀, sample volumes of 120 L or more are normally required. For Daphnia bioassay, approximately 2 L is required. Check with laboratory.

PROTOCOL

1. Rinse containers three times if quantity of sample permits.
2. Collect the required volume of effluent in a new or thoroughly cleaned container.
3. Label with the sample type, source, date and time of collection and name of sampler.
4. Store at 4°C.

(SEE REFERENCE METHOD EPS L/RM/9, 10, 11, 12, 14)

4.2.6 CARBON

DISSOLVED INORGANIC, DISSOLVED ORGANIC (DIC/DOC), TOTAL INORGANIC, TOTAL ORGANIC (TIC/TOC)

LIQUID SAMPLING POINT	Sewage treatment wastewater; surface wastewater; petroleum refinery effluent
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	HDPE, 250 mL
PRESERVATION	HCl < pH 2, 4°C
HOLDING TIME	28 days

PROTOCOL

1. Immerse container in source to a depth of 5 cm - 10 cm.
2. Fill container 95% full.
3. Add preservative; tightly cap sample.
4. Store at 4°C.

TOTAL, TOTAL ORGANIC (TC/TOC)

SOIL, SEDIMENT, BIOTA SAMPLING	Soil; sediment; biota
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	Tissue cup, 125 mL
PRESERVATION	HCl < pH 2, 4°C
HOLDING TIME	28 days

PROTOCOL

1. For fluid samples, immerse container into source to a depth of 5 cm - 10 cm.
2. Fill container to exclude air; tightly cap sample.
3. Store at 4°C.

4.2.7 CHLORINATED PHENOLS

Parameter, wood preservatives; pentachlorophenol; tetrachlorophenol (2,3,4,6- and 2,3,5,6-); trichlorophenol (2,3,4-, 2,3,5-, 2,3,6-, 2,4,5- and 2,4,6-); dichlorophenol (2,4- and 2,6-); tetrachloroguaiacol; trichloroguaiacol (3,4,5-, 3,4,6- and 4,5,6-); dichloroguaiacol (4,5- and 4,6-); monochloroguaiacol (5- and 6-); tetrachlorocatechol; 3,4,5 -trichlorocatechol; dichlorocatechol (3,4-, 3,5- and 4,5-); 4-monochlorocatechol.

LIQUID SAMPLING POINT	Wood preservative plant effluent; wood chips
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	Amber glass, 1 L, quantity may vary with lab requirements; Teflon lid liner
PRESERVATION	4°C, protect from light
HOLDING TIME	30 days

PROTOCOL

1. Fill container 95% full.
2. Add preservative; tightly cap sample.
3. Protect from light. Store at 4°C.

SOIL, SEDIMENT, BIOTA SAMPLING	Wood preservative plant effluent; wood chips
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	Amber glass, 180 mL
PRESERVATION	4°C
HOLDING TIME	30 days

4.2.8 CHLORIDE, FLUORIDE AND SUPLHATE

LIQUID SAMPLING POINT	Surface water; groundwater; effluent; sediment
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	HDPE, 1 L Sediments: tissue cup, 125 mL
PRESERVATION	4°C
HOLDING TIME	30 days

PROTOCOL

1. Rinse bottle and lid three times with sample water before filling.
2. Tightly cap sample. Store at 4°C.

4.2.9 CYANIDE

LIQUID SAMPLING POINT	Mining effluent; metal-finishing effluent
SAMPLING TECHNIQUE	Grab; or 7 day, 24 h composite
CONTAINER TYPE AND SIZE	HDPE, 250 mL
PRESERVATION	Ascorbic acid, if needed; 1.5 mL 40% NaOH/100 mL or add 2 NaOH pellets/250 mL; 4°C
HOLDING TIME	14 days, MMER 7 days

NOTE: Oxidizing agents can destroy cyanide. Therefore check for oxidants by putting a drop of sample on 5% potassium iodide-starch paper; a blue colour indicates that ascorbic acid must be added. Add ascorbic acid a few crystals at a time until sample produces no colour change on indicator paper then add 0.5 g excess. NaOH raises the pH of the sample to above 12 in order to stabilize the cyanide compound by preventing dissociation. NaOH at the concentration used and in pellet form is extremely corrosive and can cause severe burns to skin and eyes; routine safety procedures should be followed. This preservative is normally supplied in small vials by the laboratory doing the analysis. If taking composite samples, put preservative in the container before collecting samples.

PROTOCOL

1. Put on protective disposable gloves.
2. Rinse container with sample water three times.
3. Check for presence of oxidizing agents and add ascorbic acid if necessary.
4. Add NaOH preservative; tightly cap sample.
5. Store at 4°C.

SOIL, SEDIMENT, BIOTA SAMPLING	Mining and metal-finishing effluent area
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE	Tissue cup, 125 mL
PRESERVATION	4°C
HOLDING TIME	30 days

4.2.10 DIBENZOFURANS (PCDD/PCDF), POLYCHLORINATED DIBENZODIOXINS AND DIBENZO-p-DIOXINS

SAMPLING POINT	Pulp mill effluent; sediment; biota
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	Amber glass, 1 L Sediments: wide-mouth glass jar, 180 g
PRESERVATION	4°C; (Na ₂ SO ₃ for bleach plant effluent)
HOLDING TIME	30 days

See EPS L/RM/19 FEBRUARY 1992 for sampling effluents from pulp and paper mills. For other cases, collect sample directly into containers. For aqueous samples, do not rinse the containers before filling them, take 1 L samples in amber glass bottles.

PROTOCOL

1. Solids should be placed in a 180 mL wide-mouth glass jar.
2. Store at 4°C.

SAMPLING POINT	Defoamers; contaminated surfaces
SAMPLING TECHNIQUE	
DEFOAMERS	Grab
SURFACES	Wipe with hexane
CONTAINER TYPE AND SIZE	
DEFOAMERS	Amber glass; foil or Teflon lid liner
SURFACES	Amber glass for wipe sample
PRESERVATION	4°C
HOLDING TIME	30 days

PROTOCOL - DEFOAMERS

1. Collect 180 mL defoamer in a glass container.
2. Seal with cap lined with Teflon or aluminum foil.
3. Store at 4°C.

PROTOCOL - FUGITIVE EMISSIONS

1. Soak a sorbent pad with a known volume of pesticide-grade hexane. Wipe (stroking both ways) a 25 cm x 25 cm area. Insert wipe in amber glass bottle; use Teflon or foil-lined cap.
2. Protect from light. Store at 4°C.

4.2.11 GLYCOL

SAMPLING POINT	Airport effluent, storm water and drainage streams; glycol guidelines
SAMPLING TECHNIQUE	Grab; 2 samples, one taken at least 30 minutes and not more than 24 hours after the first sample
CONTAINER TYPE AND SIZE	Amber glass, 180 mL or 1 L
PRESERVATION	4°C
HOLDING TIME	7 days

PROTOCOL

1. At airport, take the first sample, then wait at least 30 minutes and not more than 24 hours before taking the second sample.
2. Store at 4°C.

4.2.12 LEACHATE (Toxicity Characteristic Leaching Procedure (TCLP))

SAMPLING POINT	Various; waste or hazardous waste material
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	Amber glass, 1 L
PRESERVATION	4°C
HOLDING TIME	7 days

PROTOCOL

1. For volatile components do not leave any headspace.
2. For liquid waste (i.e. those containing < 0.5% dry solid), the waste, after filtration thru a 0.6 µ - 0.8 µ glass fibre filter, is defined as the TCLP extract.
3. For waste containing > or = 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the partical size of the solid phase is reduced, if necessary.
4. The solid phase is extracted with an amount of extraction fluid = 20 x the weight of the solid phase.
5. Store at 4°C.

4.2.13 METALS

Including: aluminum (Al), antimony (Sb), arsenic (As), boron (B), barium (Ba), beryllium (Be), calcium (Ca), cadmium (Cd), cobalt (Co), chromium (Cr), copper (Cu), iron (Fe), lead (Pb), mercury (Hg) magnesium (Mg), manganese (Mn), molybdenum (Mo), nickel (Ni), phosphorus (P), potassium (K), selenium (Se), silicon (Si), silver (Ag), sodium (Na), strontium (Sr), sulfur (S), thorium (Th), tin (Sn), titanium (Ti), uranium (U), vanadium (V), zinc (Zn).

NOTE: Lab protocols will depend on the analyte of interest. Check with the laboratory to determine the volume of sample needed and metals available to be analyzed.

TOTAL, EXTRACTABLE, DISSOLVED OR SUSPENDED

SAMPLING POINT	Municipal wastewater treatment effluent; industrial effluent (forest products, pulp and paper, chemicals, plastics, petroleum processing, metal finishing); mining and refining
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	HDPE, 250 mL
PRESERVATION	HNO ₃ (see protocol)
HOLDING TIME	180 days

WARNING! Nitric acid is extremely corrosive; use gloves. It fumes freely. NEVER ADD NITRIC ACID TO SAMPLES THAT MIGHT CONTAIN CYANIDE, AS NITRIC ACID WILL CAUSE THE RELEASE OF DEADLY CYANIDE GAS.

Samples containing nitric acid must be shipped via a carrier certified to handle dangerous goods; they cannot be shipped by air (IATA), unless the nitric acid as a preservative is less than 20% limit.

PROTOCOL - TOTAL OR EXTRACTABLE

1. Put on protective gloves.
2. Rinse container with sample water three times.
3. Add preservative; 1 mL, 35% nitric acid per 100 mL sample or 2 mL 1:1 HNO₃/250 mL sample. Tightly cap sample; shake to mix.
4. Sample can be stored at room temperature.

PROTOCOL - DISSOLVED

1. Put on protective gloves.
2. Using a VWR disposable 60 ml latex free syringe inject sample through a 29 mm, 0.45 µ pore size, single use, hydrophilic Durapore membrane filter (Milipore).
3. Repeat to accumulate the 250 mL volume of sample required.
4. Rinse the container once with a small amount of filtrate. Fill container with remainder of filtrate; add preservative (1 mL 35% nitric acid per 100 mL sample). Tightly cap sample; shake to mix.
5. Sample can be stored at room temperature.
6. Be sure to clean up any disposable filters or syringes.

NOTE: Suspended metals are those retained on the filter.

TOTAL

SOIL, SEDIMENT, BIOTA SAMPLING	Municipal wastewater treatment sites; industrial sites (forest products, pulp and paper, chemicals, plastics, petroleum processing, metal finishing); mining and refining
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	Tissue cup, 125 mL
PRESERVATION	4°C
HOLDING TIME	180 days

HEXAVALENT CHROMIUM (CR⁶)

SOIL, SEDIMENT, BIOTA SAMPLING	Municipal wastewater treatment sites; industrial sites (forest products, pulp and paper, chemicals, plastics, petroleum processing, metal finishing); mining and refining
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	HDPE, 250 mL
PRESERVATION	4°C
HOLDING TIME	24 h

4.2.14 NITROGEN COMPOUNDS

Including: nitrate, nitrite, ammonia, total Kjeldahl nitrogen, total dissolved nitrogen, total nitrogen.

