QUALITY ASSURANCE (QA)
AND QUALITY CONTROL (QC) PLAN
FOR THE COLLECTION OF WATER SAMPLES
AT THE
FOX-2 (LONGSTAFF BLUFF)
DEW LINE SITE

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1. Introduction

During the remediation work taking place at the FOX-2 DEW Line site at Longstaff Bluff, Nunavut water samples from any wastewater that requires discharge will be collected. The collection of wastewater samples is similar to the collection of other types of water samples.

As outlined in the proposed Monitoring Program, samples will be analyzed for the following parameters:

2. For wastewater requiring discharge:
    - pH;
    - Oil and grease;
    - Inorganic elements (total arsenic, dissolved cadmium, total chromium, dissolved cobalt, dissolved copper, dissolved lead, total mercury, dissolved nickel, and, total zinc);
    - PCBs; and
    - Phenols.

2. Sample Collection

2.1. Location

Sample locations are marked with a disk stamped with the sample number and a piece of flagging tape, attached with a 150-mm nail. Each sample location is assigned a distinct sample number. These sample numbers are recorded on a map as well as in a field notebook along with a description of the associated sample location. The GPS coordinates of the sample locations will also be recorded.

2.2. Sampling Equipment

The following table summarizes the equipment and storage requirements for each water sample type collected. New bottles are used in all cases for the collection of the water samples.

Contaminant	Container	Amount	Rinse	Storage	Special Treatment
PH	1L Plastic Bottle	Full	No	Cool	Do not filter
Hg	250 mL amber glass bottle	Full – no headspace	No	Cool	
Phenols	100 mL amber glass bottle (Qorpak vial)	Full	No	Cool	Acidify with H_2PO_4 to pH<4*
Oil and grease, PCBs	1L Teflon bottle or 1 L amber glass bottle	Full	Teflon – yes Glass - no	Cool	Do not filter

*Generally it is not possible to acidify the samples in the field due to TDGA regulations. Therefore, the samples, are acidified immediately upon receipt in the laboratory, prior to extraction

2.3. Sampling Methods

Sample bottles will be filled completely at the time of sampling. Bottles are not to be filled progressively over the course of days. If there is not sufficient water to completely fill the bottle(s), then no water sample will be collected. The bottles are to be filled with no headspace remaining to guard against volatilization of dissolved phases. Generally, the samples will be collected immediately prior to departure from the site and submitted for analysis within 48 hours.

3. Sample Handling

3.1. Preservation

The water samples will be kept cool (approximately 4^0 C) prior to and during shipping. In general, water samples will be collected when transportation from the site will be available almost immediately after, as many types of the required analyses should be performed as quickly as possible after collection.

Ideally, samples collected for inorganic analyses should be acidified in the field, at the time of collection. However, regulations concerning the transportation of dangerous goods make supplying concentrated acids in the field difficult. Where samples can not be acidified in the field, it will be requested that the samples are acidified immediately upon receipt in the lab, *prior* to decanting or sample extraction. When acidifying in the lab, the container will be rinsed with 35% HNO_3 and included with the sample.

Samples are not to be filtered at any time. If samples contain excessive sediment, the samples will simply be decanted in the southern laboratory (*following* acidification, for metal analyses) prior to analysis.

3.2. Sample Identification

Each water sample will be given a blind number that is provided on the labels of samples submitted for analysis. This sample number corresponds to the number assigned to that specific sample location which will be recorded on a map and in the field notebook.

3.3. Transportation

Samples are to be shipped by guaranteed airfreight in coolers from the site to their respective accredited laboratory for analysis. Chain-of-custody forms will be filled out and checked for each sample before shipment from the North, and the contents of shipments will be verified upon receipt in the laboratory.

4. Lab Analysis

4.1. Lab Accreditation

All laboratory analysis is carried out at accredited labs. The following laboratories are the ones primarily responsible for the analysis of water samples collected at FOX-2 (Longstaff Bluff):

1) Analytical Services Unit, Queen's University, Kingston ON; and
2) Analytical Sciences Group, Royal Military College of Canada, Kingston ON.

Accreditation certificates from these laboratories are available upon request.

4.2. Detection Limits

The following table provides a summary of the detection limits for the analysis to be performed on water samples collected at FOX-2 (Longstaff Bluff).

Parameter	Detection Limit
Total arsenic	0.003 mg/L
Dissolved cadmium	0.001 mg/L
Total chromium	0.005 mg/L
Dissolved cobalt	0.003 mg/L
Dissolved copper	0.003 mg/L
Dissolved lead	0.010 mg/L
Dissolved nickel	0.005 mg/L
Total zinc	0.01 mg/L
Total mercury	0.0005 mg/L
Oil and grease	1.0 mg/L
Phenols	1.0 μg/L
PCBs	3.0 μg/L

4.3. Methodology

The following is a summary of the methods to be used in the analysis of the water samples collected from FOX-2 (Longstaff Bluff).

4.3.1. Inorganic Elements by Inductively Coupled Plasma Atomic Emission Spectroscopy

Analyses are conducted by the Analytical Services Unit, Queen's University, Kingston, Ontario. Each sample is clearly labelled and stored at low temperatures in a secured area before and after analysis.

Each water sample (400 mL), with 3 mL of nitric acid, is placed in a beaker on a hot plate and slowly boiled to dryness. To this is added 20 mL of 2% nitric acid. The sample is then heated to boiling, cooled, and made up to 25 mL with distilled deionized water. The resulting solutions are analyzed by ICP-AES for the selected eight elements: arsenic (As), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), lead (Pb), nickel (Ni), and zinc (Zn).

