QUALITY ASSURANCE (QA)
AND QUALITY CONTROL (QC) PLAN
FOR THE COLLECTION OF WATER SAMPLES
AT THE
FOX-5 (BROUGHTON ISLAND)
DEW LINE SITE

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1. Introduction

Sewage effluent samples are collected as required by the Water Use License during the clean-up of the FOX-5 DEW Line site at Broughton Island, Nunavut. The collection of wastewater samples is similar to the collection of other types of water samples.

As stated in the Proposed Surveillance Network Program Plan for FOX-5, all waste discharged from the Sewage Disposal Facility will be analyzed for the following parameters:

- Oil and grease;
- Total suspended solids (TSS);
- Biological oxygen demand (BOD_5);
- Faecal coliforms; and
- pH.

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 sampling points are recorded as well.

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
Oil and grease	1 L Teflon or Amber Glass Bottle	Full	Yes – Teflon No - Glass	Cool	
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
Bacteria and coliforms	Bacti bottles (Accutest)	Full	No	Cool	Analyze within 48 hours of collection

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-5:

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-5.

Parameter	Detection Limit
Oil and grease	1.0 mg/L
Biological oxygen demand	3 mg/L

(BOD_5)	
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 FOX-5.

4.3.1. 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).

4.3.2. 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 ^{o}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 values are reported as the difference in the weights divided by the volume of water.

4.3.3. Biological Oxygen Demand (BOD_5)

Analyses are conducted by the Analytical Services Group, Royal Military College, Kingston, Ontario. Each sample is clearly labelled and stored at 4^{o}C in a secured area before and after analysis.

An initial 500 mL sample is visually inspected for the presence of oil, grease or synthetic contaminants. Dilution with distilled deionized water is then performed, since the BOD concentration in most wastewaters exceeds the concentration of dissolved oxygen in an air-saturated sample. Dilution volume is determined by sample history. For unknown samples, a series of dilutions are made. Seeding with active bioorganisms is employed when inspection or sample history suggests the presence of toxic components. Nutrients, including nitrogen, phosphorus and trace metals are then added and the solution is buffered (6.5 to 7.5) to ensure that the pH of the incubated sample remains in a range suitable for bacterial growth. An airtight bottle is then filled to overflowing and incubated for 5 days at 20 °C in the absence of light. The BOD is determined by comparison of the electrolytically (Ag|AgCl) determined dissolved oxygen content at the beginning and the end of the incubation period. Samples are analysed in conjunction with a nutrient water blank, glucose-glutamatic acid control, and sample duplicate. Blank dissolved oxygen uptake must be less than the method detection limit of 0.2 mg/L. The nature of BOD analysis is such that control targets and limits are established as the running mean of eight control results. Limits are based on two- and three-sigma of this mean running data set.

4.3.4. Microbiological Analysis

Analyses are conducted by the Analytical Services Group, Royal Military College, Kingston, Ontario. Each sample is clearly labelled and stored at 4^{o}C in a secured area before and after analysis. Holding time for samples is a maximum of 48 h, but immediate analysis is preferred.

4.3.4.1. Total Coliforms and E.Coli

Samples are normally received in 300 mL sterile bottles. Bottles may contain sodium thiosulfate at a nominal 100 mg/L concentration to neutralise chlorine. A 100 mL water sample is vacuum filtrated onto a 47 mm diameter, 0.45 μm pore size cellulose ester membrane filter. A final colony concentration of 20-80 target organisms per filter is ideal. Thus, dilutions may be performed prior to filtration on the basis of sample history. In the absence of such history a series of dilutions are made. The membrane filter is then

placed on a differential coliform (DC) agar plate with BCIG (5-bromo-6-chloro-3-indoyl-β-D-glucuronide) and incubated at 35 ± 0.5 °C for 24 ± 2 hours. DC agar allows for the simultaneous detection of both total coliforms and *E. coli* using a single filter and incubation temperature. After the incubation period, total coliform and *E. coli* colonies are differentiated based on the colour of the colony formed. Total coliforms appear as red and/or pink colonies, while *E. coli* produces blue/purple coloured colonies. All other colonies appearing on the membrane filter after the incubation period are counted as background. Background counts of 200 or more can interfere with the proliferation of target colonies or target reactions. In addition high background counts may lead to difficulties in obtaining accurate estimations of total coliform and *E. coli* colonies.

Data is reported in accordance with the Ontario Drinking Water protocol with data represented as Colony Forming Units per 100 mL (CFU/100 mL).

4.3.4.2. Fecal Coliforms

Fecal coliforms are stored and analysed in a similar manner to total coliforms/E. coli. However, since fecal coliforms are associated with the feces of warm blooded mammals they are able to continue to ferment lactose at 44.5^{o}C, using an FC agar. Thus, incubation at this elevated temperature is sufficient to discriminate in favour of this coliform sub group which is quantitation by the formation of blue fecal coliform colonies. Rosolic acid and bile salts inhibit the formation of non-fecal and non-enteric bacteria.

Data is reported in accordance with the Ontario Drinking Water protocol with data represented as Colony Forming Units per 100 mL (CFU/100 mL).

4.3.5. 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 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.