# GUIDELINE ON SAMPLING, HANDLING, TRANSPORTING, AND ANALYZING LEGAL WASTEWATER SAMPLES

Prepared for:

**Canadian Water and Wastewater Association**

Prepared by:

**Goce Vasileski**
Consultant
391 Wiffen Pvt.
Ottawa, ON K2H 1G4
Tel: 613-421-1023
E-mail: gocevasile@yahoo.com.au

## Table of Contents

## 1. INTRODUCTION

The extent and complexity of natural processes and human activities manifested in the environmental quality of the ecosystem require adherence to the principles of international best practices. The development and implementation of universal operating procedures include the best applicable technologies for sampling, laboratory analyses and providing evidence, as well as, operational accountability, transparency in information, data exchange, and interpreting sample results.

The Environment Ministers from each province/territory and the federal government through the Canadian Council of Ministers of the Environment provide a perspective on environmental issues to bring national strategies to deal with common issues. On February 17, 2009 The Council of Ministers endorsed the Canada-Wide Strategy for the Management of Municipal Wastewater Effluent (MWWE). The Strategy sets out a harmonized framework to manage discharges from existing wastewater facilities and offers municipalities the Model Sewer Use Bylaw, recommending its adoption and implementation, as a means of helping meet the quality of effluent discharges. In order to achieve the aims of the MWWE Strategy, the approach, components and methodologies of monitoring and assessment must be clearly elaborated and scrupulously followed by those engaged at all levels of the process for providing reliable and coherent environmental data.

The implementation of the Sewer Use Bylaw inevitably implies the possibility of enforcement and prosecution in the case of violation. Successful prosecution depends on recording and maintaining a reliable chain of evidence from sample taking through the verification of an exceedance of the bylaw, confirmed through accredited laboratory analyses. Analytical results of samples taken referred to as “legal”, “court” or “evidentiary” samples demand establishment and adherence to a protocol that will demonstrate the particular results that were found are for the sample taken.

This Guideline has been created to facilitate the achievement of the purposes set out in the MWWE Strategy and serve as the basic reference for the work of the personnel involved in any stage of the legal sampling. From the aspect of the existing policy and technical requirements with respect to the environmental system protection, the document is designed to provide essential documentation needed to fulfil the legal sampling requirements. It presents standards and uniformity in undertaking legal sampling procedures for all those involved from sample taking, through handling, transport and analysis, to storage and ultimate disposal.

The Guideline is based on national and international established standards, procedures and protocols, legislative and regulatory requirements, scientific literature and the experience of Environment Canada, provincial/territorial institutions and municipal laboratory staff, as well as international environmental agencies and other associations.

The Guideline alone cannot guarantee high quality results. It is not created to be a complete document as it is written based on the analytical sampling and testing requirements associated with the issues of current interest which may from time to time change. Therefore, it is to be regularly reviewed and updated. Common sense steps to avoid contamination, ongoing programs of staff training and proactive analytical method improvement procedures are also essential.

## 1.1 OBJECTIVES

The purpose of the guideline is to define standard operating procedures, methods and considerations that are to be used and observed when collecting and handling wastewater (or other) legal samples for field screening, laboratory analysis and evaluation for potential enforcement and legal actions in court. The guideline is intended to supplement the various sampling reference methods and clearly identify the points and requirements specific to the legal sampling procedure. It can assist in ensuring that the legal sampling criteria are established on a formal basis and accepted by all involved.

The development of the Guideline primarily is led by the ISO/IEC 17025 Standard management requirements. The guideline promotes a set of appropriate forms of documentation to support the legal samples as conclusive evidence in the case of prosecution. Furthermore, it assists municipal laboratories in developing their own continuity documentation during such activities as field sampling, sample receiving, sample storage, sample analysis, sample disposal and chain of custody procedures.

The objectives also involve:

- Supporting the laboratories accredited to work with legal samples to produce analytical data of the required quality and improve quality assurance management,
- Addressing the existing data and legal sampling management protocols to develop a national standard approach,
- Ensuring and encouraging explicit acceptance and utilization of operating procedures by the personnel involved,
- Consolidating the information on legal sampling to reinforce the positive effects and aspects of its use for law enforcement and legal action in court,
- Encouraging and assisting municipalities to set a framework and develop programs on their benefits to manage wastewater discharges as a means of helping meet the quality of effluent discharges.

## 1.2 SCOPE

The Guideline describes both general and specific methods to be used by those involved when collecting and handling samples from industrial, commercial and institutional

discharge points into a municipal sewer. It also demonstrates the manner of sample transportation, analysis, storage and disposal.

The priority area of this Guideline is development of a legal sampling protocol and therefore is to be viewed as a general guide to legal sampling. Additional sources are to be referenced for detail protocols, equipment options and insights regarding the usefulness of a particular analytical method.

Examples of the proposed documentary forms can be found in Appendices 1, 2, 3, 4, 5, 6, and 7 respectively, while their contents are discussed under each section of the guideline.

## 2. LEGAL, COURT OR EVIDENTARY SAMPLE

Legal samples are samples collected and handled for court purposes and are intended to be used as evidence for a possible prosecution concerning the sample origin, type and constituents. In comparison to the non-legal samples, the legal sample should demonstrate more stringent proof of custody from the time of collection to the time of analysis and disposal. The sample collection procedure itself may be equivalent to the routine sampling, but the sample handling and documentation requirements are more stringent. The legal samples must also confirm the samples could not have been tampered with at any stage and therefore they require special care due to the influence they may have on a court case.

Legal samples are needed when there is evidence an individual or company has not complied with regulatory requirements and there is a potential for laying charges. Legal sampling is conducted under the following circumstances:

- Any known or suspected violation,
- Spills or environmental accidents,
- Previous knowledge about compliance history does not exist or it is unknown.

From the standing point of objectivity, continuity of evidence and quality of the results, the collection, handling, transport, analysis, storage and disposal of the legal samples must be defensible.

## 3. CHAIN OF CUSTODY

Samples can be collected by the field sampler, wastewater laboratory technician or a bylaw officer, labeled and sent to the laboratory for analysis. When legal litigation is indicated, an accurate written record to trace possession and handling of samples from collection through reporting is mandatory.

Chain of custody is demonstrated when there is documented evidence confirming that a sample has not been exchanged with another sample, contaminated, or tampered with in any manner. The chain of custody shows how evidence was collected, who collected it, the location where it was collected and who has had custody of the evidence throughout the various steps that followed. The forms confirming the sample has been secure should be signed by each person that had true possession of the sample.

A sample is considered to be in true possession or custody when it is:

- In actual physical possession of one person,
- In view of one person after being in their physical possession,
- In physical possession and then locked up so that no one can tamper with it,
- Kept in a secured area, restricted to authorized personnel only.

The chain of custody procedure is intended to ensure the sample is kept secure at all times and stands up to the documentation requirements associated with a legal challenge. It is a written record documenting continuity by tracing the transfer, security and possession of the sample during all stages from collection, transportation, storage, laboratory analysis through to its introduction into evidence and ultimately to disposal. Samples are not admissible as evidence before the court in a case where their continuity has been interrupted for any reason.

The chain of custody documentation should include the following forms:

- Reagents and Supplies Sheet,
- Field Data Sampling Sheet,
- Sample Shipping/Receiving Sheet,
- Sample Receipt and Record Log Sheet,
- Sample Control Record Sheet,
- Analytical Data Sheet,
- Sample Data Archiving Sheet,

Three elements must be taken into consideration to achieve a full control of sample custody:

- Control at site is achieved by a member of the sampling team responsible for correct labelling and storage of samples,
- Transport is controlled by completing a form with shipping notes including brief descriptions of the samples,
- Within laboratories custody of samples is controlled by establishing detailed procedures, as many samples must be tested and reported correctly.

