Quality Assurance and Quality Control

For
Water Sampling at CAM-F
Intermediate DEW Line Site
Sarcpa Lake
Nunavut

PWGSC Environmental Services
Western Region

1.0 INTRODUCTION

As the custodian of most federal lands in the north, the Department of Indian Affairs and Northern Development (DIAND) has responsibility, through the Northern Contaminated Sites Program (CSP), to manage a number of contaminated properties that are no longer maintained by the original occupant. DIAND's portfolio of contaminated sites in the north originates from private sector mining, oil and gas activities and government military activity dating back over half a century, many years before the environmental impacts of such activities were adequately understood. The former CAM-F Intermediate DEW Line Site at Sarcpa Lake, Nunavut is one of these sites.

The site will require remedial activities to remove contaminants either off site or to place contaminants into an engineered containment facility or landfill. Water sampling will be conducted throughout the remediation process for the following reasons:

1) To maintain a contractors camp at site with drinking water and waste facilities.
2) To determine potential leachate sources at site.
3) To determine concentrations of contaminants at site.

The purpose of this document is to provide a quality assurance and quality control program for the management of water sampling during the remediation process.

2.0 SAMPLE COLLECTION

Water samples will be collected during various activities. The following indicates the locations to be sampled and the frequency. Each sample location is assigned a distinct sample number and recorded on a map along with a description of the associated sample location. GPS coordinates of sample locations are collected and recorded, and photographs of each sample location are also taken. A spreadsheet can be found in Appendix A that details the sampling protocol, frequency and parameters. Site maps can be found in Appendix B indicating facilities and, where possible, proposed sample locations.

2.1 Location

A contractor's camp will be set up at the site to provide services and support for the remediation activities. The camp is scheduled to be at site for a period of two years with the duration of the construction season from mid June to the end of September. Sampling of water supplies will be conducted during the duration of camp operations. The site map in Appendix B indicates the location of the camp facilities.

2.1.1 Wastewater (Sewage)

All wastewater generated during camp operations is to be pumped into a 1,000L sewage tank. Waste is then transferred into the Biogreen treatment unit. During the first week of operation, wastewater will be discharged into a temporary lagoon for sampling to ensure that the treatment unit is working satisfactory.

Upon confirmation from the sampling event that the treatment unit is working and that all parameters meet the recommended criteria, the wastewater will be discharged on land at a distance of 100 m from the camp, 50 m from a drainage path, 100 m from the lake, and downwind from the camp. All camp wastewater will be treated using the biological treatment unit that consists of three zones, a settling zone, a fermentation zone (anaerobic), and an aeration zone (aerobic). Contingency measures for the wastewater in the event that the parameters exceed the criteria include addition of a disinfectant system immediately after the effluent holding chamber, the construction of a temporary lagoon, or the construction of lagoons (as identified in the specifications).

The total water requirement for the construction camp has been estimated at approximately 1,259,375 L. Of this volume, 683,438 L will be treated onsite through the Biogreen System and 250,000 L will be treated through the Biogenie waste water treatment system. More information on these systems can be found in Appendix C. The parameters to be analyzed within the wastewater include heavy end hydrocarbon chains as the contamination at this site historically occurred 40 plus years ago. Light end volatiles would have since dissipated. It should be noted that those hydrocarbon products used at site were diesel fuel and lubricating oils.

2.1.2 Drinking Water

Drinking water at the site will be sampled on a weekly basis to ensure the water quality meets the Health Canada Guidelines for Canadian Drinking Water Guidelines. The sampling and analyses will be provided at the water supply source and at the distribution source prior to consumption. Bottled distilled drinking water will be supplied at the camp should there exceedances be noted in the water sampling analysis.

2.1.3 Construction Camp Waste

All liquid hydrocarbon waste that cannot be incinerated will be turned over to the Construction Contractor for proper disposal on an as required basis.