NITRATE AND NITRITE

LIQUID SAMPLING POINT	Sewage/effluent; landfill leachate; smelters; agricultural runoff; groundwater
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	HDPE, 1 L
PRESERVATION	4°C
HOLDING TIME	3 days

PROTOCOL

1. Rinse container with sample water three times.
2. Submerge container into water to a depth of approximately 5 cm - 10 cm.
3. Store at 4°C.

AMMONIA

LIQUID SAMPLING POINT	Municipal wastewater treatment plant effluent; industrial effluent (food processing, metal finishing, mining and refining); laundries and laundromats; private domestic sewage discharge; industrial sanitary effluent; pulp and paper effluent
SAMPLING TECHNIQUE	Grab or 24 h composite
CONTAINER TYPE AND SIZE	HDPE, 1 L
PRESERVATION	4°C
HOLDING TIME	5 days

PROTOCOL

1. Rinse container with sample water three times.
2. Fill container to the rim; tightly cap sample.
3. Store at 4°C.

TOTAL KJELDAHL NITROGEN

LIQUID SAMPLING POINT	Municipal wastewater treatment plant effluent; food processing; metal finishing; mining and refining; laundries and laundromats; sewage discharge; pulp and paper industries
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	HDPE, 1 L
PRESERVATION	4°C
HOLDING TIME	28 days

PROTOCOL

1. Rinse container with sample water three times.
2. Fill container to the rim; tightly cap sample.
3. Store at 4°C.

4.2.15 NON-FILTERABLE RESIDUE (NFR)

Including: filterable residues/non-filterable residues, also known as total suspended solids (TSS).

LIQUID SAMPLING POINT	Surface water effluent; pulp and paper mill effluent; metal mining effluent
SAMPLING TECHNIQUE	
GENERAL	Grab or composite
PULP AND PAPER	Composite
METAL MINING	Grab
CONTAINER TYPE AND SIZE	
GENERAL	HDPE, 200 mL for turbid sample; 1 L for clear sample
PULP AND PAPER / MINING	HDPE, 1 L
PRESERVATION	4°C
HOLDING TIME	24 h for pulp and paper effluent; 7 days for other samples

PROTOCOL

1. Rinse container with sample water three times.
2. If compositing, keep sample at 4°C.
3. Keep sample at or below 4°C.

(SEE PROTOCOL FOR BIOCHEMICAL OXYGEN DEMAND)

4.2.16 OZONE-DEPLETING SUBSTANCES (ODS)

Including: CFCs, carbon tetrachloride, methyl chloroform, halons.

SAMPLING POINT	Commercial products
SAMPLING TECHNIQUE	n/a
CONTAINER TYPE AND SIZE	2 cans of product
PRESERVATION	none
HOLDING TIME	7 days

PROTOCOL

Most environmental protection labs do not have the capability to analyze for CFCs. Commercial samples may be contracted out for analysis.

4.2.17 OXYGEN DEMAND

Including: biochemical oxygen demand (BOD) and chemical oxygen demand (COD).

DISSOLVED OXYGEN (DO)

Tests for dissolved oxygen are normally done in the field using dissolved oxygen (DO) meters or multi-mode meters equipped with a DO sensor as part of their multiple readout capability. These meters require calibration before use. Colourmetric DO kits are also available. They are user friendly and do not require calibration.

The time frame for lab analysis of DO demand varies depending on the type of sample collected. Samples from effluent treatment facilities have a very short analytical time frame due to the possibility of high biochemical oxygen demand (BOD). Consult with the lab before sampling for DO. Lab analytical methods may require the addition of preservatives to the sample depending on the analytical time requirements.

LIQUID SAMPLING POINT	Water; effluent
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	HDPE, 1 L
PRESERVATION	None; fill to exclude air; 4°C
HOLDING TIME	ASAP

PROTOCOL

1. Submerge sample container into source. Do not skim surface.
2. Fill sample container to the top.
3. Store at 4°C.

BIOLOGICAL OXYGEN DEMAND (BOD)

LIQUID SAMPLING POINT	Water; effluent
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	HDPE, 1 L
PRESERVATION	No preservative; fill to exclude air; 4°C
HOLDING TIME	3 days

NOTE: BOD tests are highly time sensitive. Coordinate your sampling times with the lab beforehand to ensure that samples will be analyzed as quickly as possible.

PROTOCOL

1. Before preparing BOD sub-samples, ensure that the containers and lid are rinsed at least three times with the composite sample water, then fill bottles to the rim to exclude air before capping with glass lids.
2. If analysis is begun within 2 h of collection, cold storage is unnecessary. If analysis is not started within 2 h, keep sample at or below 4°C.
3. For composite samples, keep samples at or below 4°C during compositing period and limit compositing period to 24 h. Holding time in this case begins at the end of compositing. Mix vigorously and pour off samples into appropriate bottles, excluding air.

CHEMICAL OXYGEN DEMAND (COD)

LIQUID SAMPLING POINT	Water; effluent
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	HDPE, 250 mL
PRESERVATION	H ₂ SO ₄ /L < pH 2 (at lab); 4°C
HOLDING TIME	30 days

PROTOCOL

1. Rinse container with sample water three times.
2. Submerge container into source to a depth of 5 cm - 10 cm (do not skim the surface).
3. Fill container 95% full.
4. Add preservative; tightly cap sample.
5. Store at 4°C.

4.2.18 PESTICIDES

Including: herbicides, organophosphates and carbamates.

LIQUID SAMPLING POINT	Surface water; groundwater; agricultural runoff
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	Amber glass, 1 L - 4 L; Teflon lined lids
PRESERVATION	4°C; protect from sunlight
HOLDING TIME	7 days

PROTOCOL

1. Normally 1 L - 4 L of water is collected in amber glass containers.
2. Since there is no universal preservative possible, consult the lab for advice before sampling.
3. Samples must be stored at < 4°C and protected from sunlight.
4. In the case of sediment, collect 180 mL samples; in the case of biota, collect one or more whole organisms. Shellfish are good indicators.

SOIL, SEDIMENT, BIOTA SAMPLING	Agricultural sites; soil; vegetation; sediment; sludge
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	Amber glass, 180 mL
PRESERVATION	4°C; protect from sunlight
HOLDING TIME	30 days

4.2.19 PETROLEUM PRODUCTS

HYDROCARBON AND PETROLEUM PRODUCT SPILLS

LIQUID SAMPLING POINT	Areas with definite or suspected spills
SAMPLING TECHNIQUE	Grab - technique varies depending on nature of incident
CONTAINER TYPE AND SIZE	Amber glass, 1 L; Teflon or aluminum-foil lid liner
PRESERVATION	4°C
HOLDING TIME	7 days

SOIL, SEDIMENT, BIOTA SAMPLING	Areas with definite or suspected spills
SAMPLING TECHNIQUE	Grab - technique varies depending on nature of incident
CONTAINER TYPE AND SIZE	Amber glass, 180 mL; Teflon or aluminum-foil lid liner
PRESERVATION	4°C
HOLDING TIME	14 days

PROTOCOL

Because each investigation is different, it is not possible to establish a step-by-step guide for sampling spills of these substances. In general, it is best to sample the spilled oil as soon as possible, before it can disperse and weather.

Normally, for liquid samples, an oil layer of at least 3 mm thickness in a bottle is required for running the full range of tests. If the spill involves a thin surface film, it may be necessary to concentrate it as follows:

1. Tilt the edge of the sample bottle just below the water surface to skim off the maximum amount of oil for analysis.

2. If the film is too thin, cap the bottle, invert it and allow the oil to rise to the surface. Then loosen the cap enough to allow much of the water to drain from the jar. Repeat this procedure until sufficient sample is obtained for analysis.

For large spills or for a spill that may involve more than one oil, obtain several samples from different locations to insure the spilled oil is properly represented. If a spill involves tar balls of softball size or larger, slice open the tar ball and scoop a sample from the centre, where weathering effects are minimized.

Take control samples from any suspected source of a spill, to match for “fingerprinting.” You may also want to sample the product concentration in soil, using the Canadian Council of Ministers of the Environment (CCME), National Guidelines for Decommissioning Industrial Sites, 1991.

OIL AND GREASE

SAMPLING POINT	Refinery effluent; soil from contaminated sites; meat and poultry operations; transformer fluid
SAMPLING TECHNIQUE	Grab; petroleum refinery 24 h composite
CONTAINER TYPE AND SIZE	Amber glass, 1 L; Teflon or aluminum-foil lid liner
PRESERVATION	HCL < pH 2***(done at lab); 4°C
HOLDING TIME	30 days after preserved

NOTE: Oil and grease will adhere to the sides of the bottle. For this reason, DO NOT RINSE BOTTLE with sample water; simply fill the container, add preservative and cap the bottle. For petroleum refineries, a 24 h composite is required.

PROTOCOL

1. Collect approximately 1 L sample in glass bottles with Teflon or foil-lined caps.
2. Add 2 mL of conc.H₂SO₄, or HCL/per litre and store sample at 4°C.
3. For autosamplers or composite samples, add preservative to sample container before sampling begins. Keep sample at 4°C during sampling period (for petroleum refineries).

WATERBORNE OILS

The following guidelines are provided for the collection of environmental and ship source oil samples. The guidelines are appropriate for the collection of oils on water (bilge and environmental), beaches, wildlife, lubricating fluids and fuels that are destined for chemical analysis.

LIQUID SAMPLING POINT	Waterborne oils - water with thin oil sheen samples and soil samples with a suspected volatile fuel
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	Amber glass, 120 mL; Teflon lid liner
PRESERVATION	2 mg of sodium thiosulfate added to the bottles prior to sampling (optional); 4°C
HOLDING TIME	14 days

PROTOCOL

1. Select a site where the thickness of oil is greatest.
2. Gently skim the oil off the surface of the water until the jar is 3/4 full.
3. Tightly seal the container with the lid and invert the jar for 2 - 3 minutes.
4. When all the oil has risen to the top of the water level, gently unscrew the sample jar lid and allow the water to seep out of the inverted container.
5. Repeat these steps, if necessary, to obtain approximately 60 mL of oil if possible.
6. Invert the jar a final time and allow it to stand 3 - 5 minutes before draining off excess water.
7. Tighten the lid and wipe off excess oil and water from the outside surface of the container.
8. Store at 4°C.

OR

Using 120 mL amber glass bottles with Teflon lid liners, which have had 2 mg of sodium thiosulfate added to the bottles prior to sampling:

1. Do not rinse sample bottle as it has been pretreated with preservative.

2. Completely fill the glass bottle with the sample and cap immediately. For gasoline analysis, sample as per other hydrocarbons (i.e., collect as much of the water's surface as possible). All samples should be provided in duplicate.
3. Samples should be free of air bubbles (no head space). Store at 4°C.