### 3.1. CHAIN OF CUSTODY CONTINUITY

Legal sample continuity is ensured by taking the following steps:

- Sealing – proper techniques, such as custody seals; masking tape should be used to seal the sample,

- Permanent Marking – sample should be labelled by permanent water proof uniquely numbered label and/or a diamond-tipped scribe, waterproof marker, or other means of permanent identifications with sufficient information to enable sample identification,
- Information Transfer – all data from the label should be transferred into the Field Sampling Data Sheet including the reference to unique identifier,
- Security and Safety – the sample must be locked in a secure container, refrigerator, etc. or kept in the possession or view of the person dealing with the sample at all times until it can be secured. The number of people handling the sample must be limited ensuring only one person at a time has access to the sample,
- Chain of Custody Forms – the forms must be completed and included with the sample.
- Packing – the sample must be placed in an appropriate and secure shipping container.
- Delivery – during transportation to the laboratory and from the laboratory to the sender/submitter or court, the sample must be sent by appropriate means and ensured it arrives within the required holding period. Samples should be delivered to the laboratory as soon as possible to minimize sample degradation,
- Analysis – laboratory personnel are responsible for maintaining the chain of custody. The sample must be traced from the moment it was received to the time the final results are reported,
- Court Use – to introduce sample results as evidence in court the chain of custody procedure must be clear and complete, demonstrating the sample has not been exchanged, contaminated, or tampered with at any stage,
- Note taking – detailed notes of sample collection methods, container markings, packaging, shipping and details for sample analysis, storage and disposal must be retained, and made available as required.

## 4. SAMPLING PROCEDURE

The accuracy of the final result of the legal sample analytical procedure begins with sample collection. The primary aim of the sampling is to produce a set of samples representing the source of interest and suitable for analysis to identify the compound(s) in question in a case of legal investigation.

Going on-site and collecting a sample without first planning how to do so, may result in a meaningless, contaminated, or unrepresentative sample. If the sample has been improperly collected, stored, or preserved, the results can be incorrect no matter how precise the analyses are or how thorough the quality assurance and quality control methods are.

## 4.1 SAMPLING PLAN

To meet the objectives of legal sampling and ensure success of the whole procedure thorough planning is crucial. The planning process of the legal sampling procedure begins with a preparation of a detailed sampling plan.

At the very beginning, before the start of a sampling activity, it is important to perform a thorough research of existing information. All involved in the process should get familiarized with the case in a whole and understand what should be documented before, during and after the sampling to fulfil all requirements and provide sufficient, reliable and defensible data.

Since the legal sample is collected for enforcement purposes and/or to use in court, the following questions and answers should be addressed during preparation of the sampling plan:

1. What specific tests need to be run on the sample?
2. Where will the samples be collected?
3. How is sample going to be collected and what type of sample is to be obtained – is it to be a grab sample or a composite sample?
4. When does the sample need to be collected and analyzed?

The legal sampling documentation includes a record of the planning process, field work and laboratory activities. Keeping detailed notes provides much needed information and aids getting successful results when such information is used in a court case and/or other enforcement activities. For example, the raw notes, documents, notebooks and report can be used in preparation of the court brief and may be taken as evidence into legal proceedings.

The sampling plan should explain the reason of choosing a particular approach and leaves no doubt on the final results and evidence provided. A documented systematic sampling approach can be used to recall important details while testifying in court.

A comprehensive legal sampling plan should include:

- Categories:
    - Introduction and objectives,
    - Scope,
    - Sampling matrix and sampling site,
    - Method of sampling,
    - Administrative arrangements – health, safety and security provisions,
    - Preliminary site inspection,
    - Sampling equipment,
    - Containers, preservatives, holding times and shipping and additional information.

- Further details:
    - A summary of background information of the site,
    - The issue and purpose of sampling,
    - The breadth of coverage of a sampling plan,
    - Investigation methods required to characterize the site – sample types, sampling locations and field quality control,
    - Sampling protocols,
    - Personnel requirements,
    - List of standard and non-standard equipment, list of containers needed,
    - Method used to control contaminated materials including documentation procedures, solutions to be used and storage or disposal obligations,
    - Using a certified laboratory, ensuring the understanding of sample holding times, special training required, the way how to be contacted with the information.

- References
    - Information sources,
    - Sampling media background,
    - Sampling protocols and procedures,
    - Sampling tests and equipment protocols,
    - Laboratory certifications.

## 4.2. SAFETY CONSIDERATIONS

Sampling related safety consideration should be brought into the planning process from the beginning, especially for the following:

- Personal protective equipment,
- Fall protection equipment,
- Specialized air monitoring equipment for hazardous environments, and
- Specialized equipment for handling potentially harmful substances.

The field sampler should be thoroughly familiarized with existing safety guidelines and follow the sampling procedure, guidelines and practices for any analyte of particular interest. The sampler must always be alert to the possibility of danger, especially in dealing with unknown sites, situations or possible contaminants.

A number of reagents (e.g. concentrated acid and alkalis, potassium dichromate and other chemicals) should never be handled without wearing protective gloves and eyewear since they may be corrosive or strong oxidants. Water should be never added to strong acid – it may boil up. Concentrated chemical pellets, e.g., sodium hydroxide, may generate heat if dissolved, whereas concentrated nitric acid, fumes freely. Consequently, only the amount of

reagents needed to complete the field work should be taken and, while working with them, the vessels must be handled with complete care.

## 4.3 PRELIMINARY ASSESSMENT

If the circumstances of the sampling event allow, a preliminary site visit should be conducted to fully understand the legal sampling requirements. This examination assists in obtaining background information on the location and facility, as well as to identify the required equipment, safety and hazard considerations, and what will the technical approach to the site sampling consist of. For this purpose, prior to sampling, the applicable legislation, regulations, interim orders, guidelines, codes of practices and other legal or regulatory documents should be reviewed.

## 4.4 PREPARATION ACTIVITIES

The sampling preparation activities should include, but are not limited to the following:

- Verifying mean(s) of transportation e.g., checking and traffic safety plan completion, including completion of transportation vehicle inspection checklist, if applicable,
- Creation of sampling plan and chain of custody forms,
- Preparing sample containers for the parameters of interest. The containers should be inspected before taking and than placed in the lockbox,
- Obtaining additional required equipment (autosamplers along with an autosampler numbered evidentiary lock and charged batteries, appropriate tubing, strainers and any external devices such as flow loggers, flow meters, or liquid level actuators),
- Filling the required number of empty containers with de-ionized water in case travel blanks are to be collected and including them with the other sampling equipment,
- Providing evidentiary seals and a numbered evidentiary lockbox with matching lock and key,
- Prepare all the required equipment including personal protective equipment.

### 4.4.1 REAGENTS AND OTHER SUPPLIES

During preparations for sampling, the reagents and field supplies that will be used should be prepared. Each laboratory is responsible for ensuring that all reagents and equipment are suitably clean and free from contaminants and interfering substances. Reagents can be used as calibration solutions to calibrate sampling equipment or may be used as chemical preservatives and become part of the sample itself. Such reagents

should be of high purity with an expiration date, beyond which they should not be used. All exchanges of reagents and supplies must be documented on the Reagents and Supplies Sheet.

In a legal proceeding, both the methods of preparation and the competence of the preparer may be questioned. It is essential to keep records, including the preparation date, name of the preparer, and location of the reagents and supplies from preparation through use.

Field supplies may include:

- Labelled sample containers,
- Labelled shipping containers,
- Buckets for grab sampling,
- Automatic samplers,
- Portable meters (thermometer, pH meter, spectrophotometer, turbidity meter, colorimeter, dissolved oxygen meter, magnetic flow meter),
- Filtration or other preservation equipment used in sample collection,
- Ice packs and coolers,
- Personal gear (for all possible weather conditions, gloves, goggles, protective footwear),
- Camera or video equipment, GPS,
- First aid kit.

An example of the Reagents and Supplies Sheet is found in **Appendix 1**.

## 4.5 SITE RECORDINGS

Proper documentation of all site activities is an integral part of legal sampling and field investigation. In a written form, the field sampler keeps accurate notes as an inclusive documentation of sampling operations including field data, observations, field equipment, sample handling and chain of custody forms. All notes should be made at the time or as soon as possible thereafter. Whenever possible, the notes should be written in waterproof ink and kept by one person.

The chain of custody Field Sampling Data Sheet (also Field Logbook or Sample Journal) in which all field measurements should be entered directly while in the field is mandatory in the legal sampling procedure. The field sampler must note all unusual occurrences (e.g. unusual colour or odour, surface films) and any deviations from standard protocols (e.g. sample is taken from a different location due to safety or access considerations or sampling procedures differ from the set protocol). Preservation of the sample consistent with the type of analysis that will be performed as well as additional samples, if collected, and for what purposes should also be recorded.