2.2 Sampling Equipment

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

Contaminant	Container	Amount	Storage
Polychlorinated Biphenyls (PCBs)	Amber glass jar with Teflon-lined lid	30 g	Cool or ambient temperature
Total Petroleum Hydrocarbons (TPH) as F1, F2 & F3	Amber glass jar with Teflon-lined lid – no headspace	30 g	< 4ºC
Inorganic Elements (See note 1)	Whirlpak	50 g	Cool or ambient temperature

1- arsenic, cadmium, chromium, cobalt, copper, lead, mercury, nickel, zinc

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

Contaminant	Container	Amount	Rinse	Storage	Special Treatment
pH	1L Plastic Bottle	Full	No	Cool	Do not filter
Inorganic Elements (See note 1)	1L Plastic Bottle	Full	No	Cool	Do not filter
Polychlorinated Biphenyls (PCBs)	1L Amber glass *or Teflon,* with Teflon-lined lid	Full	Glass – no Teflon - yes	Cool	Do not filter
Total Petroleum Hydrocarbons (TPH)	Amber glass *or Teflon* with Teflon-lined lid	Full – no headspace	Glass – no Teflon - yes	Cool	Do not filter

1-total arsenic, total cadmium, total chromium, total cobalt, total copper, total lead, total mercury, total nickel, total zinc

2.3 Sampling Methodology

2.3.1 Soil Samples

Soil samples are collected in duplicate at several predetermined sample locations. Every sample location is marked with a sample tag and GPS coordinates are recorded. Soil samples are collected using one of two methods. Samples are either obtained using a plastic scoop and are stored in either Whirl Pak^{TM} bags or 125-mL amber glass jars fitted with Teflon- lined lids certified to be free of organic materials. All soil samples are kept at ambient temperature. Each sample is given a unique identifying number. Sample locations and descriptions are recorded in note pads and/or on field maps, and multiple photographs of all sample locations and the general areas that are to be delineated are taken throughout the sampling period.

Soil is sampled from the upper 10 cm and is collected so as to obtain representative material. Shallow depth samples to 30 cm are also collected at each sample location. All samples are shipped by guaranteed air- freight to Maxxam Analytics Inc in Mississauga, Ontario. Soil samples destined for total petroleum hydrocarbon (TPH) analysis in the south are kept below 4^{o}C where possible and samples to be analyzed for inorganic elements (As, Cd, Cr, Co, Cu, Pb, Zn, Hg) or PCBs are kept either cool or at ambient temperature prior to and during shipping. Additionally, 10% of all these analyses are repeated for quality control purposes.

2.3.2 Active Layer Water Sampling

Monitoring wells are installed at various locations throughout the site according to the cleanup's contract drawings and specifications to determine which contaminants are migrating and in which direction(s). The wells consist of 5-cm diameter stainless steel pipe with 100-cm long screen surrounded by a filter sock. The wells were installed with the use of a drill rig. Each hole is backfilled with grout at the base and filter sand around the screen, then capped with a bentonite seal. The wells are typically protected from damage by wildlife and the elements with a lockable external metal casing.

Monitoring wells are sampled so as to avoid alteration of groundwater chemistry through contact with drilling fluid, cross-contamination between wells, or by exposure to the atmosphere. Each well is purged before it is sampled. Purge water is monitored for pH, temperature, and conductivity, using an Extech Oyster Model 34145A or equivalent, with the appropriate probes immersed directly into a flow-through cell. The pH probe is calibrated using the accompanying buffer solutions of pH 4 and pH 7. When conductivity calibration standards are available, the conductivity probe is also calibrated.

Sampling commences once those parameters are stabilized. The sampling bottles are labeled with a unique sample number. Purging and sampling are accomplished using a low flow rate to minimize turbulence in the well and aeration of the water sample. The pump and tubing are rinsed with groundwater from the sampling location before the sample is taken. For TPH analysis, water samples are collected in 250-mL amber glass bottles which are not pre-rinsed, and are completely filled to avoid any gas pockets in the sample. Teflon bottles are used for organic, non-TPH analysis, and are pre-rinsed with the sample water. For inorganic analysis, water samples are collected the same way, but in 1-L polyethylene bottles.