OR

1. If collection of an oil screen cannot be accomplished by any other method, a clean piece of absorbent may be used to collect a sample of the sheen from the surface. Care must be taken not to cross contaminate the absorbent in any way.
2. After a sample has been collected the absorbent is to be placed in a sample bottle, labeled and secured.
3. A clean piece of absorbent from the same batch that was used to collect the sample from the water surface should be collected and placed in another sample bottle, labeled and secured for analysis by the lab. Store at 4°C.

VOLATILE FUEL SAMPLES

Including: gasoline and kerosene, white gas, camp fuel, etc.

LIQUID SAMPLING POINT	Volatile fuel samples (gasoline and kerosene, white gas, camp fuel, etc.)
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	Amber glass, 120 mL; Teflon lid liner
PRESERVATION	—
HOLDING TIME	3 days

OILED SOIL

Including: soil samples suspected to contain a volatile fuel.

SOIL, SEDIMENT, BIOTA SAMPLING	Oiled soil - soil samples suspected to contain a volatile fuel
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	Amber glass; Teflon lid liner
PRESERVATION	—
HOLDING TIME	3 days

PROTOCOL

1. Obtain a clean sample jar and remove the lid.
2. Using the lid, scoop the oiled soil into the jar; tightly cap sample.
3. If there is oil on large debris such as wood, seaweed and plastics scrape the oil off into a sample jar using a clean, disposable, stainless steel razor blade.
4. Where large tar balls are involved, it is desirable to slice open the tar ball using a clean, disposable, stainless steel razor blade and scoop a sample from the center as this area is usually less weathered. Record method of collection in a notebook.
5. Wipe excess material from the outside of the jar.
6. After collecting the sample the razor blade is to be disposed of in a method appropriate for sharps disposal.

OIL CONTAMINATED WILDLIFE

SOIL, SEDIMENT, BIOTA SAMPLING	Oil contaminated wildlife
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	Amber glass, 120 mL; Teflon lid liner
PRESERVATION	4°C; protect from sunlight; freezing of samples destined for oil match analysis should be avoided
HOLDING TIME	3 days

PROTOCOL

1. Using a clean pair of disposable gloves, pluck contaminated feathers or fur and place in a clean sample container for oil analysis. In some cases it may be necessary to collect the whole animal for identification but this is not necessary for oil analysis. Record method of collection in a notebook.
2. Store at 4°C.

OR

1. Using a clean, disposable stainless steel razor blade, cut and remove contaminated feathers or fur and place in a clean sample container for oil analysis. After collecting the sample the razor blade is to be disposed of in a method appropriate for sharps disposal. Record method of collection in a notebook.
2. If there are areas of feathers or fur that are uncontaminated by oil, using a new stainless steel razor blade, cut off an uncontaminated sample and place in a second clean sample bottle.
3. Store at 4°C.

STORAGE AND TRANSPORT

Samples must be securely stored, protected from light, in an evidence bag, following TDG Regulations and Legal Sampling guidelines, for all oil samples.

4.2.20 PHOSPHORUS

Including: P_2O_5 or P and phosphates.

INORGANIC PHOSPHORUS (P_2O_5 OR P)

SAMPLING POINT	Laundry detergent (commercially packaged and bulk); unleaded gasoline
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	
DETERGENTS	Unopened commercial product or 125 mL glass jars for bulk samples, liquid or solid; 1 L for bulk products
PRESERVATION	4°C
HOLDING TIME	3 days

NOTE: For sampling gasoline products, check with the lab and with the regulations to determine container, preservation, holding time and sampling protocol.

PROTOCOL

1. Purchase product.
2. Send product to laboratory unopened.
3. For bulk samples: choose small quantities from different locations of the bulk product.
4. Store at 4°C.

PHOSPHATES (ORTHO-, TOTAL DISSOLVED AND TOTAL)

LIQUID SAMPLING POINT	Effluent; surface water
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	HDPE, 1 L
PRESERVATION	4°C
HOLDING TIME	3 days

NOTE: Phosphates adhere to glass; hence the samples should be taken into individual glass bottles that the lab then uses for its analysis. Check with the lab beforehand to obtain the containers it requires.

PROTOCOL

1. Rinse container with sample water three times.
2. Collect effluent in a HDPE, 1 L bottle.
3. Do not freeze samples; store at 4°C.

SOIL, SEDIMENT, BIOTA SAMPLING	Effluent
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	Tissue cup, 125 mL
PRESERVATION	4°C
HOLDING TIME	30 days

4.2.21 POLYCHLORINATED BIPHENYLS (PCBs)

SAMPLING POINT	Mill effluent; soil and sediment; transformer and waste oil; biota; oil sprays for suppressing dust on roads
SAMPLING TECHNIQUE	
LIQUIDS	Grab or composite
SOLIDS	Core
CONTAINER TYPE AND SIZE	
LIQUIDS	Amber glass, 1 L
SOLIDS, SEDIMENTS, WIPES	Amber glass, 180 mL
PRESERVATION	4°C
HOLDING TIME	30 days

NOTE: When sampling road surfaces to verify compliance, consult with the lab to determine the information needed.

PROTOCOL

1. Collect effluent in 1 L amber glass bottles as per the protocol for chlorinated phenols.
2. Oil samples should be collected in 180 mL, amber glass jars.
3. Sediment samples should be collected in 180 mL, amber glass bottles.
4. Biota samples should be wrapped in heat-treated aluminum foil, placed in an amber glass bottle and frozen as soon as possible.
5. Wipe samples should be taken from non-porous smooth surfaces; 100 cm² is sufficient. Use a small piece of cheesecloth, folded to give at least four layers, dampened with hexane. Swab surface in both directions. The wipe is then placed in a 180 mL, amber glass bottle.
6. Store at 4°C.
7. Oil samples must be shipped in conformance with the Transportation of Dangerous Goods Act and Regulations.

4.2.22 POLYCYCLIC AROMATIC HYDROCARBONS (PAHs)

LIQUID SAMPLING POINT	Wood preservative plant effluent; aluminum smelters; coal mine effluent
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	Amber glass, 1 L; Teflon or heat-treated aluminum foil lid liner
PRESERVATION	4°C
HOLDING TIME	7 days

PROTOCOL

1. Rinse container with sample water three times.
2. Insulate container mouth and lid with a piece of heat-treated aluminum foil, or use Teflon-lined lid. Tightly cap sample
3. Store at 4°C.

SOIL, SEDIMENT, BIOTA SAMPLING	Wood preservative plant effluent; aluminum smelters; coal mine effluent
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	Amber glass, 180 mL; Teflon or heat- treated aluminum foil lid liner.
PRESERVATION	4°C
HOLDING TIME	30 days

SOIL, SEDIMENT, BIOTA SAMPLING	Wood preservative plant effluent; aluminum smelters; coal mine effluent; sediment; soil
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	Amber glass, 180 mL
PRESERVATION	4°C
HOLDING TIME	30 days

4.2.23 RADIONUCLIDE – RADIUM-226 (^{226}Ra)

SAMPLING POINT	Metal mining effluent
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	HDPE, 1 L
PRESERVATION	HNO_3
HOLDING TIME	30 days

PROTOCOL

1. Put on protective gloves.
2. Rinse container and lid three times with sample.
3. Add preservative. 1 ml 35% nitric acid per 100 ml sample, to a pH <2 sample or 2 mL 1:1 HNO_3 /250 mL sample. Preservatives can be added up to 5 days after sampling.
4. Tightly cap sample and shake.
5. Samples may be stored at room temperature.

4.2.24 RESIN ACIDS

Including: abietic, chlorodehydroabietic, dehydroabietic, isopimaric, levopimaric, neoabietic, sandaracopimaric and dichlorodehydroabietic acids.

LIQUID SAMPLING POINT	Municipal wastewater treatment effluent; wood products/industries (pulp and paper, forest products)
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	Amber glass, 1 L; Teflon or heat-treated aluminum foil lid liner
PRESERVATION	NaOH > pH 12*** (done at lab)
HOLDING TIME	30 days

PROTOCOL

1. Insulate container mouth and lid with a piece of heat-treated aluminum foil, or use Teflon-lined lid. Tightly cap sample.
2. Protect from sunlight.

SOIL, SEDIMENT, BIOTA SAMPLING	Municipal wastewater treatment effluent; wood products/industries (pulp and paper, forest products)
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	Amber glass, 180 g; Teflon or heat-treated aluminum foil lid liner
PRESERVATION	4°C
HOLDING TIME	30 days

4.2.25 SULPHUR COMPOUNDS

SULPHATE

LIQUID SAMPLING POINT	Effluent; wastewater
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	HDPE, 1 L
PRESERVATION	4°C
HOLDING TIME	30 days

PROTOCOL

1. Rinse container with sample water three times.
2. Collect sample in HDPE bottle.
3. Store at 4°C.

SULPHATE

SOIL, SEDIMENT, BIOTA SAMPLING	Effluent; wastewater
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	Tissue cup, 125 mL
PRESERVATION	4°C
HOLDING TIME	30 days

SULPHIDES

LIQUID SAMPLING POINT	Effluent and wastewater from mills, mines, refineries, petroleum industry
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	HDPE, 500 mL
PRESERVATION	field ZnAc
HOLDING TIME	7 days

NOTE: Sulphides oxidize readily. Check with the lab for the preservatives to use; zinc acetate and sodium bicarbonate are frequently used. Add preservative to the collecting bottle before starting composite sampling.

PROTOCOL - GRAB

1. Rinse sample bottle three times with sample water.
2. Collect sample.
3. Add preservative to sample bottle.
4. Tightly cap sample; store at 4°C.

PROTOCOL - COMPOSITE

1. Rinse sample bottle three times with sample water.
2. Add preservative.
3. Collect sample.
4. Store at 4°C.

SULPHIDES

SOIL, SEDIMENT, BIOTA SAMPLING	Effluent and wastewater from mills, mines, petroleum industry, fish farms
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	Tissue cup, 125 mL
PRESERVATION	field ZnAc
HOLDING TIME	30 days

4.2.26 SURFACTANTS (ANIONIC)

LIQUID SAMPLING POINT	Refinery effluent; sewage wastewater; detergent plants; mines
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	Amber glass, 1 L; Teflon or heat-treated aluminum foil lid liner
PRESERVATION	4°C
HOLDING TIME	30 days

PROTOCOL

1. Rinse container with sample water three times.
2. Collect sample in an amber glass container with Teflon or foil-lined screw cap.
3. Store at 4°C.

4.2.27 TURBIDITY

LIQUID SAMPLING POINT	Surface water; effluent
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	HDPE, 1 L
PRESERVATION	4°C
HOLDING TIME	3 days

PROTOCOL

1. Collect water from below the surface; do not skim, fill container to approximately 95% full.
2. Tightly cap sample; store at 4°C.

4.2.28 VOLATILE ORGANIC CARBON (VOC)

Also known as head-space or purgeable organic analysis; including benzene, toluene, ethylbenzene and xylene (BTEX).

LIQUID SAMPLING POINT	Effluent; surface water; groundwater; petroleum industry
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	Amber glass, 40 mL x 2; Teflon septum cap
PRESERVATION	4°C
HOLDING TIME	7 days

PROTOCOL

1. Fill vial to rim. Add one drop concentrated hydrochloric acid.
2. Tightly cap sample, making sure that there is no head space in the sample; top up as necessary.
3. Store at 4°C.