The Field Sampling Data Sheet is the basis for later reports and therefore should contain objective factual information, free of personal opinion and feelings or other terminology which might prove inappropriate. Data that has been entered incorrectly should be corrected by drawing the line through the incorrect entry and initialling or signing and dating the lined-through entry. The incorrect material should not be erased, made illegible or obscured so that it cannot be read.

The Field Sampling Data Sheet for each sampling program should include common site and sample information:

**A. Site Information**

The site information could include, but is not limited to the following:

- Sample location description – site name and global positioning system (GPS) coordinates including the type and number of sample point access (e.g. sewer access, manhole), photo/video recordings
- Name of the sample collector and names and affiliation of the persons present on the spot (e.g. business card) including all those attending the site and witnessing the sample collection,
- Name and address of the facility, reason for visit and any other characteristics of the sampling location,
- Date and time of sampling including the times of arrival and departure, details for any stops made between departure and arrival at the sample location,
- General description of the area along with the land use and/or production practices upstream and down-stream of the sampling location,
- Weather conditions on the day before and the day of sampling.

**B. Sample Information**

The sample information could include but is not limited to the following:

- Sample identification number,
- Numbers and natures of quality control samples and time of collection,
- Time and date and other details of collection (e.g. completion of evidentiary labels, the time when labels and seals are affixed to the sample containers, seal number),
- Method of sample collection and any factors that may affect sample quality,
- Equipment used and work performed on site (equipment blanks, grab sample collection, auto sampler, duplicate sample collection),
- Discharge characteristics along with all pertinent observation (e.g. discharge flow rate, colour, clarity, any noticeable odours),
- Time when samples were sent/transported to the laboratory, including the times and details for any stops made between departure and arrival, time of arrival at the laboratory and details for relinquishment of the cooler and samples.

An example of the Field Sampling Data Sheet is found in **Appendix 2**.

### 4.5.1 PHOTOGRAPHIC DOCUMENTATION

Legal sampling procedure should be supported by photographic documentation as the easiest, most accurate and convenient way to record and elaborate the observations and provide accurate identification of sampling points. Photographs should be taken showing the labels along with clearly visible sample numbers and linking the sample to the source. Documenting the sampling locations by photographs should include two or more references that will help to identify the point at a letter date. A series of photographs should be taken at each sampling location looking upstream, downstream, to the left, and to the right at the same time as the sampling is done. By using specialized software digital images can be sent via internet to the central database and documented in the Field Sampling Data Sheet notifying the following information:

- Date and time of the photograph,
- Name of the photographer,
- Name and coordinates of the site,
- Compass directions or GPS location and description of the subject taken,
- Photograph number and/or film roll number.

### 4.5.2 VIDEO RECORDINGS

Video coverage can be used to describe the sampling location and prove that samples were taken properly. It gives an idea of the sampling circumstances to all involved in the process that have not been present on-site. Complete and accurate perception is ensured when a verbal documentary of the scene is included on the video camera's sound track. While choosing the camera angle, maximum care should be taken to get valuable information. The video recording memory medium within a device (DVD, USB flash drive, memory card) should be labelled with the name of the person who has made it, location, date and time.

Video recordings and photography are included to assist the process of documentation, but they can not be used to the exclusion of written documentation. Photographs and video recordings have to be saved and kept for future reference (e.g. for planning of sampling, future sampling trips, investigation, or for use in court as means of consultations).

## 4.6 SAMPLING SITE SELECTION

Selection of a representative sampling site is one of the most important steps in investigation, monitoring, or surveillance planning. A sampling site must be selected at the

point which is sufficiently characteristic to reflect the objectives and meet the goals of the legal sampling program. The reasons for choosing a particular sampling location should always be documented.

The objective of legal sampling in most occasions is to find unknown sources of known contamination. It has to match the strategy for choosing the sampling locations. Along with the sampling objectives and the analyte of interest, the site selecting process depends on the following factors and concerns:

- Sample quantity - what number and volume of the sample is required?
- Legislation - are regulatory requirements a factor?
- Method of sampling - what is the most appropriate method of sampling?
- Sample characteristics – for example do the substances mix with water, float on it, or sink?
- Sample type - should the sampler collect a grab or composite sample?
- Sampling equipment - is the sample to be collected by an automatic sampler or manually?
- Accessibility – is access to the site a problem?
- Location – can the location be found easily?
- Re-sampling – will it be easy to repeat the sampling if required?

The field sampler has the responsibility to locate all sampling locations accurately. Satisfactory knowledge about on-site conditions prior the sampling is crucial. Physical site inspection and research of available records (e.g. file information or previous reports or consultation with others who may know the site) should be done before sampling.

Sampling location must be identified in a simple and unambiguous way. A detailed sewer site map, when the sample is to be taken within the industrial premises, or a detailed area map, when sample is taken outside industrial premises, can be helpful in selection and description of the sampling location. A map describing the sample location should accompany the Field Sampling Data Sheet.

The GPS is most frequently used to determine the latitude and longitude of each location and confirm the exact sample collection location during subsequent sampling events, particularly when a return to the same location is necessary. Nowadays, the most GPS systems are accurate to within 3-5 meters and offer multiple station locations, data recording, way marking and navigation capabilities. It is important to note that the locations with a restricted view from the sky have reduced accuracies. GPS measurements should be recorded on the Field Sampling Data Sheet.

## 4.7 SAMPLING CONTAINERS

An equipment check list including sampling and safety equipment, sample containers, reagents, shipping materials and other supplies should be prepared during the preparation for the sampling event. When taken in the field, the field sampler takes possession of the materials and accompanied documentation. When in the laboratory, the lab-sample custodian should store the materials prepared by the lab technician in a locked storage with limited-access until they are needed.

Most wastewater samples can be collected in either plastic or glass containers. Sample containers that will not affect or react with the sample must be chosen. The sampler should be aware of the composition of sampling containers when analyzing for trace contaminants. Containers may decompose and release contaminants or sorb materials onto the walls of the bottle. Cracked, chipped and etched glass bottles, as well as crazed plastic bottles and bottles which are leaking should be discarded.

When choosing sampling containers, the field sampler should consider the following:

- Glass can be used for sampling hazardous materials,
- Amber glass is used to prevent photo degradation of the sample. If not available, the sample should be protected from light by other appropriate means,
- Plastic or Teflon can be used for alkali solutions, hydrofluoric acid and mercury samples,
- Teflon or glass can be used for organic samples and media,
- High-density polyethylene should be used for metals, except mercury, most inorganic samples and media.

The field sampler should be also aware of the type of container lid used. Lids with metal foil liners should not be used when collecting metal samples. Inappropriate lids or lid liners can cause serious problems; for example, lid liners made of paper or cardboard are potential source of contamination. Lids should be of the screw type to form a leak-proof seal and should be lined with Teflon or Teflon-coated material. In certain situations, polyethylene liners may be acceptable. Heat-treated aluminium foil can be used when sampling petroleum products to cover the mouth of a container before the lid is screwed on to avoid contamination from a plastic lid.

## 4.8 SAMPLING APROACHES

Selection of the sampling method for representative sample collection must account for the unique characteristics of a specific sampling location, including the physical features of the site and the discharge characteristics (e.g. floating debris, turbulence, flow velocity and depth).

There are three sampling approaches: random, systematic, and judgmental. The random and systematic approaches are used in routine sampling, whereas the judgemental sampling is often the method of choice for regulatory and emergency response sampling.

The judgmental approach used in combination with one of other two may be useful in most occasions. The validity of judgemental sampling depends on the accuracy of the knowledge of distribution of the parameter(s) of interest, prior site history and visual assessment of technical judgement. This knowledge is also critical in determining locations that will provide the most representative samples.

## 4.9 SAMPLE COLLECTION

When collecting wastewater samples, minimum safety precautions such as gloves and goggles should be required. Selecting sampling sites that present a hazard such as leaning over railings, slippery decks, etc. should be avoided for the safety reason. It is very important that maximum attention to be paid for the possible hazardous and infectious materials.

Legal samples can be collected as grab samples or composite samples. Composite samples can be collected manually or by using an auto sampler. When sampling involves more than one staff member on-site at the sampling location, only one person should collect the sample(s). The other person(s) may only observe the event without any active participation.