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 is 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 is 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 are simply be decanted in the southern laboratory (*following* acidification, for metal analyses) prior to analysis.

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

3.0 Remediation Activities

Remediation activities or site contaminants that may impact site water resources are to be mitigated by providing the following:

1) Excavation of contaminants and materials,
2) Leachate testing of barrel residuals,
3) Waste processing and rinsing,
4) Testing of secure soil disposal facility runoff, and
5) Testing of secure soil disposal facility monitoring wells.

All soils that are to be either landfilled or transported off site will be stored in such a manner so as not to provide surface run off to water sources. All contaminated materials (residuals in barrels etc) will be either incinerated at site or transported off site for disposal. Temporary storage will be provided for these materials to ensure no run off is entering into the surrounding environment.

Sampling equipment to be used at site will be dependent on the parameters and materials to be sampled. The following is a breakdown of the sampling equipment required for the site activities as well as the associated methodology. The *CCME Guidance Manual on Sampling, Analysis and Data Management for Contaminated Sites* will be complied with.

During all sampling episodes there is the requirement for the use of new disposable nitrile gloves for each individual sample collection to avoid the potential for cross-contamination of samples. Also all water sampling equipment will be used on a one time basis to avoid cross-contamination within samples. All bottles required for the sampling episodes will be new and no bottles will be reused for sampling purposes.

Duplicate, trip and field blanks will be required for each parameter during each sampling episode as detailed in the spreadsheet in Appendix A. A blank consists of a laboratory method blank of all analytical steps but excluding the sample. It is used to determine any laboratory background contamination or contamination for the test method. A duplicate sample is a repeat analysis of a sample within the same batch including all steps of the testing method is and used as a control to assess the precision of the test method for the sample matrix. Duplicates will be provided “blind” to the laboratory. A trip blank is a sealed sample bottle containing an inert matrix that is carried to the field and returned unopened to the laboratory. Field blanks are sealed sample bottles containing an inert matrix that is carried into the field and opened under the same conditions and length of time as the regular sample. It is then re-sealed and shipped to the laboratory for analysis with the other samples. This control is used to assess possible sample contamination due to sample procedure completed in the field.

Sampling frequency for blanks, trip blanks and field blanks for each parameter can be found in Appendix A.

3.1 Wastewater

The first round of water sampling from the treatment system will be conducted out of the temporary lagoon to ensure the system is functioning to the capacity that it was designed. Once this has been confirmed, sampling will take place directly from the treatment system on a biweekly basis for the first month and then monthly thereafter to ensure compliance with the recommended criteria. The criteria for the sampling program can be found in Appendix A.

All samples will be collected from the same collection point to maintain consistency. All samples will be stored in the laboratory identified bottles.

3.2 Drinking Water

All sampling and analyses of the drinking water at the construction camp supply source must satisfy the Health Canada Guidelines for Canadian Drinking Water Quality.

The sampling and analysis will be provided at the water source and at the distribution source prior to consumption and continued thereafter on a weekly basis. Sampling will include the use of new bottles and the water collected will be stored in the laboratory identified bottles. A blank sample will be required every 1 in

3.3 Barrel Residues

Barrels will be sampled using disposable PVC barrel sleeves. The contents of the sleeves will then be deposited into the proper container for each parameter required. These residuals will be analyzed and incinerated if possible. All residuals not slated for incineration will be stored in a watertight container and disposed of as hazardous materials.

3.4 Secure Soil Disposal Facility Monitoring Wells

The monitoring wells will be sampled on an annual basis at the end of each field season.

Water levels in each of the monitoring wells will be recorded using a Solinist oil/water interface probe. The instrument will be cleaned between boreholes by field rinsing/washing using a solution of bio-wash and rinsing with de-ionized water.