4.3.2. Mercury in Water

Analyses are conducted by the Analytical Services Group, Royal Military College, Kingston, Ontario. Each sample is clearly labeled and kept at a low temperature before and after analysis.

Samples are analyzed using cold vapour generated mercury hydride atomic absorption spectrophotometry. Water samples are preserved with nitric acid and 5% potassium dichromate solution. The samples are analyzed using a Perkin-Elmer FIMS-100 Mercury System equipped with a 253.7 ηm source mercury lamp, quartz cell, Perkin-Elmer AS-90 autosampler, and the Perkin Elmer AA WinLab Analyst software. The carrier solution is 3% HCl and the reducing agent is 1.1% $SnCl_2$ in 3% HCl. Ultrahigh purity argon is used as the carrier gas, with the flow rate set between 40 and 70 mL/min. Three 500-µL replicates for each sample are analyzed. A signal is generated in the quartz cell by measuring the amount of light (wavelength 253.7 ηm) absorbed. The mercury concentrations in the samples are determined by comparing sample absorbance responses to a calibration curve generated from standards of known concentration. Duplicates, blanks and control samples are included in each run. The sample results are reported as mg/L (ppm) for water.

4.3.3. Oil and Grease in Water

Analyses are conducted by the Analytical Services Unit, Queen's University, Kingston, Ontario. Each sample is clearly labelled and stored at low temperatures in a secured area before and after analysis.

A sample volume (800 mL) is placed in a 1-L separatory funnel. The sample is extracted three times with 25 mL of dichloromethane. The extract is filtered through a funnel containing anhydrous sodium sulphate and into a round-bottom flask. The extract is rotoevaporated to approximately 2 mL and transferred to a preweighed vial, which is allowed to stand overnight in a fume hood and reweighed when dry. All values are reported as mg/L dry weight (ppm).

4.3.4. Phenols in Water

Analyses are conducted by the Analytical Services Unit, Queen's University, Kingston, Ontario.

Water samples are analyzed for phenols by means of a colorimetric assay using the Technicon Autoanalyzer system. The term "phenolic compounds" is applied to those hydroxy derivatives of benzene, which react under the conditions of the test, with the reagents used. Phenol is used as a standard and any colour produced by reaction with the reagent is reported as phenol.

Prior to analysis, turbid samples are filtered using a 0.45um Millipore filter attached to a syringe. If phenol levels are known or suspected to exceed 25 ppb, the samples are diluted to within the appropriate range in deionized water. The lower detection limit is 1 ppb.

Aliquots of each sample are applied to an Autoanalyzer, where they are mixed with phosphoric acid prior to entering an automated distillation system. The distillate is then mixed with a tartrate-borax buffer, pH 9.4, and 4-aminoantipyrine to produce an antipyrine dye, which is finally oxidized by alkaline ferricyanide. The absorbance of the colour complex is measured in a 5-cm flow cell at 505 nm. The concentration of each sample in ug/L phenol is determined by comparing a chart recorder trace of the sample with peaks produced by a similarly treated series of standards.

4.3.5. PCBs in Water

An 800-mL sample is placed in a 1-L separatory funnel and spiked with DCBP, an internal standard. Dichloromethane (25 mL) is added to the separatory funnel, which is then shaken with frequent venting. The bottom layer is decanted through a funnel containing anhydrous sodium sulphate and into a round-bottom flask. This extraction step is repeated twice more, giving a collected volume of 75 mL in the round-bottom flask. The solvent in the flask is then exchanged for hexane by rotary evaporation of the original 75 mL down to 1 mL, and 5 mL of hexane are added and again evaporated to 1 mL. The addition of 5 mL of hexane is repeated twice more, to give a final volume of 1 mL after the last rotary evaporation. The 1-mL volume remaining in the flask is pipetted onto a LC-Florisil solid phase extraction tube (Supelco) and eluted with hexane. The PCB concentrations are determined by running the resulting solutions on an HP/Agilent 6890 Plus gas chromatograph with ECD detector.

Analyses requiring ultra-low detection limits are similarly treated, but are concentrated to a known volume of approximately 0.5 mL after Florisil elution. Injection volumes of 2 mL are used in the GC analysis.

4.3.6. pH Measurement

Measurements on water samples are conducted by the Analytical Services Unit, Queen's University, Kingston, Ontario.

Water samples are measured directly using a 50 mL sample. pH is measured with a Fischer Scientific Accumet Model 10 pH meter and probe

4.4. Reporting Requirements

The following types of QA/QC samples will also be collected as part of the water sampling program. Note that if more than one type of bottle is used for each water sample, QA/QC samples will be submitted in each type of bottle used for the collection of the samples.

Field duplicates*:* Approximately 10% of the samples are collected as field duplicates. That is, two samples are collected from one sample location. These samples are handled in the same way and submitted blindly to the laboratories for analysis.

Field blanks: Field blanks consisted of distilled water and are collected to ensure that there is no corruption of samples from the sampling method. The distilled water is poured from its container into the sample container at the same time and using the same techniques as used to collect the regular water samples.

Travel blanks: The purpose of travel blanks is to ensure that there is no corruption of the sample or sample container during travel. Ideally, a full set of travel blanks should accompany each shipment of water samples. However, in cases where very few samples are shipped at a time, this guideline can be extended to a more reasonable number. Travel blanks are filled at ESG prior to leaving for the field. They are shipped with the sample bottles, stored with the sample bottles on site, brought out to the sampling location in the field, returned to the lab, and shipped to the labs with the water samples. They should not be opened unless the other bottles or water samples are opened for some reason during shipping.