The following steps are recommended during the sample collection:

- Check in with security or company staff, if required; this should be done upon arrival to gain an access to the sampling location,
- Survey for hazards (traffic, equipment, other) should be done on the sampling location,
- Traffic safety set-up equipment and traffic safety plan should be implemented, if required. Traffic cones for example can be used to make people more aware of the sampling staff presence particularly when the sampling location is easily accessible by the public,
- Precise sampling location should be selected,
- Gas detector and manhole lifter should be used to remove a manhole cover and gain access to the sampling point, if applicable,
- The evidentiary lockbox should remain within the unobstructed view of the person taking samples at all times unless it is securely locked in the transportation vehicle,
- Sampling equipment should be gathered, including:
    - Stainless steel grab containers or sterilized steel grab container if collecting bacteria samples,
    - Sampling containers (amber glass jugs) with lab grade water if equipment blanks are to be collected,

- Sample container - bottle carrier and required bottles, pre-labelled with numbers, including sample containers with first or second part grab samples from previous site visits, if applicable,
- Paper towels, sampling pole or rope, when collecting grab samples,
- Spotlight,
- Auto sampler with appropriate length of sample tubing, strainer, battery and any required external connections as well as steel suspension ring for auto sampler, only if an auto sampler is to be installed.

### 4.9.1 TIME-PROPORTIONAL SAMPLING

Samples of equal volume collected by hand grab method must be taken sequentially at regular time intervals. When taking such samples certain considerations for representative sampling collection should be acknowledged, such as:

- The grab container should be made of material that has no possibility of leaching a contaminant(s) of interest or to chemically react with the sample,
- The grab container should be attached to the sampling pole or rope and then lowered into the sampling point and into the discharge stream with the opening facing the flow, where possible,
- The grab container should be filled by the discharge flow and after being retrieved, it should be emptied back into the sample point to rinse the container,
- The grab container should be lowered into the sampling point and discharge stream again, filled by the discharge flow, retrieved and emptied back into the sample point to rinse the container a second time,
- A sample should be taken after the grab container is lowered into the sampling point and discharge stream for third time, filled by the discharge flow and retrieved. The sample is kept for transfer to sample containers/bottles,
- The cap(s) from the first bottle or set of bottles to be filled should be removed by setting the outside surface of the cap(s) down on a clean surface,
- Sufficient volume of the sample from the grab container should be poured into the bottle(s) until they are filled. If necessary, the procedure should be repeated until all the required bottles are filled.

In addition to the sample collection procedure, immediately after taking hand grab sample(s) a separate evidentiary label should be completed and affixed to each sample container. The corresponding legal custody seal should also be placed across each container lid with each end affixed to the sides of the container. At the

end, the samples should be placed in the cooler - lockbox and locked inside. The key of the lockbox should be kept in possession of the field sampler.

### 4.9.2 AUTOSAMPLING

Anutosampler is used when a representative sample over a fixed time period is required. Based on the standard installation procedure, an autosampler is installed when a new sample and pump tubing are installed on the autosampler prior to use. For further use manufacturer's operating manuals and specifications for the operating details should be consulted. Frozen gel packs should then be placed in the autosampler's base along with either glass or polyethylene plastic composite container. Prior to lowering the autosampler into the sampling point, the autosampler lock should be attached to the appropriate spot on the carrying handle and the key kept by the field sampler.

During the automated composite sampling that may take from few hours to several days the sampling probe can be exposed to the environment and possible tampering. In order to prevent such occurrences, the sampling location will need to have some degree of security and the sampling device should be checked by the field sampler at random intervals.

An autosampler used for legal sampling should be removed immediately after the sample containers are filled and an evidentiary label is completed and affixed to each container. The corresponding legal custody seal should be placed across each container lid end affixed to the sides of the container. The samples should be then placed in the cooler - lockbox, which should be locked and the key kept in the field sampler's possession.

### 4.9.3 WATER SUPPLY SYSTEM SAMPLING

A water sample from a distribution system entering the premises and/or water that is discharged from a cooling or heating system could be taken to verify the possible presence of a violating parameter. This is recommended when the contaminant in question is also present in the water supply system and could influence the total concentration of the violating parameter in wastewater sample.

### 4.9.4 SAMPLING COMPLETION

After the sampling procedure has been completed, the sampling equipment and cooler - lockbox with collected samples are returned to the transportation vehicle. Dirty grab containers, if any, should be placed into "dirty" plastic storage containers. The traffic safety set-up, if applicable, need to be dissembled and all safety set-up equipment and traffic cones returned to the transportation vehicle. Finally, protective gloves should be removed and hands cleaned with a hand sanitizer. Unless it is securely locked in the

transportation vehicle at all times, the cooler - lockbox along with the samples should remain within unobstructed view of the field sampler or person authorized.

Collected samples and its chain of custody documentation are in the custody of the field sampler until relinquished to the laboratory personnel.

## 5. SAMPLE IDENTIFICATION - SEALING AND LABELING

Once the legal sample is collected, the containers should immediately be properly sealed and labelled. A legal custody seal is applied over the top of the lid and down the side of the container showing the identification number of sample and the field sampler's signature. To prevent container mix-up each sample container must also be labelled with waterproof, permanent, adhesive, polyester labels or equivalent.

Different labelling systems can be used, such as:

- Pre-printed and two-part labels with corresponding unique numbers. One part of the label is attached on the container and the other one is kept for the sampling documentation,
- Another way of labelling is to etch the container with a glass etcher. The etchings should be marked directly into glass, forming a permanent record,
- Evidentiary labels that match container identification numbers on three separate stickers can also be used. In such a case, one of the stickers is used to seal the lid of the sample container (the lid cannot be removed without damaging the seal), one is put on the sample container and the last is placed in the Field Sampling Data Sheet along with the sampler's notes for the sampling event.

Proper labelling of samples is also important for quality assurance purposes. Information on the labels provides uniformity of sample records, assists the sampler and helps ensure that vital information is not omitted. The label could contain, but is not limited to the following information:

- Container identification number,
- Sampling date and time,
- Sampling location
- Field sampler's name
- Name of the witness(es).

In a case the sample label is lost or was never prepared, a written statement should be made detailing the sample collection and transportation from the field to the laboratory, description of the known chain of custody data and references to any sample associated entries.

## 6. SAMPLE PRESERVATION, STORAGE AND HOLDING TIME

Following, both the proper holding times and required preservation will help to retain the integrity of the sample so that accurate, legally defensible results can be obtained.

### 6.1 PRESERVATION AND STORAGE

The purpose of sample preservation is to minimize any degradation until the samples can be analyzed. Complete preservation of samples is practically impossible because complete stability for every constituent can never be achieved. Sample that cannot be delivered to the laboratory within 2 hours must be preserved to minimize changes in samples that begin immediately after collection. It is the field sampler's responsibility to preserve and store the samples until delivered to the laboratory.

There are no absolute methods of preservation and no single method is entirely satisfactory. The preservation methods are generally intended to retard biological action, retard hydrolysis of chemical compounds and complexes and reduce volatility of organic molecules. Between sampling and analysis samples should preferably be kept as cool as possible. Using of dry ice should be avoided because it will freeze samples, may cause glass containers to break and affect a pH change in samples.

Every parameter has a specific preservation technique. Chemical preservation should be added to the sample where required to fix the analyte of interest from loss or breakdown. The samples must be analyzed as quickly as possible on arrival at the laboratory. If immediate analysis is not possible samples should be refrigerated at 4°C. It is the responsibility of the laboratory to maintain the sample preservation and assure timely analysis.

Some analyses are more affected by sample storage than others. Certain cations are subject to loss by adsorption on, or ion exchange with the walls of sampling containers. Sample collection using the proper containers, preservation, and analyzing method within the required holding times lead to the accuracy of sample results. For samples that are not being analyzed immediately after collection, analytical results cannot be accepted for reporting purposes if samples have not been correctly preserved and stored.

### 6.2 HOLDING TIME

Holding time is the maximum time allowed between collection and the moment when sample preparation or analysis on that sample must begin. Turnaround time in addition is the length of time it takes from when a sample is received by the laboratory to the time when a result of analysis is issued.

Correct handling of sample and prompt delivery is essential in order to obtain the best quality of analytical results. The sample should be delivered to the laboratory immediately after collection and analysis should start as soon as possible to ensure the results are representative. Samples begin to change as soon as they are removed from their environment. In a case when the holding times are exceeded the samples should be flagged in the laboratory analytical report.

The samples that cannot be preserved should be analyzed either in-situ or on site immediately after collection. For example, samples collected for parameters such as pH, dissolved oxygen, chlorine residual and temperature have no acceptable holding times and must be analyzed immediately. For composite samples, it is common practice to use the time at the end of composite collection as the sample collection time.