SOIL, SEDIMENT, BIOTA SAMPLING	Storage tanks; contaminated sites; petroleum industry
SAMPLING TECHNIQUE	Grab
CONTAINER TYPE AND SIZE	Amber glass, 180 mL; Teflon septum cap
PRESERVATION	4°C
HOLDING TIME	14 days

4.2.29 VOLATILE RESIDUES IN SEDIMENT

SOIL, SEDIMENT, BIOTA SAMPLING	Domestic wastewater; agricultural fumigants; refinery and mill outfall; metals and metal processing; soil; sediment
SAMPLING TECHNIQUE	Grab or composite
CONTAINER TYPE AND SIZE	Tissue cup, 125 mL
PRESERVATION	4°C
HOLDING TIME	7 days

PROTOCOL

1. Collect sediment in tissue cup.
2. Store at 4°C.

4.3 SAMPLE SHIPPING

Inspectors go to considerable effort and expense to obtain representative samples, but all this work can be negated if the samples fail to arrive at the lab in good condition within the required time frame.

Samples should be shipped so they reach the laboratory as soon as possible, within the analytical holding time period prescribed in PART 4.1: TABLES - CONTAINERS, PRESERVATIVES AND HOLDING TIMES. Delays in transporting samples must be avoided and care must be taken to ensure that samples are kept at the proper temperature.

SAMPLE LABELING

Ensure that each sample is identified and marked with your initials. Use polyester labels with permanent adhesive. Samples can also be placed in a sealable plastic bag to minimize any chance of the sample coming into contact with ice water during shipment.

SEALS

If possible, use manufactured, numbered seals. The numbers should be recorded by the sampler. As an alternative if you do not have these seals, strapping tape can be used.

- Seal containers with strapping tape, wrapped tightly, glue side to glue side.
- Initial the tape.
- Label container with site name, unique sample number, date, time and initials.
- Scribe this information directly onto the containers.

SHIPPING PREPARATION

Sample containers must be securely closed and packed in order to prevent leaking or breakage. Ensure that each shipping container is labeled with the destination, return address and any required safety markings and labels, as well as any special instructions, such as “FRAGILE,” “KEEP COOL,” “DO NOT FREEZE,” “KEEP FROZEN,” “THIS END UP.” The shipping containers must also be marked with the appropriate labels and other information as required under Transportation of Dangerous Goods (TDG) Act requirements and the International Air Transport Association/International Civil Aviation Organization (IATA/ICAO).

Samples from any one location should be kept together. If samples must be separated and placed in more than one shipping container, a copy of the sample submission form pertaining to those samples should be enclosed with the containers.

- Samples should be bagged and put into a sturdy container or cooler that has been lined with a large plastic bag.
- Add freezer packs if the sample needs to be kept cool and pack with non-flammable absorbent material.
- Seal the liner bag.
- Put the sample submission sheet into a moisture-proof bag and enclose it with the sample.
- Seal the container and attach a second copy of the sample submission sheet to the outside.
- Label the container and complete the bill of lading.

SOME SPECIFIC CONSIDERATIONS

- Packages must be secure and rugged and must meet the TDG Act and/or IATA Dangerous Goods Regulations. In general, shipping containers should protect samples from a 1.8 m drop and be able to be stacked 3 m high.
- Do not overload shipping containers. Remember that 10 kg is the cut-off weight under TDG requirements. Ten 1 L bottles of sample, plus packing materials, plus the container weight, would exceed this.
- To help ensure that liquid samples stay upright, you may block them in with empty sample bottles, freezer packs, or pieces of cardboard.
- Freezer packs are ideal for keeping samples cool. If you must use ice, ensure that sample bottles are bagged (zip-lock bags are ideal) to keep the labels out of contact with the ice or ice water.

One useful tip is to pack a small tightly capped vial of water with your sample and alert the lab that it is enclosed in the package. When lab staff receive the sample, they can check the temperature of the water in the vial, without disturbing the sample, to determine if the sample has been kept cool.

SHIPPING

Samples can be sent by registered mail, courier, ground transportation (truck, bus), or air. Keep in mind the holding time for the parameters of interest. Ship by the quickest, most direct route possible and make sure that the route can be traced. Sample shipping can be expedited for an extra charge. Samples should arrive at the lab when there is someone present to receive and deal with them. Contact the lab beforehand, to ensure that the lab knows to expect the sample.

Once the shipment has been sent, contact the laboratory and if possible, speak to the section in charge of sample reception, giving details of types and number of samples, sampling date, estimated arrival date, means of sample shipment and (if applicable) the courier company name and waybill number.

4.3.1 SHIPPING LEGAL SAMPLES

Legal samples require that the physical control or continuity of a sample be documented from the time it is taken until the time it is destroyed. This is documented on a chain-of-custody form. To establish continuity, a witness must be able to satisfy the court that there was no tampering with or contamination of the sample at any point. Keep the number of people involved in handling the samples to a minimum. Continuity of the samples must be maintained until the inspection is closed.

- The labeled sample container is to be sealed using a numbered seal, then placed into a ziplock bag.
- The ziplock bag is to be sealed using an unnumbered seal or security tape — only one sample is to be placed into each bag to avoid confusion should the labels come off the bottle.

OR

- Label the sample container using a diamond-tipped scribe, waterproof marker, or other means of permanent identification with sufficient information to enable you to identify the sample later in court.
- Strapping tape can be used to seal sample bottles; wrapped tightly, glue side to glue side ensures that no one can tamper with the container.
- Write your initials across the edge of the tape and the body of the package.

CHAIN-OF-CUSTODY FORM

All samples are to be logged onto a chain-of-custody form including seal numbers for the samples and containers. If samples are being delivered by courier, the chain-of-custody form is to be placed in a plastic bag.

LOCKED SHIPPING CONTAINERS

All samples are to be placed into a security box (tool box) packed with freezer packs and packing material. A vial of water is to be included in the security box for temperature measurement upon arrival at the laboratory. The security box is to be sealed using a wired, numbered seal.

- If using key locks, forward the key separately by registered mail.

OR

- If using numbered seals, record each seal number in your notebook. If more than one shipping container is being sent, note which container has which seal. Include a completed chain-of-custody form in the sample box along with the request-for-analysis form.

AND

- The security box and chain-of-custody form are to be placed into a cooler with freezer packs and packing material and the cooler is to be sealed using a numbered security seal, security tape or lock and hasp.
- All samples and seal numbers are to be recorded.
- Take photographs of the samples of the sealed security box and the sealed cooler.
- The cooler is to be labeled with a “LEGAL SAMPLE” and “THIS SIDE UP” sticker.

If samples are being delivered to the laboratory in person, you should maintain custody of the samples at all times (e.g., lock vehicle doors).

The sampler shall contact the laboratory in advance of any sampling to inform them of the type and number of samples that will be shipped or brought to the laboratory. Alternately, this may be combined with the picking up of sample containers from the laboratory.

If the samples are being shipped by courier, a bill of lading must be prepared and samples taken to the courier or authorized agent. Request next day service for the sample delivery. The bill of lading must identify the laboratory staff who are to receive the samples. Inform the courier of the importance of the legal samples and ensure that they understand the destination. A copy of the bill of lading is to be retained as evidence and the courier information is to be entered into the field notebook.

A copy of the sample submission/chain-of-custody and the shipping documents are to be submitted to the lab. Once the shipment has been sent, phone or fax the laboratory giving details of types and number of samples, sampling date, estimated arrival date, means of sample shipment and transportation company waybill number(s).

If you deliver samples in person to the laboratory, fill out a sample submission form as fully as possible, deliver the samples to the appropriate receiver at the laboratory and make a note of the receiver's name. Have the receiver sign the chain-of-custody form and leave the original form with the receiver. You may want to take a photocopy for your records.

NOTE: Where samples are not required to be kept below 4°C, the freezer pack, vial of water and cooler are not required. Shipment of oil samples must be in accordance with TDG Regulations. If shipping by air, a shipping agent may be used to insure proper packaging and documentation.

4.3.2 DANGEROUS GOODS

Some samples contain dangerous goods (for example fuel samples) and thus are covered by the Transportation of Dangerous Goods Act. Section 1.19 (1) allows for a conditional exemption or samples if the samples are carried by the inspector and are in a suitable means of containment. 1.19 (1) These regulations do not apply to samples of goods including forensic samples, that are reasonably believed to be dangerous goods if, for the purposes of inspection or investigation duties under an Act of Parliament or of a provincial legislature, the samples are:

- (a) in transport under the direct supervision of a federal, provincial or municipal government employee acting in the course of employment; and
- (b) in a means of containment that is designed, constructed, filled, closed, secured and maintained so that under normal conditions of transport, including handling, there will be no accidental release of dangerous goods that could endanger public safety.

Section 1.19 (2) allows for a conditional exemption for any person shipping samples.

1.19 (2) These regulations do not apply to samples of goods that the consignor reasonably believes to be dangerous goods, if:

- (a) the samples are in transport for the purposes of classifying, analyzing, testing or demonstrating;
- (b) the samples are believed not to contain explosives, infectious substances or radioactive materials;
- (c) the gross mass of the samples is less than or equal to 10 kg;
- (d) the samples are accompanied by a shipping document that, despite sections 3.5 and 3.6 of part 3, documentation, includes the name and address of the consigner and the words “test samples” or “échantillons d’ épreuve”;
- (e) the samples are in a means of containment that is designed, constructed, filled, closed, secured and maintained so that under normal conditions of transport, including handling, there will be no accidental release of dangerous goods that could endanger public safety;
- (f) the means of containment has marked on it the words “test samples” or “échantillons d’ épreuve” in letters not less than 25 mm high and in a colour that contrasts with the background colour of the means of containment.

SAMPLE PRESERVATIVES AS DANGEROUS GOODS

Section 1.17 of the Transportation of Dangerous Goods Regulations provide for conditional exemption for the transport of limited quantities. This section applies to the transport of small quantities of preservatives which are dangerous goods often used in sampling. Of particular interest is the shipment of nitric acid (for use in sampling metals) nitric acid preservatives which is less than 70% nitric acid may be shipped by ground under section 1.17 of TDGR if it is in quantities of less than 0.5 liters in a secure means of containment and is marked on the outer package as limited quantities. This section would apply to other preservatives such as hydrochloric acid, sodium hydroxide, formaldehyde, hexane and chromic acid. Use section 1.17 and Schedule 1 of the TDGR to determine the conditions for each dangerous good.

The more stringent requirements of the International Air Transport Association (IATA)/ International Civil Aviation Organization (ICAO) must be met if the shipment is by air. Airlines may refuse a shipment if it is not properly documented, even if there is an exemption for limited quantities. You may have to phone ahead to make an appointment with a TDG-trained employee at the airline company, since one may not always be readily available at smaller airports.

