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QUALITY ASSURANCE (QA) AND QUALITY CONTROL (QC) PLAN FOR THE COLLECTION OF WATER SAMPLES AT THE **PIN-3 (LADY FRANKLIN POINT)** DEW LINE SITE

Nunavut Water Board
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Public Registry

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Prepared by:
ENVIRONMENTAL SCIENCES GROUP
for
DEFENCE CONSTRUCTION CANADA
&
UMA ENGINEERING LTD.

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1. Introduction

Sewage and wastewater samples will be collected as per the Water Use Licence during the clean-up of the PIN-3 DEW Line site at Lady Franklin Point, Nunavut. The collection of wastewater samples is similar to the collection of other types of water samples.

As outlined in the Surveillance Network Program, samples will be analyzed for the following parameters:

1. For sewage effluent:
   - Oil and grease;
   - Total suspended solids (TSS);
   - Biological oxygen demand (BOD_5);
   - Faecal coliforms; and
   - pH.
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
TSS, pH	1L Plastic Bottle	Full	No	Cool	Do not filter
BOD_5	250 mL amber glass bottle	Full – no headspace	No	Cool	Do not filter
Hg	250 mL amber glass bottle	Full	No	Cool	
Phenols	100 mL amber glass bottle (Qorpak vial)	Full	No	Cool	Acidify with H_2PO_4 to pH<4*
Bacteria and coliforms	Bacti bottles (Accutest)	Full	No	Cool	Analyze within 48 hours of collection
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 nitric acid in the field difficult. Where samples cannot 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 PIN-3 (Lady Franklin Point):

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

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 PIN-3.

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
Biological oxygen demand (BOD_5)	3 mg/L
Total suspended solids (TSS)	1 mg/L
Faecal coliforms	0 counts/100 mL

4.3. Methodology

The following is a summary of the methods to be used in the analysis of the water samples collected from PIN-3.

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 were 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 was 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. Biological Oxygen Demand (BOD_5)

Analyses are conducted by Taiga in Yellowknife, Northwest Territories.

The Biological Oxygen Demand 5-day (BOD_5) test is used.[1] An airtight bottle filled to overflowing with sample is incubated for 5 days at 20 °C +/-1 °C. Dissolved oxygen (DO) is measured initially and after incubation, and BOD is calculated as the difference between initial and final DO (mg oxygen/L). The test measures the molecular oxygen used for the biochemical degradation of organic material, and the oxygen used to oxidize inorganic material such as sulphides and ferrous iron, over the incubation period.

4.3.6. Total Suspended Solids

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.

Total suspended solids (TSS) in water are determined by filtration of a sample through a glass fibre filter. A glass fiber filter is first dried in the oven at 105 °C for 15 minutes, allowed to cool and weighed. A measured volume of water (usually 500 ml) is poured through the filter, and the filter is oven-dried for two hours, cooled and reweighed. The TSS is reported as the difference in the weights divided by the volume of water.

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Analyses are conducted by Taiga Laboratory in Yellowknife, Northwest Territories.

The coliform group is defined as comprising many facultative anaerobic, gram-negative, non-spore forming, rod-shaped bacteria. Fecal coliform (FC), total coliform

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(TC), and *E. coli* (EC) are determined according to standard methods of the American Public Health Association,[2] using variations of the Membrane Filter Technique.[3]

Dilutions of the original sample are filtered through a 0.45 μ filter. Each filter is placed on an agar plate containing an indicator medium specific for the organism being assayed. For total coliform (TC),[4] the indicator medium, LES Endo Agar is used. This medium contains lactose, which is fermented by all coliforms, producing aldehydes to form red colonies with a metallic sheen. Agar plates are incubated 24 hours at 35 °C +/- 0.5 °C. For fecal coliform (FC), detecting only those coliforms from the feces of warm-blooded animals,[5] the lactose-enriched medium, M-FC, which contains an aniline blue indicator, is used. Fecal coliforms produce blue colonies after 24 hours incubation at 44.5 °C +/- 0.2 °C. Note the difference in incubation temperature between total and fecal coliforms. For *E. coli* (EC), the indicator medium, mColiBlue24 (Millipore[6]), is used. *E. coli* forms blue colonies on this medium.

4.3.8. 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.

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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

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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 were handled in the same way and submitted blindly to the laboratories for analysis.

Field blanks: Field blanks consist 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.

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