## 7. SAMPLE TRANSPORTATION

Shipment of samples is time critical to ensure their integrity. The field sampler is responsible for preserving and storing the samples until they are delivered to the laboratory. After collection all samples should be handled as little as possible. They should be put on ice for transport to laboratory but never frozen. Each collected sample stored in the cooler should have its own Field Sampling Data Sheet that must accompany the sample at all times. The Field Sampling Data Sheet should be placed in a waterproof bag, sealed and taped under the lid of the cooler along with the samples to which it applies.

Samples can be hand delivered, delivered by mail, or private carrier. Samples that are expected to be used for court purposes are preferably transported by the sampling personnel. They remain in field sampler's custody and must be secured until they are relinquished.

Samples transported by the postal service must be sent by registered mail with return receipt. When sent by private carriers, samples require a description of the items on the bill of lading. Either way the sample shipping bags or containers should be marked "deliver to addressee only."

The packages should be sealed with custody seal tape so the recipient can tell if the package has been tampered with. This seal should not be broken until the samples arrive at the laboratory and are checked in by laboratory staff. The seal label should include:

- Shipping and receiving facility name,
- Sampler's name,
- Date the container is sealed for shipment, and
- It should read "Chain of Custody Sample – Authorization Required to Open".

A copy of the Sample Shipping/Receiving Sheet should always accompany the transported samples. The field sampler should keep a copy of this form and any other shipping documents

for the record and store them in a secure place. The original form should be sent with the sample and signed by the sender and lab-sample custodian upon receipt. If the package is sent by private carrier, the carrier needs to make sure the package tracking information is included on the form so that samples can be tracked if necessary.

An example of the Sample Shipping/Receiving Sheet is found in **Appendix 3**.

## 8. SAMPLE CONTAMINATION

The possibility of a sample contamination is always present. A contaminated sample is considered useless and contamination between containers, tools and equipment could become a big issue during legal proceedings. Samples that do not require the addition of preservative must be kept tightly capped and out of contact with anything that may cause contamination.

Samples may be contaminated by:

- Inappropriate containers or equipment,
- Dirty container caps,
- Containers that have not been properly cleaned,
- 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.

As preventive measures, samples should not be stored in containers whose origin is not known to the field sampler. The sample containers should be sealed tightly and stored in clean areas. Sampling containers and other sampling equipment should never be stored near solvents, gasoline, or other volatile substances that might cause contamination. Sampling equipment should remain in wrapping material until it is used in the field. It must be decontaminated according the appropriate protocol when transferred between sampling sites. Disposable gloves should be worn at all times when handling preservative, sample containers and sampling equipment.

Polyethylene and other kinds of plastic tubing can leach out phenolic compounds and phthalates, which interfere with some organic analyses. All lengths of polyethylene, surgical and polyvinyl chloride tubing should be as short as possible, thoroughly cleaned and replaced with Teflon tubing where feasible.

## 9. CLEANLINESS

Cleanliness is a high priority. To prevent contamination all sampling containers and equipment must be kept clean and in a good condition, fit to be used at all times.

Pre-cleaned sample containers can be purchased from commercial suppliers and used for sampling, only under condition that the laboratory is familiar with its cleaning protocol.

### 9.1 CLEANING OF FIELD EQUIPMENT

The equipment or its parts that come into contact with the sample should be cleaned to avoid cross-contamination. The cleaning should be done before going into the field whenever possible.

Prior to the trip all visible particulate matter and other residue should be removed by washing the equipment accessible surfaces with phosphate-free laboratory grade detergent and hot water. A steam or high-pressure water washer can be used if possible to remove dirt or residue from the sampling equipment. In any case, an approved laboratory cleaning methodology for analyte of interest must be followed.

While in the field, if washing is needed, the sampling equipment should be scrubbed with detergent to remove all visible particulate matter and other residue, rinsed several times with tap water and than few more times with de-ionized water to remove detergent.

### 9.2 CLEANING OF CONTAINERS

Sample containers must be properly cleaned prior to the sampling event. As a minimum, the sample containers should be washed with phosphate-free laboratory detergent and hot water. All surfaces should be scrubbed with a brush kept for this purpose only and then rinsed thoroughly several times with tap water to remove all traces of detergent, followed by several rinses with de-ionized water.

Sample containers should never be rinsed with the sample prior to collection. Pre-rinsing may allow some contaminants to stick to the sides of the container or settle to the bottom prior the final sample being collected. For example, pre-rinsing with wastewater sample may cause extra oil and grease to stick to the sides or extra settle-able solids to remain in the bottom of the bottle, causing a high bias to the sample answer.

Cleaning method is determined by the specific analyses to be performed on the sample:

- Metal Analyses - containers should be soaked in 10% - 20% nitric acid and rinsed few times with de-ionized water before taking them into the field,

- Organic Analyses - cross-contamination with organic solvents or substances can be a serious problem, therefore, the treatment of containers and equipment is frequently recommended,
- Bacterial and Microbiological Analyses – bottles and equipment for microbiological sampling and for any other samples requiring sterile conditions, must be sterilized before being used.

## 10. LABORATORY SAMPLE SUBMISSION

Once the sample is received, the laboratory personnel are responsible for the care and custody of the sample. The samples must be secured in the laboratory at all times. As they await analysis, the samples should be put in locked and secured storage and stored in a way that satisfies requirements for analyte stability, safety and ease of retrieval.

Few people as necessary should handle the sample. The laboratory usually designates one person (lab-sample custodian) to be responsible for the custody of all samples. This person should be able to testify that the sample was secured in the laboratory at all times from the moment it was received until the time it was disposed.

The lab-sample custodian that receives incoming samples verifies the received package and its content by checking the following:

- The integrity and condition of all sample cooler-lockboxes,
- The integrity and condition of all sample containers,
- Checks for leakage, cracked or broken closures or containers, evidence of grossly contaminated container exteriors or shipping cooler-lockbox interiors and obvious odours,
- Verifies the receipt of complete Field Sampling Data Sheet for each container,
- Verifies that each sample container identification number on the Field Sampling Data Sheet and Shipping/Receiving Sheet corresponds to sample container identification number on the container label,
- Verifies that each sample container seal number on the Field Sampling Data Sheet and Shipping/Receiving Form corresponds to sample container seal number on the container itself,
- Verifies proper field preservation of each sample has been done. There must be clear note of any chemical preservation of the sample on the Field-Sampling Data Sheet done in accordance with approved preservation protocols,
- For samples that require thermal preservation, lab-sample custodian will verify proper storage temperature by determining that sample containers are in adequate contact with wet ice in the shipping chest and by documenting sample temperatures.

The acceptance of the package is confirmed by lab-sample custodian signing the Shipping/Receiving Sheet and the Field Sampling Data Sheet for each container.

All information from the Reagents and Supplies Sheet, Field Sampling Data Sheets and Shipping/Receiving Sheet should be entered into laboratory's master log. The lab-sample custodian assigns to each sample a laboratory unique identification number and records it in the Sample Receipt and Record Log Sheet. The unique identification numbers are used to track the sample through the laboratory and should also be written on the sample container labels. In addition, bar coding technology may be used to help reduce transcription errors.

All transfers of samples within the laboratory and all procedures performed on the sample should be documented. The lab-sample custodian should maintain the chain of custody procedures and indicate on the Sample Control Record Sheet who removed the sample from the storage area and when it was removed and/or destroyed. After analysis, the sample should be returned to its original location in the storage. A label should be placed on a container signifying that it has been analyzed.

Examples of the Sample Receipt and Record Log Sheet and the Sample Control Record Sheet are found in **Appendices 4 and 5**.

## 11. SAMPLE ANAYSIS

Laboratories dealing with legal sample analysis have to have appropriate accreditation to carry out specific tests. The recognition of the laboratory competence requires ongoing demonstration of performance through proficiency testing and laboratory audits to maintain capabilities.

According to the ISO/IEC 17025 Standard requirements and Canadian Association for Laboratory Accreditation's (CALA) accredited procedures and methodologies, samples should be analyzed in a timely manner to ensure the integrity of the results. Laboratories should develop chain of custody procedures in order to verify the sample information is tracked from its reception through analysis, storage and disposal and the results obtained are related to the respective samples. Developing and maintaining of such procedures is extremely important for laboratories required to present evidence and testify in court.

