

QUALITY ASSURANCE (QA) AND QUALITY CONTROL (QC)

GUIDELINES

**FOR USE BY CLASS "A" LICENSEES
IN MEETING SNP REQUIREMENTS**

AND FOR SUBMISSION OF A QA/QC PLAN

JULY 1996

**DEPARTMENT OF INDIAN AND NORTHERN AFFAIRS CANADA
WATER RESOURCES DIVISION
AND THE
NORTHWEST TERRITORIES WATER BOARD**

Table of Contents

| | | |
|------------|--|----|
| 1.0 | Introduction and Definitions | 1 |
| 2.0 | Field Sampling..... | 1 |
| 2.1 | Sample Collection..... | 1 |
| 2.1.1 | Location | 1 |
| 2.1.2 | Sampling Equipment..... | 2 |
| 2.1.3 | Sampling Methods | 3 |
| 2.2 | Sample Handling..... | 4 |
| 2.2.1 | Preservation | 4 |
| 2.2.2 | Sample Identification..... | 5 |
| 2.2.3 | Transportation..... | 5 |
| 3.0 | Lab Analysis | 6 |
| 3.1 | Outside Laboratories | 6 |
| 3.1.1 | Lab Accreditation | 6 |
| 3.1.2 | Detection Limits..... | 6 |
| 3.1.3 | Methodology | 6 |
| 3.2 | In-House Laboratories..... | 6 |
| 3.2.1 | Identification of Analytical Laboratory/Detection Limits | 7 |
| 3.2.2 | For Overall Analytical Methods, Precision and Accuracy | 7 |
| 3.2.3 | Accuracy Requirements | 7 |
| 3.2.4 | Precision Requirements | 8 |
| 3.2.5 | Methodology | 8 |
| 4.0 | Reporting Requirements | 9 |
| 4.1 | General Submissions | 9 |
| 4.2 | Outside Laboratories..... | 9 |
| 4.3 | In-House Laboratories | 9 |
| Appendixes | | |
| | Appendix 1..... | 11 |
| | Table 1 - Summary of Preservation Requirements | |
| | Appendix 2..... | 12 |
| | References | |

1.0 Introduction and Definitions

In order to assist Licensees in completing their Quality Assurance and Quality Control (QA/QC) Plan, the following guidelines are provided, which indicates the minimum information that should be included.

These Guidelines are divided into three sections:

- 1) Field Sampling
- 2) Lab Analysis
- 3) Reporting Requirements

It is recognized that there may be different interpretations as to what is covered by "Quality Assurance/Control" due to the fact that certain Licensees have their own laboratories, while others only use commercial laboratories. For licence purposes, "Quality Assurance" and "Quality Control" refer to the following:

Quality Assurance: is the system of activities designed to better ensure that quality control is done effectively; while

Quality Control: is the use of established procedures to achieve standards of measurement for the three principal components of quality: precision, accuracy and reliability.

2.0 Field Sampling

2.1 Sample Collection

2.1.1 Location

A QA/QC plan must identify the locations of all sampling stations and the markers used to identify the stations. If the Surveillance Network Program (SNP) of the Water Licence does not specify sampling locations, locations should be chosen with help from an Inspector.

Buoys and landmarks identify sampling stations in tailings ponds and lakes, while sign post positioning usually marks stream sample stations. Stations should be used repeatedly, with the same personnel and techniques to reduce operational error. The use of Global Positioning System (GPS) to identify Latitude and Longitude for sampling stations is recommended.

2.1.2 Sampling Equipment

The Plan must include a detailed section on the equipment used for sampling, the rationale behind the choices of equipment, and descriptions of how the equipment is maintained and calibrated. Equipment and bottles should be selected so that they do not contaminate or otherwise alter the concentrations of parameters of interest.

Sampling devices, sample bottles and filtration devices should be constructed of non-metallic material. Most samples are now collected in containers constructed of high density polyethylene plastic. However, there are some exceptions, when testing for oil and grease or phenols glass containers are to be used. When conducting a fish bioassay, plastic drums are used while hydrocarbon based containers are not to be used for the collection of organic samples.

This section should also identify whether new or used bottles are used for each sample analysis. New bottles are preferred,

but sample containers may be used repeatedly with proper handling measures.

If old bottles are used, a detailed description should be included, noting how they are maintained, stored and cleaned. Usually, this will closely resemble the product manufacturer's instructions. An example of how bottles should be cleaned is outlined below:

- Rinse well with hot tap water for one minute or more.
- Empty bottle and add 30% HNO₃ to approximately 1/3 container capacity. Shake well for three to four minutes.
- Rinse vigorously with hot tap water for two minutes.
- Rinse thoroughly three times with tap water and three times with distilled water.
- Store with 0.2% HNO₃ for a minimum of one week.
- Rinse again with distilled water at least three times.