4.3.3 SPECIAL CASES

Toxicity samples are to be placed in 5 gallon plastic pails with plastic (bag) liners. Once the sample has been taken, the pail liner is to be sealed with a numbered seal and then the labeled sample container is to be sealed using two numbered seals or security tape.

All samples must be logged onto a chain-of-custody form including seal numbers for the samples and containers. If samples are being delivered by courier, the chain-of-custody form is placed in a plastic bag. Pictures are to be taken of the sealed pails and the seal numbers recorded in your notebook. The pails are to be labeled with a legal sample label and the bag containing the chain-of-custody form is to be attached to one of the pails.

Bioassay samples, because of their size, can be shipped without special packaging if they are in 20 L - 40 L polyethylene containers, provided the container is properly marked and sealed.

Do not allow water samples to freeze. Freezing may cause the containers to crack as well as alter the sample integrity. Samplers should also be aware of TDG and IATA requirements when shipping preservatives – most are classified as dangerous goods.

5.0 APPENDICES

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- 5.7.18 SECONDARY LEAD SMELTER RELEASE REGULATIONS
- 5.7.19 SULPHUR IN DIESEL FUEL REGULATIONS
- 5.7.20 SULPHUR IN GASOLINE REGULATIONS
- 5.7.21 VINYL CHLORIDE RELEASE REGULATIONS

REFERENCES

5.1 CONVENTIONS

5.1.1 ACRONYMS

AEH	Acid extractible herbicide
AOX	Adsorbable organic halides
BOD	Biochemical (Biological) oxygen demand
BTEX	Benzene, toluene, ethylbenzene, and xylene (see VOC)
CGI	Combustible gas indicator
COD	Chemical oxygen demand
COLIWASA	Composite liquid waste sampler
CP	Chlorinated phenols
CR ⁶	Hexavalent chromium
CRM	Certified reference material
Cu-8	Copper 8
DDAC	Didecyltrimethylammonium chloride
DI	Deionized water
DIC	Dissolved inorganic carbon
DO	Dissolved oxygen
DOC	Dissolved organic carbon

DQO	Data quality objective
EPH	Extractible petroleum hydrocarbon
FC	Fecal coliform
GPS	Global positioning system
HEPH	High extractable petroleum hydrocarbons
H ₂ S	Hydrogen sulphide
IATA	International Air Transport Association
ICAO	International Civil Aviation Organization
IPBC	3-iodo-2-propynylbutyl carbamate
LC ₅₀	Lethal concentration
LEL	Lower explosive limit
LEPH	Low extractable petroleum hydrocarbons
LT ₅₀	Lethal time
LOD	Limits of detection
MMER	Metal mining effluent regulation
NFR	Non-filterable residue (also know as TSS)
NO ₂₊₃	Nitrite + Nitrate
NP	Nitrogen phosphorus
OC	Organochlorine
OD	Outer diameter
ODS	Ozone-depleting substances
OP	Organophosphate
Ortho-P	Ortho Phosphorous
PAH	Polycyclic aromatic hydrocarbons
PCB	Polychlorinated biphenyl
PCDD	Polychlorinated dibenzodioxins
PCDF	Polychlorinated dibenzofurans
PERC	Tetrachloroethylene
ppb	parts per billion
ppm	parts per million
SRM	Standard reference method
Strep	Streptococci
SWOG	Special waste oil and grease
Redox	Oxygen reduction potential
TC	Total coliform

TCDD	tetrachlorodibenzo-para-dioxin
TCDF	tetrachlorodibenzo furan
TCLP	Toxicity Characteristic Leaching Procedure
TCMTB	2-(thiocyanomethylthio) benzothiazole
TDG	Transportation of Dangerous Goods (Act and regulations)
THM	Trihalomethane
TIC	Total inorganic carbon
TOC	Total organic carbon
Total-P	Total Phosphorous
TSS	Total suspended solids, also known as NFR non-filterable residue
TVR	Total volatile residues
SAD	Strong acid dissociable
UEL	Upper explosive limit
VACSAM	Vacuum sampler
VH/VPH	Volatile Hydrocarbon/Volatile Petroleum Hydrocarbon
VOC	Volatile organic carbon
WAD	Weak acid dissociable

5.1.2 CONVERSION FACTORS

WHEN YOU KNOW	MULTIPLY BY	TO FIND
inches	25.4	millimetres (mm)
inches	2.54	centimetres (cm)
feet	0.305	metres (m)
yards	0.914	metres (m)
miles	1.61	kilometres (km)
square inches	6.45	square centimetres (cm ²)
square feet	0.093	square metres (m ²)
square yards	0.834	square metres (m ²)
acres	0.405	hectares (ha)
square miles	2.59	square kilometres (km ²)
cubic inches	16.39	cubic centimetres (cm ³)
cubic feet	0.028	cubic metres (m ³)
cubic yards	0.765	cubic metres (m ³)
ounces	28.35	grams (g)
pounds	0.454	kilograms (kg)
tablespoons	14.71	millilitres (mL)
fluid ounces	28.41	millilitres (mL)
cups	227	millilitres (mL)
quarts (U.S.)	0.95	litres (L)
quarts (Imperial)	1.14	litres (L)
gallons (U.S.)	3.79	litres (L)
gallons (Imperial)	4.55	litres (L)

TO CONVERT DEGREES FAHRENHEIT TO DEGREES CELSIUS

Subtract 32, then multiply the result by 5/9.

5.1.3 UNITS OF MEASURE ABBREVIATIONS

alk	alkali
aq	aqua; aqueous; water
at, atmos	atmosphere
av, avg	average
bar	barometer
bp	boiling point
Bq	Becquerel
C	Coulomb
C	concentration
ci	curie
ca	circa; approximately; about
cm	centimetre
conc	concentration; concentrated
cu	cubic
cyl	cylinder
dB	decibel
dil	dilute
f	frequency
ft	foot
g	gram
h	hour; hecto (prefix)
h	Planch constant; height
H	hydraulic charge
ha	hectare
in	inch
insol	insoluble
J	joule
k	kilo (prefix)
K	Kelvin; absolute temperature
kg	kilogram
km	kilometer
L	litre
L	length
LD ₅₀	median lethal dose
liq	liquid

m	metre; milli (prefix)
M	mega (prefix); molar
m ²	square metre
m ³	cubic metre
max	maximum
mg	milligram
min	minute
mL	millilitre
mol	mole; molecule
mole	gram- molecular weight
mp	melting point
n	nano (prefix)
N	normal
ng	nanograms (10 ⁻⁹)
no	number
°C	degree(s) Celsius
°F	degree(s) Fahrenheit
p	pico (prefix)
P	pressure
pH	measure of acidity/alkalinity
ppb	parts per billion
ppm	parts per million
precip	precipitated
P _s	standard pressure
psi	pounds per square inch
rpm	revolutions per minute
s	second
sq	square
sol	soluble
stp	standard temperature and pressure
t	general temperature; time; tonne
T	absolute temperature; tera (prefix) (3 900 (1-a/d) 1.88)
μ	micro; micron μm (prefix)
μg	microgram
V	volume
W	watt; d ^{2.48}
wt	weight

giga (G) 1 000 000 000	a thousand millions = a billion
mega (M) 1 000 000	a million
kilo (k) 1 000	a thousand
hecto (h) 100	a hundred
deca (da)10	ten
deci (d) 0.1	a tenth
centi (c) 0.01	a hundredth
milli (m) 0.001	a thousandth
micro (μ) 0.000 001	a millionth
nano (n) 0.000 000 001	a thousand millionth

5.2 ENFORCEMENT REGULATIONS AND GUIDELINES



Many enforcement regulations and guidelines contain specific sampling and analytical requirements (by way of standard reference methods) that will affect how sampling is to be done at regulated facilities or when analyzing certain regulated substances.

Since regulations may be in the process of revision and new regulations appear regularly please see web sites for a list of current regulations and acts 1) administered by the minister of the Environment, 2) acts administered in part by Environment Canada and 3) acts administered by others with Environment Canada assistance.

www.ec.gc.ca/ceparegistry

www.ec.gc.ca/enviroregs

5.3 CHECKLIST OF EQUIPMENT FOR FIELD SAMPLING

This list is by no means exhaustive, but will serve as a guide. Not all equipment is required for each field sampling trip, but the list may be helpful in reminding you of items you might otherwise overlook.

STATIONERY

- Analysis request forms
- Calculator
- CEPA/FA inspector card
- Chain-of-custody forms
- Conversion tables
- Notebook, waterproof
- Occurrence report forms
- Paper, waterproof
- Pens, pencils, permanent markers
- Space pen
- Statement forms
- Warning card

ELECTRONIC EQUIPMENT

- Audio recorder
- Batteries and battery charger
- Binoculars
- Camera
- Cellular phone
- Computer
- Gas monitors
- Global positioning system
- Pager
- Photographic film
- Spare video or audio tape

Video camera

VHF radio

REFERENCE MATERIALS

Aerial photos (if possible)

Charts (hydrographic)

Compass

Compliance files

Emergency Response Guide (Canutec)

THE INSPECTOR'S SAFETY GUIDE

THE INSPECTOR'S FIELD SAMPLING MANUAL

Instrument operation manuals: pH meter, conductivity meter, etc.

Maps (topographical, road)

Other protocols or manuals for sampling

Regional emergency contact list

SAFETY AND FIRST AID

Air pack (self contained breathing apparatus-SCBA)

Apron, splash

Aspirin

Blanket

Boot covers, disposable

Ear protection gear

Emergency rations/survival kit

Eye wash kit

Fire extinguisher

Fire resistant clothing

First aid kit

Flares

Flotation vest/suit

Goggles, 2 pair

Hardhat

Respirator

Respirator filters

Rubber boots

Safety harness (fall protection)

Suits, disposable yellow, x 3

Whistle

Work boots (CSA)

PERSONAL SUPPLIES

Backpack

Hand lotion

Hand soap

Insect repellent

Matches in waterproof container

Rain gear

Reflective blanket

Reflective vest

Sample vest

Sunglasses

Sunscreen

Toilet paper

Water

SAMPLE PACKING MATERIALS

Cooler

Electrical tape

Gel ice packs

Labels, sticky

Masking tape

Sample box, lockable (tool kit box with lock)

Seals, legal

Shipping labels

Shipping waybills

Stickers (“THIS END UP,” “FRAGILE,” “KEEP COOL,” “DO NOT FREEZE,” “KEEP FROZEN,” etc.)

Tags, cardboard, with wire attachments

TDG and WHMIS labels

TOOLS

Axe

Batteries (dispose of as hazardous waste)

Flashlight

Knife, exacto

Knife, regular

Ladder

Manhole opener

Measuring tape

Pick

Scissors

Shovel

Stopwatch

Toolbox and tools; hammer, screwdrivers, nails, wrenches, measuring tape, etc.