The laboratory analyst is responsible for maintaining the chain of custody procedures during sample analysis. The laboratory analyst should record the sample into the Laboratory Sample Journal and assign the sample a unique laboratory analytical number. The Laboratory Sample Journal contains the information recorded in the Field Sampling Data Sheet as well as any other pertinent information.

The laboratory analyst should sign the Sample Control Record Sheet every time the sample is removed from and returned to the secured storage. A record should be made in the Laboratory Sample Journal whenever the container seal is broken and sample aliquots are taken for sample analysis. The sample container should be sealed with a new numbered seal each time it is

returned to the secured storage and the new seal number should be noted in the Laboratory Sample Journal. Sample aliquots taken should be kept locked in the refrigerator locked container or incubator awaiting the analysis. If the sample is given to another analyst or person, the exchange should be recorded and signed in the Laboratory Sample Journal. After analysis samples must be returned to the lab-sample custodian for secure storage.

Each analytical method used for sample analysis should have its own Analytical Data Sheet where obtained readings are recorded. The readings are copied to the Laboratory Sample Journal where final sampling results are calculated and together with other relevant information transcribed to the Report.

All written records must be documented in permanent ink. Any errors during the analytical process should be corrected by drawing a single line through the error. The correct value is then entered beside the incorrect entry with the initials and date of the individual making the correction. The laboratory analyst has the primary responsibility of assuring the data is correct and complete.

An example of the Analytical Data Sheet is found in **Appendix 6**.

## 12. SAMPLE DATA RECORDKEEPING AND ARCHIVING

The lab-sample custodian is responsible for maintaining the chain of custody procedures during sample data recordkeeping. This involves documenting the entire sampling and analysis procedures and placing all related forms, notes, calculations, test reports, and chain of custody records in limited-access locked storage. The Sample Data Archiving Sheet should identify where the data will be stored, what form it will be stored in, and how long it will be stored.

Records may be preserved in hardcopy or electronic format. Hard copies should be kept in folders and filed using the following guidelines:

- By year,
- By analytical parameter,
- By assigned lab number.

If stored electronically there should be written procedure where the copies will be stored, how many copies will be made, and how the database will be secured. For example, photographs and video recordings can be stored in special photo/video storage-vault or USB stick with security encryption/password protection. All records should be linked to internal records via laboratory identification number. If samples are expected to be part of a legal action all laboratory information must be easily accessed and defensible.

Documents and records must have designated minimum retention times, which must comply with applicable legislated requirements. Some storage times are legislated, while other storage

times may be set by the municipality. Minimum storage time will depend on internal policies and procedures developed pertaining to record retention.

An example of the Sample Data Archiving Sheet is found in **Appendix 7**.

## 13. SAMPLE DISPOSAL

After sample analyses were done and the test report has been issued, the authorized person should dispose of the sample in a safe and convenient manner according to the laboratory practice following appropriate federal, provincial and municipal health and safety requirements.

The sample will be disposed when it is certain that the information is no longer required, or the sample has deteriorated. Samples can be stored for reuse for the maximum holding time, after which it is considered to have deteriorated.

## 14. POST SAMPLING CLEANING

After sampling, used equipment should be properly cleaned and a label should be attached indicating when and how it was cleaned and who cleaned it. An equipment log should be kept on pertaining information.

Reusable collection containers should be washed and rinsed according to the proper cleaning procedure and then labelled with the information of the person who cleaned the container, how and when it was cleaned.

Dirty grab containers should be thoroughly cleaned, rinsed and returned to the “clean” storage container. Autosamplers should be cleaned and repaired following the equipment manual. The sampling vehicle and other sampling equipment should be cleaned and restocked as per future sampling requirements.

## 15. QUALITY ASSURANCE /QUALITY CONTROL PROGRAM

Quality Assurance (QA)/Quality Control (QC) is a program designed by the laboratory that specifies the methods and procedures required to produce measurement-based, technically valid, legally defensible and known quality information. The QA/QC activities are designed to evaluate precision and accuracy of the sample collection and analysis and to ensure that any problems that may occur are quickly identified and rectified.

The QA/QC Program has two components:

1. Quality Assurance (QA) - describes the overall measures that a laboratory uses to ensure the quality of its operations. It is designed to evaluate the precision and accuracy of the sample collection, laboratory analysis and potential sources of contamination encountered during sample collection and delivery to the laboratory.
2. Quality Control (QC) – is part of the overall QA. It consists of operational techniques and activities that are used to fulfil requirements for quality.

An effective QA/QC Program is essential for any laboratory seeking accreditation according to ISO/IEC 17025 Standard and CALA accreditation program.

## 15.1 QUALITY ASSURANCE MANUAL

The QA/QC Program should be defined in a written document called Quality Assurance (QA) Manual as a top tier of the document hierarchy. The manual should describe the approaches to achieve quality data and address any differences in handling between process control samples, compliance samples and samples headed to court. It should also include policy statements describing the intent and goal of the laboratory to conform to ISO/IEC 17025 Standard requirements. The quality policy statement should be written by senior management. The QA Manual which is to be reviewed and updated regularly should include the following topics:

- Laboratory organization and responsibility,
- Field sampling procedures,
- Laboratory sample handling procedures,
- Standard Operating Procedures for each analytical method used,
- Quality control procedures,
- Data reduction, validation, and verification,
- Preventative maintenance procedures,
- Laboratory audits,
- Corrective actions,
- Recordkeeping procedures,
- Proficiency testing.

For more detailed structure of documentation to be used in implementation of QA/QC Program, the ISO/IEC 17025 Standard and CALA accreditation program should be referenced.

### 15.1.1 LABORATORY ORGANIZATION AND RESPONSIBILITY

An organizational chart should be created showing the laboratory organization and lines of responsibility. For small systems the organizational chart is simpler. As the

system gets larger, the organizational chart will expand and the lines of communication become more complex.

Each position in the laboratory should have a well defined job description including training requirements to assure the laboratory personnel are updated on regulations and methodology. Each person in the organizational chart should know well their job duties and the job duties of all other personnel. A clear job description will minimize laboratory communication problems and provide an efficient movement of samples through the laboratory.

### 15.1.2 FIELD SAMPLING PROCEDURES

The field sampling procedures should be part of the Sampling Plan that describes required sample preservation, proper containers, correct sample container cleaning procedures, sample holding times from collection to analysis and sample shipping and storage conditions. The Plan should also provide copies of appropriate chain of custody forms.

### 15.1.3 LABORATORY SAMPLE HANDLING PROCEDURES

The QA Manual should address the way how samples are processed within the laboratory from the moment of receiving, through the storage, to analysis and disposal. It should describe:

- How unprocessed and processed samples are stored at the proper temperature, isolated from laboratory contaminants,
- Practices to ensure that holding times will not be exceeded,
- How personnel maintain integrity of samples, by tracking them from receipt by laboratory through analysis to disposal,
- Chain of custody forms.

### 15.1.4 STANDARD OPERATING PROCEDURES

Standard operating procedures (SOPs) describe the analytical procedures used by the laboratory in sufficient detail that an experienced analyst, unfamiliar with the specific test procedure should be able to perform the analysis. SOPs should list or describe the:

- Method title and referenced documents,
- Scope of method and application,
- Method summary,
- Materials and instruments needed,

- Operating instructions for instrumentation as a supplement to the manufacturer's operating manuals,
- Health and safety precautions, including personal protective equipment,
- Personnel qualifications,
- Calibration or standardization methods,
- Detailed analytical procedure,
- Calculations and data reduction,
- Chain of custody forms,
- Quality control parameters, frequency, corrective actions and trouble-shooting.

SOPs should be reviewed on an annual basis or whenever there is new equipment being introduced. All revisions must be dated and signed. Test methods that are new to the laboratory, methods that have been developed in-house, and changes and modifications to existing methods must be validated.

### 15.1.5 QUALITY CONTROL PROCEDURES

The laboratory QA Manual should identify procedures used by the laboratory to demonstrate the laboratory data is valid and defensible. It should define control samples used and their frequency of use.

Control samples include:

- Method blanks to monitor possible contamination,
- Duplicates and split samples to monitor precision,
- Certified reference samples to monitor accuracy,
- Internal reference samples to monitor accuracy,
- Blank and matrix spikes to monitor recoveries.