Bottles that are to be used for bacteria testing should be acid washed or autoclaved if possible.

Note: Additional information on bottle washing is also available from Water Resources Division.

2.1.3 Sampling Methods

This Section will include details on methods for sample collection and the equipment that is to be used for each station.

In lakes and ponds, regular sample bottles are used the majority of the time, but Van Dorn samples are often utilized. The sample or the sample bottle is usually lowered to mid

depth and washed three times before collecting the sample on the fourth submersion. Approximately 2% of the sample container capacity should remain to provide for mixing, preservative addition and thermal expansion.

Stream water sampling is usually done by plunging a sample bottle toward the current and allowing it to fill. Once again, the bottle should be rinsed three times before filling and room should be left for preservative addition and mixing.

A glass bottle should be used when sampling for oil and grease with the sample being collected during the first submersion and not rinsed three times first.

This section should also describe how often field blanks and replicate samples will be collected. Field blanks are samples of distilled/deionized water that are to be treated in exactly the same manner as the other samples. Blanks should therefore be taken to the field and handled and preserved as part of the sample program. They indicate when a sample may be contaminated and are indicative of general sample integrity. Replicate samples (duplicates and triplicates) are two or three samples collected from the same station at the same time. They help to ensure sample precision at the laboratory.

2.2 Sample Handling

2.2.1 Preservation

After collection, most samples must be preserved in order to prevent chemical or biochemical changes to the sample. The QA/QC plan must describe how samples from each station are to be preserved.

Preservation is generally done by the addition of certain chemicals into the bottle immediately after the sample is collected. **Table 1** is a general guide to preservatives and their appropriate concentrations. The QA/QC plan should contain more detailed information on the concentrations and amount of preservatives that will be used.

2.2.2 Sample Identification

The plan should include a description of the system used to identify samples. The system must provide positive sample identification and ensure that the identification is maintained. It is advisable to keep a logbook of samples that have already been delivered.

The identification can be maintained by marking the bottle itself or a label, with a water resistant, non-smear felt pen. The information should be clear to persons uninvolved in the sampling and may include such details as company name, sample area, SNP number, time and date.

2.2.3 Transportation

The section on transportation will describe how sample integrity will be ensured from the time of collection to completion of delivery. Delivery to the lab should be done as soon as possible after the samples have been collected.

Usually, samples are sealed and stored upright in a box with other samples to provide a snug, immobile storage space during transfer. Any samples that require refrigeration for preservation should be kept cool during transport.

3.0 Lab Analysis

Because certain Class "A" Licensees have their own analytical laboratory and others rely on commercial laboratories, this section of the Guidelines is divided accordingly.

3.1 Outside Laboratories

3.1.1 Lab Accreditation

The Licensee will identify in the plan the name of the commercial laboratory that will be conducting the analyses. A letter must be provided from the commercial lab indicating that they are accredited to conduct analyses on each of the required sampling parameters. Ideally, the lab should be accredited by the Canadian Associated for Environmental Analytical Laboratories (C.A.E.A.L.) and should provide a certificate stating parameters for which they are accredited.

3.1.2 Detection Limits

Detection limits for the commercial lab should be identified for all parameters and should be reported when any SNP data is submitted.

3.1.3 Methodology

Descriptions should be included for any methods of analysis used that are not outlined in "Standard Methods for the Examination of Water and Wastewater".

3.2 In House Laboratories

3.2.1 Identification of Analytical Laboratory/Detection Limits

Licensees using in-house labs shall identify their detection limits for all parameters and report them when any SNP data is submitted. The Licensee shall also identify the commercial lab they use to check for quality control.

3.2.2 For Overall Analytical Methods, Precision and Accuracy

The plan must describe how the Licensee will ensure precision and accuracy in their analytical methods. This includes what action will be taken if any sample results are found to be outside the appropriate ranges.

All analyses should be conducted in accordance with methods prescribed in the current edition of "Standard Methods for the Examination of Water and Wastewater" or by other approved methods. In addition, the lab should analyze standard reference material for each parameter measured. For each parameter (group) to be measured, a complete description of the sampling procedure must be documented and adhered to.

If any sample results are outside the appropriate QA/QC ranges, attempts should be made to correct the problem and the sample shall be immediately re-analyzed. If any analysis indicates a violation of a licence condition, an Inspector shall be notified of the violation, any corrective action taken, and the results of retests.