SAMPLING TOOLS

Acid-washed membrane filters, 0.45 μm (for metals)

Aluminum foil

Bioassay container

Bottles, HDPE, with caps (500 mL, 1 L, 2 L)

Bottles, amber glass, with Teflon-lined caps (1 L)

Buckets, graduated

Drip trays

Filters, disposable (for field filtering of metal samples)

Funnel

Gloves, latex

Gloves, polyethylene

Kimwipes

Paper towels

Pipettes, disposable (5 mL)

Pipettes, disposable (50 mL, for transformer sampling)

Scoops, plastic and stainless steel

Spatulas, plastic or metal

Stainless steel chain for bucket

Syringes, disposable (for field filtering of metal samples)

Test tubes

Trowel

Tweezers, teflon-coated

SAMPLERS

COLIWASA

Extended bottle sampler (Weaton Dip)

Flowmeter and tables

Grain sampler

Kemmerer depth sampler

Open tube (thief) sampler

Ponar dredge

Pond (dip) sampler

Pump heads

Pump, peristaltic

Pump, submersible

Pump tubing connectors

Pump tubing, silicon

Sampling iron

Sampling pole

Sampling trier

Sequential autosampler

Soil-sampling rod

Split-spoon corer

Thin-wall corer

VACSAM

Weighted bottle sampler

LEGAL SAMPLING EQUIPMENT

Bioassay container
Cooler
Cooler seals
Chain-of-custody form
Disposable gloves
Duct tape
Gel ice packs
Labels, two-part uniquely numbered, permanent adhesive
Notebook
Sample box, lockable (tool kit box with lock)
Sample bottles
Seals, legal
Sealable plastic bags
Scribes
Permanent marker
Preservatives

FIELD TEST EQUIPMENT

Conductivity meter and calibration standards
pH meter
pH buffers
pH paper
Thermometer
Dissolved oxygen meter and standards

CHEMICALS/PRESERVATIVES

Acetone
Deionized (DI) water
Dye, colour marker
Hexane
Hydrochloric acid, concentrated (for chlorinated phenols)
Mercury preservative (1 mL, 5% potassium dichromate + 1 mL, 70% nitric acid; obtain from lab)
Metal preservative (1 mL, 35% nitric acid; obtain from lab)

Nitric acid (70%)

Potassium iodide (5%)

Rexonic N25-7 solution

Sodium carbonate (0.5N for sulphide sample)

Sodium hydroxide (3N)

Sodium hydroxide (40% for cyanide samples)

Sodium hydroxide pellet

Sodium sulphite, granular (for chlorinated dioxins/furans)

Sodium thiosulphate solution (10% for fecal coliform samples)

Starch solution (BDH or Fisher Scientific)

Sulphuric acid, concentrated (for COD)

Zinc acetate (0.1M, for sulphide sample)

CLEANUP SUPPLIES

Garbage bags, kitchen

Garbage bags, large

Paper towels

Spill kit, commercial or baking soda

Vinegar

Cat litter or vermiculite

Absorbent pads (for hydrocarbons)

Disposable gloves

5.4 CONTACT NUMBERS

Laboratory contacts – Environment Canada

ATLANTIC REGION

ECB Laboratory

Moncton, New Brunswick

Organics	(506) 851-2892
Inorganics	(506) 851-2896
Toxicology	(506) 851-2885
Sample submission	(506) 851-2899
General	(506) 851-6606

QUEBEC REGION

ECB Laboratory

St. Lawrence Centre

Montreal, Quebec

Chemistry	(514) 283-2665
Toxicology	(514) 496-7106
General	(514) 283-7000

ONTARIO REGION

ECB Laboratory

National Capital Region

Organics (613) 990-8559

Inorganics (613) 990-8560

Waste Water Technology Centre

Burlington, Ontario

Organics/Inorganics (905) 336-4750

Sample Submission (905) 336-6933

General (905) 336-6447

PRAIRIE AND NORTHERN REGION

EP Laboratory

Edmonton, Alberta

Organics (403) 435-7251

Inorganics (403) 435-7376

Toxicity (403) 435-7242

General (403) 435-7335

PACIFIC AND YUKON

ECB Laboratory

North Vancouver, British Columbia

Scientific Support (604) 924-2532

Chemistry (604) 924-2531

Environmental Toxicology (604) 924-2513

General (604) 924-2500

Sample Submission (604) 924-2507

5.5 HEALTH AND SAFETY LEGISLATION

DUTIES OF EMPLOYERS

(SECTION 125 OF THE CANADA LABOUR CODE)

The general duty of the employer is to “ensure that the health and safety at work of every person employed by the employer is protected.”

The following specific duties of employers and employees relate directly to the field operations of inspectors and investigators.

. . (.)every employer shall, in respect of every workplace controlled by the employer:

ACCIDENT INVESTIGATION AND REPORTING

“investigate, record and report in the manner and to the authorities as prescribed all accidents, occupational diseases and other hazardous occurrences known to the employer.” [part (c)]

FIRST AID

“provide prescribed first-aid facilities and health services.” [part (h)]

POTABLE WATER

“provide, in accordance with prescribed standards, potable water.” [part (j)]

VEHICLES AND MOBILE EQUIPMENT

“ensure that the vehicles and mobile equipment used by the employees in the course of their employment meet prescribed standards.” [part(k)]

PERSONAL PROTECTIVE EQUIPMENT

“provide every person granted access to the workplace by the employer with prescribed safety materials, equipment, devices and clothing.” [part(l)]

FIRE AND EMERGENCY MEASURES

“comply with prescribed standards relating to fire safety and emergency measures.” [part (o)]

INFORMATION AND TRAINING

“provide, in the prescribed manner, each employee with the information, instruction, training and supervision necessary to ensure their health and safety at work.”[part (q)]

HAZARD AWARENESS

“ensure that each employee is made aware of every known or foreseeable health or safety hazard in the area where the employee works.” [part (s)]

MACHINERY, EQUIPMENT AND TOOLS

“ensure that the machinery, equipment and tools used by the employees in the course of their employment meet prescribed health, safety and ergonomic standards and are safe under all conditions of their intended use.” [part (t)]

SAFETY CODES AND STANDARDS

“adopt and implement prescribed safety codes and safety standards.” [part v]

USE OF PERSONAL PROTECTIVE EQUIPMENT

“ensure that every person granted access to the workplace by the employer is familiar with and uses in the prescribed circumstances and manner all prescribed safety materials, equipment, devices and clothing.” [part w]

ORAL AND WRITTEN DIRECTION

“comply with every oral or written direction given to the employer by an appeals officer or a health and safety officer concerning the health and safety of employees.” [part(x)]

DUTIES OF EMPLOYEES (SECTION 126 OF THE CANADA LABOUR CODE) While at work, every employee shall...

PERSONAL PROTECTIVE EQUIPMENT

“use any safety materials, equipment, devices and clothing that are intended for the employee’s protection and furnished to the employee by the employer or that are prescribed.” [part (a)]

PRESCRIBED PROCEDURES

“follow prescribed procedures with respect to the ‘...health and safety...’ of employees.” [part (b)]

PRECAUTIONS

“take all reasonable and necessary precautions to ensure the health and safety of the employee, the other employees and any person likely to be affected by the employee’s act or omissions.” [part (c)]

INSTRUCTIONS

“comply with all instructions from the employer concerning the health and safety of employees,” [part (d)]

HEALTH AND SAFETY COMMITTEE

“cooperate with the policy and work place committees or the health and safety representative.” [part (f)]

HAZARDS

“report to the employer any thing or circumstance in a work place that is likely to be hazardous to the health or safety of the employee, or that of the other employees or other persons granted access to the work place by the employer.” [part (g)]

ACCIDENTS AND INJURY

“report in the manner prescribed every accident or other occurrence arising in the course of or in connection with the employee’s work that has caused injury to the employee or to any other person.” [part (h)]

ORAL AND WRITTEN DIRECTION

“comply with every oral or written direction of a health and safety officer or an appeals officer concerning the health and safety of employees.” [part (i)]

WORKPLACES NOT CONTROLLED BY ENVIRONMENT CANADA

Many of your inspections and investigations will occur at other federal facilities or at private facilities regulated by provincial legislation. The legislative protection under the Canada Labour Code still applies, no matter where you are working.

FEDERAL FACILITIES

If you are inspecting another federal facility, Environment Canada must continue to protect your safety and health. The federal facility must also ensure your safety and health. To them, you are a visitor or “person granted access to the workplace.” [Section 125, parts (l) and (w)]

PROVINCIAL FACILITIES

If you are inspecting private corporations or facilities regulated by provincial health and safety legislation, both you and your employer continue to be responsible for your health and safety. Some provincial occupational health and safety standards may differ from federal standards. However, the differences will probably be insignificant. Most provincial Occupational Health and Safety legislation protects you as a visitor (or a person granted access to the workplace) in the same manner as the Labour Code.

Under Section 128 of the Labour Code, there are provisions for employees to refuse dangerous work. You should familiarize yourself with the procedures and conditional clauses of this section, so that you fully understand its limitations.

TREASURY BOARD POLICIES, GUIDELINES AND PROCEDURES

All inspectors and investigators should familiarize themselves with the following Treasury Board Directives, Standards and Guides, etc. which may be applicable to the nature of their field duties:

TREASURY BOARD OSH

CHAPTER	TITLE
Directive 2-05	First-Aid Safety and Health Directive
Directive 2-07	Hazardous Confined Space
Directive 2-09	Tools and Machinery Directive
Directive 2-11	Motor Vehicle Operations Directive
Standard 2-13	Occupational Health Evaluation Standard
Directive 2-14	Personal Protective Equipment Directive
Directive 2-16	Elevated Work Structures Directive
Directive	Hazardous Substances Directive
Guide 5-3	Safety Guide for Operations Over Ice
Guide 5-4	Safety Guide for Field Operations
Advisory Notice 6-3	The Effects of Extreme Cold
Advisory Notice 6-4	Occupational Exposure to Benzene

OTHER LEGISLATION

You must always be aware of and comply with:

- provincial/territorial OSH legislation
- municipal health and safety laws
- bylaws and other regulations that govern local jurisdictions

If you are uncertain as to the applicability of any laws, statutes or regulations, consult:

- your supervisor
- your local health and safety committee
- a regional health and safety advisor or the appropriate governing authority

5.6 FLOW CALCULATIONS AND FORMULAS

5.6.1 CIRCLES: AREAS OF SEGMENTS

H = HEIGHT
D = DIAMETER
A = AREA

H/D	A
0.001	0.000 04
0.010	0.001 33
0.020	0.003 75
0.030	0.006 87
0.040	0.105 4
0.050	0.148 6
0.060	0.019 24
0.070	0.024 17
0.080	0.029 43
0.090	0.035 01
0.100	0.040 87
0.110	0.470 1
0.120	0.053 38
0.130	0.060 00
0.140	0.066 83
0.150	0.073 87
0.160	0.081 11

H/D	A
0.170	0.088 54
0.180	0.096 13
0.190	0.103 90
0.200	0.111 82
0.210	0.119 90
0.220	0.128 11
0.230	0.136 46
0.240	0.144 94
0.250	0.153 55
0.260	0.162 26
0.270	0.171 09
0.280	0.180 02
0.290	0.189 05
0.300	0.198 17
0.310	0.207 38
0.320	0.216 67
0.330	0.226 03

H/D	A
0.340	0.235 47
0.350	0.244 98
0.360	0.254 55
0.370	0.264 18
0.380	0.273 86
0.390	0.283 59
0.400	0.293 37
0.410	0.303 19
0.420	0.313 04
0.430	0.322 93
0.440	0.332 84
0.450	0.342 78
0.460	0.352 74
0.470	0.362 72
0.480	0.372 70
0.490	0.382 70
0.500	0.392 70

To calculate the amount of cross-sectional area in a pipe that is not occupied by water, use this table:

1. Divide the height of the segment occupied by water (H) by the diameter D .
2. From the table, determine the area A for the ratio of H/D .
3. Square the diameter D .
4. Multiply $A \times D^2$.