#### 15.1.5.1 TRAVEL BLANKS

A travel blank (also called trip blank or transport blank) is an aliquot of analyte-free reagent water used to determine if airborne factors present during the handling and transport of samples to the laboratory may have affected sample quality. Travel blanks are prepared in the laboratory by filling containers with de-ionized water and transported to the field with the regular sample containers and then returned in the same condition together with the field samples to the laboratory. They must not be opened in the field and the seal remains unbroken.

#### 15.1.5.2 FIELD BLANKS

A field blank is prepared in the field by filling containers with de-ionized water during the sample collection process. Field blanks are treated exactly the same way as the samples are treated using the same preservative, if any being used. During the transport and shipping, the field blank is packed labelled and sent to the laboratory with the field samples. This way it is exposed to the same environmental conditions as the sample of interest.

Field blanks can be used to verify the sampling equipment, collection environment, storage, transport and sample preparation as well as correct ambient conditions. An appropriate number of extra field blanks should be treated at each sampling point when conditions vary significantly between sampling points at a facility or sampling points are more than a kilometre in distance. The laboratory should report both the trip and field blank results along with the genuine sample results.

#### 15.1.5.3 FIELD DUPLICATES

Field duplicates also called replicates are multiple (two or more separate samples) collected by the same person using the same equipment and procedures, at approximately the same time and location under identical circumstances. They are treated exactly the same way through the field and laboratory procedures. The results of the duplicates give a measure of the precision for each analyte associated with the collection and analytical processes.

#### 15.1.5.4 SPLIT SAMPLES

Split samples are two or more portions taken from the same sample container that are processed and analyzed separately. They can be split either in the field or in the laboratory. They are analyzed separately and used to calculate test method precision. Legal samples should not be split under any circumstances.

#### 15.1.5.5 CALIBRATION STANDARDS

Primary calibration standards are prepared from traceable stock solutions which can be purchased (certified reference samples) or prepared in-house (internal reference samples) from reagent grade chemicals. These standards are used to calibrate instruments, prepare standard curves, and verify method of analysis.

#### 15.1.5.6 BLANK SPIKES

Blank spikes are prepared by adding known quantities of a standard to a volume of de-ionized water. The concentration prepared should be typically mid-range for the method used.

#### 15.1.5.7 MATRIX SPIKES

Matrix spikes are prepared by adding known quantities of a standard (blank spike) to a sample prior to analysis. The results of the matrix spike should be reported as percent recovery. In a case when the percent recovery is outside the established acceptable range, interferences with the method may be present.

### 15.1.6 DATA REDUCTION, VALIDATION AND VERIFICATION PROCEDURES

Data reduction is the process of transforming the raw analytical data printouts into reportable units. It should be specified by formula for each parameter tested and is specific to the laboratory used. Quality controls obtained must be within the control range. If data is outside the acceptable control limits, the sample must be either re-analyzed or invalidated. If the data is invalidated, it should not be reported.

The QA Manual should describe how invalidated samples are handled and the procedures used to prevent the release of incorrect data, as well as identify what corrective actions are to be performed when data cannot be validated.

### 15.1.7 PREVENTATIVE MAINTENANCE PROCEDURES

Laboratory and field instruments must be properly maintained to ensure they continue to work properly. The QA Manual should describe preventative maintenance procedures needed to assure proper operation. Effective preventative maintenance will reduce downtime, poor performance and interruption of analysis. Consumable materials such as batteries and membranes should be readily available. Replacement and repair of laboratory equipment must be planned. Routine maintenance performed by laboratory personnel should be recorded. Service contracts on major equipment should also be documented.

### 15.1.8 LABORATORY AUDITS

Laboratory internal and external audits should be conducted as part of the routine QA Program. The internal audits check that the quality procedures are in place and fully implemented and ensure the quality system is effective and achieves objectives. The review is carried out by senior management with responsibility. The internal audits are useful in preparation for external audits.

QA Manual should contain internal auditing predetermined schedule covering all activities over a reasonable period of time. Since it is inconvenient to audit all activities in a single audit, it should be spread over several quarterly or monthly audits.

External audits are carried out by an independent external body as part of the accreditation process. It verifies that the laboratory is operating in compliance with ISO/IEC 17025.

Audits may be carried out in two basic ways:

- Horizontal audit - the auditor will examine in detail single aspects of the quality system, for example calibration or reports, and
- Vertical audit - the auditor will select a sample and follow its progress from receipt to disposal, examining all aspects of the quality system relating to its testing.

### 15.1.9 CORRECTIVE ACTIONS

Any quality control tests that are suspect require the laboratory to evaluate the analytical process for errors. The errors could be from the method used, the instrument used, or personal errors by the analyst. The QA Manual should discuss what corrective actions the laboratory will take to remedy errors, such as:

- Identify and define the problem,
- Assign responsibility for investigating the problem,
- Determine the cause of the problem,
- Determine actions needed to correct the problem,
- Implement the corrective action,
- Establish the corrective action is effective,
- Management verifies the corrective action effectiveness.

### 15.1.10 RECORD KEEPING PROCEDURES

The QA Manual should describe recordkeeping for all aspects of the laboratory activities. Record keeping not only includes tracking sample and analytical information but also QC

information, equipment maintenance, calibrations, reagent purchase/preparation, etc. The QA Manual should identify where all data, reports and chain of custody forms must be stored, what form they will be stored in, and how long they will be stored.

### 15.1.11 PROFICIENCY TESTING

One of the best ways for an analytical laboratory to monitor its performance against both its own requirements and the norm of other laboratories is to participate regularly in proficiency testing schemes. Proficiency testing helps to highlight not only repeatability and reproducibility performance between laboratories, but also systematic errors, i.e. bias. Proficiency testing and other types of inter-comparisons are accepted as being an important means of monitoring quality at national and international levels. Accredited laboratories are normally required to participate in proficiency testing as an integral part of their QA Program. It is important to monitor proficiency testing results and to take corrective action as necessary.

## 16. ACRONYMS

| | |
|---|---|
| **CALA** | Canadian Association for Laboratory Accreditation |
| **GPS** | Global Positioning System |
| **ISO/IEC** | International Organization for Standardization/International Electrotechnical Commission |
| **MWWE** | Canada-Wide Strategy for the Management of Municipal Wastewater Effluent |
| **QA** | Quality Assurance |
| **QC** | Quality Control |
| **SOP** | Standard Operating Procedure |
| **VOC** | Volatile Organic Compounds |

## 17. GLOSSARY

| | |
|---|---|
| **Accreditation** | Procedure by which an authoritative body gives formal recognition that a body or person is competent to carry out specific tasks |
| **Aliquot** | Any representative portion of the sample regardless of whether a remainder is left or not |
| **Analyst** | Person responsible for the chemical analysis of samples according to the appropriate method |
| **Autosampler** | Device used to collect samples automatically, either in proportion to the wastewater flow, or as equal volumes at equal time intervals |
| **Calibration** | Procedure that checks or adjusts an instrument's accuracy by comparison with a standard or reference |
| **Certification** | Procedure by which a third party gives written assurance that a process or service conforms to specified requirements |
| **Chain of Custody** | A legal term that refers to the ability to guarantee the identity and integrity of the sample (or data) from collection through reporting of the test results. It is a process used to maintain and document the chronological history of the sample (or data) |
| **Composite Sample** | Combination of a number of smaller samples collected over a period of time and/or locations. This sampling method will produce an average sample composition when pollutant concentrations vary over time or when the frequency of individual samples containing the analytes of interest is low. |
| **Field Sampler** | Person authorized to collect the environmental sample in the field |
| **Grab Sample** | Single sample of water collected at a particular time and place and represents the composition of the water only at that time and place. It is a valid and often used sampling method in regulatory sampling of industrial wastewater effluent. Sometimes it can be the only method available to the sampler. |