3.2.3 Accuracy Requirements

The plan should document how the Licensee will go about

ensuring accuracy in the laboratory. Accuracy is the measurement of how closely a value approximates a standard, or true value. The Licensee should identify the frequency at which certified or reference standards will be analyzed during each sampling period.

3.2.4 Precision Requirements

Precision is a measure of the closeness or repeatability of a set of values. This section will describe how and when replicate samples are taken to ensure lab precision. It is recommended that the Licensee take triplicates at one SNP station during each sampling period. If daily sampling is required at only one station, a duplicate sample should be taken each time, with a triplicate sample taken one a week.

3.2.5 Methodology

Descriptions should be included of any methods of analysis that are not taken from "Standard Methods for the Examination of Waste and Wastewater." Standard methods should be referenced.

4.0 Reporting Requirements

4.1 General Submission

The QA/QC plan will contain a section outlining what information will be reported in the monthly SNP reports. Any control charts or graphs which display the precision and accuracy of the methods used to analyze the samples should be submitted with the report. This includes warning and control limits used to determine acceptability of the data.

4.2 Outside Laboratories

The Licensee shall outline the number of replicate samples that will be collected and submitted with each SNP report. It is recommended that one set of duplicates or triplicates from an assigned SNP site, as well as the results from field blanks, be submitted with each required SNP report. This would serve as an internal/external check for the Licensee and the commercial lab.

4.3 In-House Laboratories

The Licensee shall outline the number of results from replicate samples that will be included with each required SNP report. It is recommended that two duplicate sets be collected per month at an assigned SNP site, with one set being sent to a commercial lab while the other is to be analyzed by the Licensee's lab. Analytical results from both labs should be submitted with each required SNP report. This would serve as an external check for the lab. Any results from a commercial lab should be presented on the lab's letterhead.

**FOR FURTHER INFORMATION, CONTACT THE WATER
RESOURCES DIVISION AT:**

**Box 1500
Yellowknife, NWT
X1A 2R3
(867) 669-2654 Phone
(867) 669-2716 Fax**

Appendix 1

Table 1: General Summary of Special Sampling or Handling Techniques

| Determination | Container | Minimum Sample Size (ml) | Preservation | Maximum Storage Recommended |
|------------------|---|--------------------------|---|-----------------------------|
| BOD | Sterile polyethylene | 1000 | Refrigerate 4°C | 24 hours |
| Conductivity | Polyethylene | 500 | Refrigerate 4°C | 28 days |
| Total Cyanide | Polyethylene | 500 | Add NaOH to raise pH>12 refrigerate in dark | 24 hours |
| Hardness | Polyethylene | 100 | Add Conc. HNO ₃ to lower pH<2 OR (*) unpreserved | 6 months |
| Metals, General | Polyethylene | 250 | For dissolved metals filter immediately, add Conc. HNO ₃ to pH<2 | 6 months |
| Mercury | Glass (rinsed with 1 + 1 HNO ₃) | 500 | Add Conc. HNO ₃ or pH<2 or H ₂ SO ₄ + 1 ml of 5% K ₂ Cr ₂ O ₇ , refrigerate 4°C | 28 days |
| Nitrogen: | | | | |
| Ammonia | Polyethylene | 500 | Analyze as soon as possible or add H ₂ SO ₄ to pH<2, refrigerate OR (*) unpreserved | 7 days |
| Nitrate | Polyethylene | 100 | Analyze as soon as possible or refrigerate | 48 hours |
| Oil and Grease | Glass or wide-mouth calibrated | 1000 | Add H ₂ SO ₄ to pH<2, refrigerate | 28 days |
| pH | Polyethylene | -- | Analyze immediately | 2 hours |
| Suspended Solids | Polyethylene | -- | Refrigerate | 7 days |
| Temperature | Polyethylene | -- | Analyze immediately | 0 |
| Turbidity | Polyethylene | -- | Analyze same day; store in dark up to 24 hours, refrigerate | 24 hours |
| Bacteria | Polyethylene (sterilized) | -- | None: Keep cool | 6 - 48 hours |

(*) Unpreserved = check with lab that will be analyzing the samples

Appendix 2

References:

Gilbert, Andrew (1993). "Echo Bay Mines Ltd. Environmental Laboratory Quality Assurance Plan".

Soniassy, R. (1980). "A Guide for the Collection of Water and Effluent Samples"; pp 1-16;
INAC

"Standard Methods for the Examination of Water and Wastewater" (1989); AHPA, AWWA and WPCF, 17th edition.

Water Resources Division, Indian and Northern Affairs Canada (1990). "Generic Quality Assurance (QA) Plan Guidelines for Use by the Licensees in Meeting SNP Requirements for Submission of a QA Plan"; INAC.