If the ratio H/D is greater than 0.500, go through the same calculation (steps 1 to 4), using the height of the segment that is not occupied by water. Determine the cross-sectional area of the pipe ($A = \pi r^2$) and subtract the segment area.

5.6.2 FLOW ESTIMATING - PARTIAL-FILLED CHANNELS OR PIPES

BUCKET AND STOPWATCH: This is the simplest method, provided that the entire discharge can be retained in the container for a reasonable length of time. The procedure is to catch the entire flow for a predetermined period, measured by a stopwatch. The contents of the bucket are then measured. The results of three catches should be averaged to provide a final figure for flow determination. How long? The usual minimum time is 5 seconds, but a shorter period may still give more accurate results than other methods.

VELOCITY AND CROSS-SECTIONAL AREA: Either the surface velocity or average velocity can be measured. The average velocity is equal to approximately 0.85 of the surface velocity.

To measure surface velocity, measure and record the travel time of a floating object (cork, piece of wood) over a given distance where the flow is straight and free of obstructions. The longer the distance, the more accurate the result. Repeat at least three times; average the results then determine the average stream velocity by multiplying the average surface velocity by 0.85.

To measure average velocity directly, use a velocity meter (or pitot tube) and measure the velocity at the 0.6 depth; or, if the flow is uniform, determine the average flow by taking several readings throughout the flow cross-section (see below).

To determine the cross-sectional area of flow in a pipe:

1. If the water fills the pipe completely, determine the cross-sectional area by measuring the diameter of the pipe and using the formula $A = \pi r^2$.
2. If the water only partly fills the pipe, measure the diameter and the height of the water. Divide the height by the diameter and use Table 5.6.1 to find the cross-sectional area.
3. Multiply the cross-sectional area by the average velocity of flow.

IRREGULAR CHANNEL: To determine flow in an such as a ditch or stream bed, take several depth measurements across the channel. Plot these on graph paper. If the results take a regular shape such as a rectangle, determine the area of the rectangle. Alternatively, count squares to determine the area. Calculate flow rate by multiplying the estimated cross-sectional area by the average velocity of flow.

CALIFORNIA PIPE METHOD: To determine the rate of discharge when the flow drops from the end of a horizontal pipe, first ensure that the length of the pipe is at least six times the diameter. Measure the pipe diameter (D) and the distance from the water surface to the top of the pipe (A). Divide A/D. Because the tables are American, measure in feet; the results will be in U.S. gallons per minute.

Calculate the flow rate (Q) from the following equation:

Q	=	TW
T	=	$8.69 (1-a/d)^{1.88}$
W	=	$d^{2.48}$

Consult Tables 5.6.3 and 5.6.4 for values for (T) and (W) for A/D.

5.6.3 FLOW BY CALIFORNIA PIPE METHOD

Values of T for California Pipe Flow Formula based on A/D values

A/D (FT.)	T
0.00	3 900
0.01	3 830
0.02	3 760
0.03	3 690
0.04	3 610
0.05	3 540
0.06	3 470
0.07	3 400
0.08	3 330
0.09	3 260
0.10	3 200
0.11	3 130
0.12	3 070
0.13	3 000
0.14	2 930
0.15	2 870
0.16	2 810
0.17	2 750
0.18	2 690
0.19	2 630
0.20	2 570
0.21	2 510
0.22	2 450
0.23	2 390
0.24	2 330
0.25	2 270
0.26	2 210
0.27	2 160
0.28	2 100
0.29	2 050

A/D (FT.)	T
0.30	1 990
0.31	1 940
0.32	1 890
0.33	1 840
0.34	1 790
0.35	1 740
0.36	1 690
0.37	1 640
0.38	1 590
0.39	1 540
0.40	1 490
0.41	1 450
0.42	1 400
0.43	1 350
0.44	1 310
0.45	1 270
0.46	1 230
0.47	1 180
0.48	1 140
0.49	1 100
0.50	1 060
0.51	1 020
0.52	930
0.53	915
0.54	905
0.55	870
0.56	830
0.57	800
0.58	760
0.59	730

A/D (FT.)	T
0.60	700
0.61	660
0.62	630
0.63	600
0.64	570
0.65	540
0.66	510
0.67	480
0.68	460
0.69	430
0.70	410
0.71	380
0.72	360
0.73	330
0.74	310
0.75	290
0.76	270
0.77	250
0.78	230
0.79	210
0.80	190
0.81	170
0.82	160
0.83	140
0.84	125
0.85	110
0.86	97
0.87	85
0.88	73
0.89	61
0.90	51
0.91	42
0.92	34
0.93	26
0.94	20
0.95	14
0.96	9
0.97	5
0.98	3
0.99	1

Where A = distance from water surface to top of pipe, D = diameter of pipe

Values of W for California pipe flow formula

PIPE DIAMETER (INCHES)	A (FEET)	W
3	0.25	0.032
4	0.33	0.064
6	0.50	0.179
8	0.67	0.370
10	0.83	0.630
12	1.00	1.00
14	1.17	1.48
15	1.25	1.74
16	1.33	2.03
18	1.50	2.73
20	1.67	3.57
21	1.75	4.01
22	1.83	4.48
24	2.00	5.58
27	2.25	7.47
30	2.50	9.70
33	2.75	12.29
36	3.00	15.25

5.6.4 WEIRS AND FLUMES



Weirs and flumes are specially designed shapes or obstructions in a channel over which water passes. Because they are designed to specific dimensions the height (head) of the water in the device measured at a specified point can be related by equation or table to a specific flow rate. Common designs are; rectangular weir, V-notch weir, Cipolletti weir, Parshall flume, Palmer-Bowlus flume, h-type flume, Trapezoidal flume, etc. Extensive information on weirs and flumes are available in the Water Measurement Manual, 3rd edition, revised 2001, Bureau of Reclamation, U.S. Department of the Interior. It can be downloaded

from the Water Resources Research Laboratory web site:
www.usbr.gov/pmts/hydraulics_lab/pubs.

TRIANGULAR (V) NOTCH WEIRS

The angle of a triangle V-notch is usually 90° but can be 60°. Portable weirs can be installed in ditches or streams.

In installing weirs, consider the following criteria:

- The end contraction should be at least 3/4 the maximum notch width (L).
- The head (H) should be at least 9 cm.
- (H) should be measured at least 2.5 times (H) upstream of the weir.

The flow rate (Q) can be calculated from the following equations:

$$Q = 2.49H^{2.5} \text{ for a } 90^\circ \text{ weir}$$

$$Q = 1.41H^{2.5} \text{ for a } 60^\circ \text{ weir}$$

DISCHARGES FROM TRIANGULAR NOTCH WEIRS
WITH END CONTRACTIONS

HEAD OF WATER (IN)	FLOW IN GAL/MIN 90° NOTCH	FLOW IN GAL/MIN 60° NOTCH
1	2.19	1.27
2	12.4	7.16
3	34.2	19.7
4	70.2	40.5
5	123	70.8
6	193	112
7	284	164
8	397	229
9	533	308
10	694	401
11	880	508
12	1 094	632
13	1 337	772
14	1 609	929
15	1 912	1 104
16	2 246	1 297
17	2 614	1 509
18	3 016	1 741
19	3 452	1 993
20	3 924	2 266
21	4 433	2 560
22	4 950	2 875
23	5 565	3 213
24	6 190	3 574

PARSHALL FLUME OPEN CHANNEL FLOW MEASUREMENT

FLUME SIZE (METERS)	OR	FLUME SIZE (INCHES)	APPLICABLE DISCHARGE RANGE (M ³ S ⁻¹)		EQUATION Q = Ch ^N / 1000 (M ³ S ⁻¹)*
			Min.	Max	
0.025		1	0.000 3	0.005 4	0.0479h ^{1.55} /1 000
0.051		2	0.000 6	0.013 2	0.0959h ^{1.55} /1 000
0.076		3	0.000 9	0.032 1	0.141h ^{1.55} /1 000
0.152		6	0.001 4	0.111	0.264h ^{1.58} /1 000
0.229		9	0.002 6	0.251	0.393h ^{1.53} /1 000
0.305		12	0.003 1	0.457	0.624h ^{1.522} /1 000
0.457		18	0.004 2	0.695	0.887h ^{1.538} /1 000
0.61		24	0.011 9	0.937	1.135h ^{1.550} /1 000
0.914		36	0.017 3	1.427	1.612h ^{1.566} /1 000
1.219		48	0.036 8	1.923	2.062h ^{1.578} /1 000
1.524		60	0.045 3	2.424	2.5h ^{1.587} /1 000
1.829		72	0.073 6	2.929	2.919h ^{1.595} /1 000
2.134		84	0.085 0	3.438	3.337h ^{1.601} /1 000
2.438		96	0.099 1	3.949	3.736h ^{1.607} /1 000

Table adapted from American Society for Testing and Materials (ASTM) Standard D1941-91

* Where h = head (in cm), measured upstream from the throat, 2/3 of the distance of the converging section length.

NOTE: Assumption is that flume is free flowing. If the flow is submerged, the discharge calculation will have to be compensated.

EXAMPLE

6 inch flume at 27 cm of head

$$= 0.264 \times 27 \text{ cm}^{1.58} / 1\ 000$$

= 0.048 cubic meters per second

To convert inches to centimeters, multiply by 2.54

For additional information on flumes refer to EPS 2/MM/4 APRIL 2001, GUIDANCE DOCUMENT FOR FLOW MEASUREMENT OF METAL MINING EFFLUENTS.