| | |
|---|---|
| | Grab samples are considered simpler in maintaining the sample continuity in legal sampling situations. They are best collected directly in a sample container either by hand or by using simple field equipment such as buckets or other containers. |
| **Holding Time** | Elapsed time between sample collection and sample analyses |
| **Identification Number** | Unique number assigned to a test sample |
| **In-Situ Testing** | Testing of occurring substances in their found state |
| **Laboratory** | Facility that conducts scientific tests using established methodology |
| **Lab-Sample Custodian** | Person with responsibility for overseeing the possession, exchange, and storage of reagents, sample(s), records, and data as they are transferred between authorized personnel or stored within the laboratory |
| **Laboratory Sample Journal** | Book kept in the laboratory containing information on sample collection, analyses and other pertinent information |
| **Laboratory Technician** | Person with responsibility for preparing reagents and supplies used in the laboratory and in the field |
| **Legal Sample** | Sample taken and handled in such a manner that evidence may be given in court concerning its origin, type and constituents |
| **Method** | Body of procedures and techniques for performing a task (e.g. sampling) systematically presented in the order in which they are to be executed |
| **Procedure** | Set of systematic instructions for performing an operation |
| **Proficiency testing** | Process, procedures, and activities for standardizing a given measurement system to ensure that laboratories participating in the same program can produce comparable data |

| | |
|---|---|
| **Protocol** | Detailed written procedure for a field and/or laboratory operation (e.g. sampling, analysis) |
| **Quality Assurance** | Overall measures that a laboratory uses to ensure the quality of its operations |
| **Quality Control** | Consists of operational techniques and activities used to control and assess the quality of the measurements, product and services |
| **Sample** | Portion of material that is taken for testing |
| **Sample Analyses** | All procedures carried out on samples including any chemical alteration to sample |
| **Sample Collection** | All procedures carried out on a sample at the time of sample collection, including addition of chemical preservatives |
| **Sample Handling** | Manipulation to which samples are exposed during the sampling process, from the selection from the original material through to the disposal of all samples and test portions |
| **Site** | Area within boundaries established for a defined activity |
| **Standard Operating Procedures** | Established analytical procedures used by the laboratory |
| **Volatile Organic Compounds** | Organic chemical compounds that have high enough vapour pressures under normal conditions to significantly vaporize and enter the earth's atmosphere |

## 18. REFERENCES


1. Practices for the Collection and Handling of Drinking Water Samples, Ontario Ministry of the Environment, Version 2.0, 2009

2. British Columbia Environmental Laboratory Manual, British Columbia Ministry of Environment, 2009

3. Water and Wastewater Sample Collection and Analysis, Prince Edward Island Environment, Energy and Forestry, 2008

4. Example Small Wastewater Treatment Plant Laboratory Quality Manual - Fourth Edition, Wisconsin Department of Natural Resources, 2008

5. Implementing Quality Management: A Guide for Ontario's Drinking Water Systems, Ontario Ministry of the Environment, 2007

6. Operational Guidelines for Monitoring and Reporting Public and Semi-Public Water Systems, Manitoba Water Stewardship, 2007

7. USEPA Science and Ecosystem Support Division Operating Procedure - Wastewater Sampling, 2007

8. USEPA Science and Ecosystem Support Division Operating Procedure - Logbooks, 2007

9. New Mexico Laboratory Certification Study Guide, Version I, 2007

10. Training Manual for Supervised Persons Conducting Operational Checks, Ontario Ministry of the Environment, 2006

11. Atlantic Canada Wastewater Guidelines Manual for Collection, Treatment and Disposal, 2006

12. Laboratory Procedures Manual – 4.0 Edition, National Wild Fish Health Survey, US Fish and Wildlife Service, 2006

13. ISO/IEC 17025, General Requirements for the Competence of Testing and Calibration Laboratories, 2005

14. The Inspector's Field Sampling Manual – Second Edition, Environment Canada, 2005

15. Sample Submission Guidance Document, North Carolina Division of Water Quality Laboratory Section, 2005

16. Alberta Environment Laboratory Data Quality Assurance Policy Procedures and Guidelines, Alberta Environment, 2004

17. Code of Practice for Wastewater Systems Consisting Solely of a Wastewater Collection System, Alberta Environment, 2004

18. British Columbia Field Sampling Manual, British Columbia Ministry of Water, Land and Air Protection, 2003

19. T. Loftus: Sample Handling for USEPA National Pollutant Discharge Elimination System, 2003

20. Guide to Quality in Analytical Chemistry - An Aid to Accreditation, CITAC/Eurachem Guide, 2002

21. Protocol for the Sampling and Analysis of Industrial/Municipal Wastewater, Ontario Ministry of the Environment, 1999

22. Standard Procedures for Field Sampling, Measurement and Sample Preparation, Gulfwatch Project, 1991-1992

23. City of Greeley, USA, Industrial Pretreatment Program, Wastewater Sampling Procedures, 1984

24. Chain of Custody Procedures, USEPA Training Course

25. S J Couper, F McInnes and R Dexter: Wastewater Characterization. Comparison of Conventional With Real Time Techniques

**APPENDIX 1 - REAGENTS AND SUPPLIES SHEET**

*(Section 4. Sampling Procedure)*

| Reagent or Supplies | Preparation date | Prepared by | Quantity | Comments |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |
| | | | | |

Relinquished by: _____

Received by: _____

Time: _____

Date: _____

Are any seals used: yes no

Seal No.: _____

Date sealed: _____

Name (print): _____

Signature: _____

Seal broken: _____

Date broken: _____

Name (print): _____

Signature: _____

**APPENDIX 2 - FIELD SAMPLING DATA SHEET**

*(Section 4. Sampling Procedure)*

Industry: _____

Sampler (print): _____

Witness(es) (print): _____

Location (Site Name or Number): _____

Sample Date: _____

GPS Coordinates: _____

Photo/Video: yes no

Sample container identification number: _____ Duplicate sample: yes no

Seal number: _____

Sample Time: _____

Sample Method: composite grab

| Weather Conditions: | Sampling Day | Previous Day |
|---|---|---|
| Dry | _____ | _____ |
| Precipitation | _____ | _____ |

Air Temperature: _____

Water Temperature: _____

Discharge Flow Rate: _____

Colour: without or _____

Foam: yes no

Clarity: yes no

Immiscible Matter: none fecal paper
oil/grease branches/foliage other (describe)

Odour: none faecal earthy mouldy
slurry ammonia chlorine hydrogen sulphide
mineral oil organic aromatic compounds other (describe)

| Sample preservation description and other remarks: |
| --- |
| |

Sample Relinquished by: _____

Date and Time of Laboratory Custody: _____

Laboratory Personnel Taking Custody: _____

## APPENDIX 3 - SAMPLE SHIPPING/RECEIVING SHEET

*(Section 7. Sample Transportation)*

**Sender**

Name (print): _____

Signature: _____

Date: _____

Sent from: _____

**Carrier**

Company: _____

Signature: _____

Date: _____

Bill of Landing No.: _____

Tracking No.: _____

**Receiver**

Lab-sample custodian name (print): _____

Signature: _____

Date: _____

Shipment condition upon receipt: _____

Comments: _____

**Shipment Description**

Number of packages: _____

| Package No. | Sealed (yes or no) | Seal intact (yes or no) | Seal No. | Condition prior to shipment |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |
| | | | | |

**Shipment contents**

| Type of container | Sample container ID no. | Sealed (yes or no) | Seal intact (yes or no) | Seal No. | Condition: |
|---|---|---|---|---|---|
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

## APPENDIX 4 - SAMPLE RECEIPT AND RECORD LOG SHEET

*(Section 10. Laboratory Sample Submission)*

| Sample Container ID No. | Sample Lab ID No. | Date Received | Date Analyzed | Date Reported |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

**APPENDIX 5 - SAMPLE CONTROL RECORD SHEET**

*(Section 10. Laboratory Sample Submission)*

| Sample container ID No. | Sample Lab ID No. | Date and Time Removed from Storage | Released to: Name (print) and Signature | Reason | Sample Location | Date and Time Returned to Storage | Returned by: Name (print) and Signature | Sample Destruction | Date and Time Sample Destroyed |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |

Lab-sample custodian name (print): _____

Signature: _____

**APPENDIX 6 - ANALYTICAL DATA SHEET**

*(Section 11. Sample Analysis)*

**DETERMINATION OF THE CHEMICAL OXYGEN DEMAND (COD)**

| Sample Lab ID No. | Date | Sample (ml) (dilution) | Readings | | Result ($mgO_2/l$) | Remarks | Analysis done by: Name (print) and Signature |
|---|---|---|---|---|---|---|---|
| | | | ml FAS (0,12 M) | ml FAS (0,005 M) | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |

## APPENDIX 7 - SAMPLE DATA ARCHIVING

*(Section 12. Sample Data Recordkeeping and Archiving)*

| Archive No. | Document | Minimal Storage Time | Form of Storage | Place of storage |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

Lab-sample custodian name (print): _____