5.7 REGULATORY TABLES

TABLE 5.7.1 ASBESTOS MINES AND MILLS
RELEASE REGULATIONS

PARAMETER	MILL OPERATION	LIMIT	TYPE
Asbestos fibres	Crushing, drying or milling operations; dry rock storage; or primary dry drilling operations in an open pit	Two asbestos fibres per normal cubic centimetre	Filter

TABLE 5.7.2 BENZENE IN GASOLINE
REGULATIONS

PARAMETER	SOURCE	LIMIT % BY VOLUME	TYPE
Benzene	Gasoline	1.0 (flat)	Grab
		1.5 (cap)	Grab
		0.95 (pool average)	Average

TABLE 5.7.3 CHLOR-ALKALI MERCURY
RELEASE REGULATIONS

PARAMETER	SOURCE	LIMIT	TYPE
Mercury (Hg)	Ventilation gases from cell rooms	5 g/day per 1 000 kg of rated capacity	Stack samples
	H gas stream from denuders	0.1 g/day per 1 000 kg of rated capacity from each source	Stack samples
	Ventilation gases from end boxes and tanks	0.1 g/day per 1 000 kg of rated capacity from each source	Stack samples
	Gases exhausted from retorts	0.1 g/day per 1 000 kg of rated capacity from each source	Stack samples
	Total from all sources	1.68 kg/day	Stack samples

TABLE 5.7.4 CHLOROBIPHENYL
REGULATIONS

PARAMETER	SAMPLE*	LIMIT (MG/L)	TYPE
PCB	Products, machinery or equipment	50	Grab
	Oil applied to a road surface	5	Grab

*See regulations for more details

TABLE 5.7.5 DISPOSAL AT SEA
REGULATIONS

PARAMETER	MATERIAL	LIMIT	TYPE
Cadmium and its compounds	Waste or other matter	0.6 mg/kg (dry weight)	Grab
Mercury and its compounds	—	0.75 mg/kg (dry weight)	Grab
PAHs	—	2 500 µg/kg (dry weight)	Grab
PCBs	—	100 µg/kg (dry weight)	Grab
Persistent plastics and other persistent synthetic materials in a comminuted form	4% by volume		Grab

TABLE 5.7.6 FEDERAL MOBILE PCB TREATMENT AND DESTRUCTION REGULATIONS

PARAMETER	RELEASE INTO ENVIRONMENT	LIMIT	TYPE
PCB	Operating to reduce PCBs in oil to:	2 mg/kg	Grab
Particulate matter	Gas	50 mg/m3	Stack sample
HCL	Gas	75 mg/m3	Stack sample
2,3,7,8-PCDDs / PCDFs	Gas	12 ng/m3	Stack sample
PCB	Liquid	5 µ/L	Grab
2,3,7,8-PCDDs / PCDFs	Liquid	0.6 ng/L	Grab
PCB	Solid	0.5 mg/kg	Grab
2,3,7,8-PCDDs / PCDFs	Solid	1 µg/kg	Grab
PCB	Gas from destruction systems	1 mg/kg of PCB treated	Stack sample

TABLE 5.7.7 GASOLINE REGULATIONS

PARAMETER	USE OF GASOLINE	LIMIT (MG/L)	TYPE
Lead	Produced for farm vehicles, boats or trucks > 3 856 kg	30	Grab
	Imported for farm vehicles, boats or trucks > 3 856 kg	26	Grab
	For all other uses	5	Grab
Phosphorus	Unleaded fuel	1.3	Grab

TABLE 5.7.8 GLYCOL GUIDELINES

PARAMETER	SOURCE	LIMITS *	TYPE
Glycol	Discharge to surface water	100 mg/L	Grab

*Average of two samples, taken between 30 minutes and 24 hours apart

TABLE 5.7.9 MEAT AND POULTRY
PRODUCTS PLANT LIQUID
EFFLUENT REGULATIONS

PARAMETER	EFFLUENT SOURCE	LIMITS*		TYPE
		Actual	Monthly Average	
pH	All plants> 6.0 and < 9.0			Composite
BOD	Red meat	1.0	0.5	Composite
TSM	Integrated plant	1.2	0.6	
Grease		1.6	0.8	
BOD	Processing plant	0.7	0.35	Composite
TSM		0.5	0.25	
Grease		0.8	0.4	
BOD	Poultry	1.4	0.7	Composite
TSM	Integrated plant	1.0	0.5	
Grease		1.0	0.5	
BOD	Rendering plant	0.4	0.2	Composite
TSM		0.4	0.2	
Grease		0.3	0.15	

*Authorized daily deposit) (kg/t of product

TABLE 5.7.10 METAL MINING LIQUID
EFFLUENT REGULATION

DELETERIOUS SUBSTANCE	MAXIMUM MONTHLY MEAN	MAXIMUM IN A COMPOSITE SAMPLE	MAXIMUM IN A GRAB SAMPLE
Arsenic (As)	0.50 mg/L	0.75 mg/L	1.00 mg/L
Copper (Cu)	0.30 mg/L	0.45 mg/L	0.60 mg/L
Cyanide (CN)	1.00 mg/L	1.50 mg/L	2.00 mg/L
Lead (Pb)	0.20 mg/L	0.30 mg/L	0.40 mg/L
Nickel (Ni)	0.50 mg/L	0.75 mg/L	1.00 mg/L
Zinc (Zn)	0.50 mg/L	0.75 mg/L	1.00 mg/L
TSS	15.00 mg/L	22.50 mg/L	30.00 mg/L
²²⁶ Ra	0.37 Bq/L	0.74 Bq/L	1.11 Bq/L
pH	not < 6.0 but not >9.5		

TABLE 5.7.11 PETROLEUM REFINERY
EFFLUENT REGULATIONS

PARAMETER	SAMPLE	MONTHLY AMOUNT KG/M ³	ONE DAY AMOUNT KG/M ³	MAX. DAILY AMOUNT KG/M ³	TYPE
Oil and grease	Liquid effluent or once-through cooling water	0.008 6	0.016	0.021	24 h composite, see Schedule IV subsection 2(2)
Phenol	Liquid effluent or once-through cooling water	0.000 86	0.001 6	0.002 1	24 h composite, see Schedule IV subsection 2(2)
Sulphide	Liquid effluent or once-through cooling water	0.000 28	0.000 86	0.001 4	24 h composite, see Schedule IV subsection 2(2)
Ammonium nitrogen	Liquid effluent or once-through cooling water	0.010	0.016	0.021	24 h composite, see Schedule IV subsection 2(2)
Total Suspended Matter	Liquid effluent or once-through cooling water	0.021	0.034	0.043	24 h composite, see Schedule IV subsection 2(2)
pH	Liquid effluent or once-through cooling water	Limit between 6.0 and 9.5			24 h composite, see Schedule IV subsection 2(2)

TABLE 5.7.12 PHOSPHORUS
CONCENTRATION
REGULATIONS

PARAMETER	SAMPLE	LIMIT %	TYPE
P ₂ O ₅	Per batch	5	Grab
or as P	Per batch	2.2	Grab

**TABLE 5.7.13 POLLUTION PREVENTION
PROVISIONS OF THE
FISHERIES ACT**

PARAMETER	SOURCE	LIMIT	TYPE
Bioassay test	Any substance, whether liquid, solid or gas	A substance is deleterious if, when added to water, it would degrade or alter or form part of a process of degradation or alteration of the quality of that water so that water is rendered deleterious to fish or to use by man or to fish that frequent that water. Deleterious meaning to be harmful to health.	Grab

**TABLE 5.7.14 POTATO PROCESSING
PLANT LIQUID EFFLUENT
REGULATION**

PARAMETER	EFFLUENT SOURCE	LIMITS*		TYPE
		Actual	Average	
pH	All plants	> 6.0 and	< 9.0	Composite
BOD	Potato chip plant	1.5	0.5	Composite
TSM		2.1	0.7	
BOD	Other potato	2.7	0.9	Composite
TSM	product plants	2.4	0.8	

*Authorized daily deposit (kg/t raw potato processed)

TABLE 5.7.15 PULP AND PAPER
EFFLUENT REGULATIONS

PARAMETER	SAMPLE	LIMITS*	MAX. MONTHLY	TYPE**	
		MAX. 24 H		SINGLE EFFLUENT	MULTIPLE EFFLUENT
BOD	Effluent	12.5 x RPR	8.75 x RPR x D	Composite	Composite of grabs
TSS	Effluent	7.5 x RPR	11.25 x RPR x D	Composite	Composite of grabs
Bioassay	Effluent	96 h LC ₅₀ > than 100%		Grab	Grab
Daphnia magna	Effluent	48 h LC ₅₀ > than 100%		Grab	Grab

*x reference production rates

**For reduced monitoring use Grab samples

TABLE 5.7.16 PULP AND PAPER MILL
DEFOAMER AND WOOD
CHIP REGULATIONS

PARAMETER	PRODUCT	LIMIT (PPB)	TYPE
Dibenzofuran	Defoamer	40	Grab
Dibenz-para-dioxin	Defoamer	10	Grab
Polychlorinated phenols	Wood chips	Undetectable	Grab

TABLE 5.7.17 PULP AND PAPER MILL
EFFLUENT CHLORINATED
DIOXINS AND FURANS
REGULATIONS

PARAMETER	SAMPLE	LIMIT	TYPE
2, 3, 7, 8-TCDD	Effluent	Undetectable	24 h composite
2, 3, 7, 8-TCDF	Effluent	Undetectable	24 h composite

TABLE 5.7.18 SECONDARY LEAD
SMELTER RELEASE
REGULATIONS

PARAMETER	SOURCE	LIMIT (G/M ³)	TYPE
Particulate matter	Operations using blast furnaces, cupolas or reverberatory furnaces	0.046	Stack sample
—	Operations using holding furnaces, kettle furnaces or lead oxide units using scrap handling and material handling, crushing, furnace tapping, furnace slagging, furnace cleaning or casting	0.023	Stack sample
Lead matter	Operations using blast furnaces, cupolas or reverberatory furnaces	0.029	Stack sample
—	Operations using holding furnaces, kettle furnaces or lead oxide units using scrap handling and material handling, crushing, furnace tapping, furnace slagging, furnace cleaning or casting	0.014	Stack sample

TABLE 5.7.19 SULPHUR IN DIESEL
FUEL REGULATIONS

PARAMETER	SOURCE	LIMIT	TYPE
Sulphur	Diesel on road	500 ppm	Grab
As of June 2006		15 ppm	Grab

TABLE 5.7.20 SULPHUR IN GASOLINE
REGULATIONS

PARAMETER	SOURCE	LIMIT	TYPE
Sulphur	Gasoline	80 ppm (cap)	Grab
		30 ppm (pool average)	Pool Average
As of April 1, 2005		40 ppm (cap)	Grab
Retail sales, as of April 1, 2005		80 ppm (pool average)	Pool Average

TABLE 5.7.21 VINYL CHLORIDE RELEASE REGULATIONS

PARAMETER	SOURCE	LIMIT	TYPE
Vinyl Chloride	Process vents	> 10 ppm and 2 kg/day	Stack sample
	Opening of polymerization reactor (homo-polymer suspension polymerized resins)	0.002 kg/100 kg	Stack sample
	Downstream of slurry stripper (bulk polymerized resins)	0.02 kg/100 kg	Stack sample
	Downstream of resin stripper (bulk polymerized resins)	0.04 kg/100 kg	Stack sample
	Downstream of slurry stripper (dispersion polymerized resins/copolymer resins)	0.2 kg/100 kg	Stack sample
	Release from recovery system; exhausting/purging of polymerization reactor, slurry stripper or resin stripper; slurry stripping or resin stripping procedures; depressurizing of polymerization reactor	10 ppm	Stack sample

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