After monitoring and recording the above data, each monitoring well will be purged at least three well casing volumes or until field measurements for temperature, EC and pH have stabilized or until bailed dry. This will be completed using either PVC water tubing with foot valves or with disposable PVC bailers. The wells will be allowed to recharge and representative groundwater samples will be taken. There are various parameters to be sampled in the wells; therefore the sampling protocol will follow the *CCME Guidance Manual on Sampling, Analysis, and Data Management for Contaminated Sites* document. All samples will be placed in a laboratory-supplied container and stored in ice-chilled coolers until delivery to the laboratory.

3.5 Waste Processing and Rinsing

Rinsate from barrel and or tank cleaning will be treated on-site using an oil-water separate and filtration through an activated carbon filter. Following treatment, the water will be stored and sampled to ensure that it meets the discharge requirements defined in the specification (provided in Appendix C). Once it is confirmed that the treated wastewater meets the discharge criteria, it will be released at a location a minimum 30 m from natural drainage courses and 100 m from fish bearing waters.

4.0 LABORATORY ANALYSES & METHODOLOGY

Maxxam Analytics Inc in Mississauga, Ontario, will conduct the laboratory analyses; this laboratory is accredited by the Canadian Associated for

Environmental Analytical Laboratories (CAEAL). Their membership number is 2340 and their accreditation can be viewed at the following web address:

http://216.58.107.84/lab_info.lasso?mId=2340&wId=p&-session=caeal:C667A714162b618B77tGKHTFDE05

Detection limits is predetermined by the laboratory and will be below the recommended criteria limits. Analytical methodology will be included in the laboratory chemical findings. The following is a summary of the methods to be used in the analysis of the water samples collected from CAM-F (Sarcpa Lake).

4.1 Digestion of Various Matrices for Inorganic Elements

Maxxam Analytics Inc in Mississauga, Ontario, will conduct the laboratory analyses. Each sample will be clearly labeled and stored in a secured area (before and after analysis) at a temperature appropriate for the analytical method.

4.1.1 Soil

Samples are air-dried and ground to a fine powder with a mortar and pestle. Large stones are removed, as they would not be expected to contain any anthropogenic environmental contaminants. Approximately 0.5 g of powdered sample is heated with 2 mL of nitric acid and 6 mL of hydrochloric acid overnight so that the volume is reduced to 1-2 mL. This solution is then diluted up to 25 mL with distilled deionized water. Although not all metals may be brought into solution by this procedure (some may be locked into silicate minerals), metals that are released are considered to be of greater environmental significance than are true total metals.

4.1.2 Water

For total metals, the sample is shaken vigorously. A 400-mL aliquot of sample is acidified with nitric acid, and concentrated by boiling to dryness. The sample is acidified, refluxed, transferred to a volumetric flask, and diluted up to 25 mL. The sample is then filtered and analyzed for the requested elements.

4.2 Inorganic Elements by Inductively Coupled Plasma Atomic Emission Spectroscopy

Maxxam Analytics Inc in Mississauga, Ontario, will conduct the laboratory analyses. Each sample is clearly labeled and stored in a secured area (before and after analysis) at a temperature appropriate for the analytical method.

Concentrations of the following eight elements are measured: arsenic (As), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), lead (Pb), nickel (Ni), and zinc (Zn). Samples are analyzed in batches of up to 36, which comprise up to 28 samples, 2 blanks, 4 duplicates and 2 samples of reference material (NRC MESS-3 or SS-2).

4.3 Mercury in Soil and Water

Maxxam Analytics Inc in Mississauga, Ontario, will conduct the laboratory analyses. Each sample was clearly labeled and kept at a low temperature before and after analysis.

The samples were treated in one of two ways:

(i) Water samples reported as total mercury are similarly preserved. A 5-mL aliquot is digested with 5 mL of a 1:4 mixture of concentrated nitric acid: concentrated sulphuric acid. Digestion conditions are typically 195ºC for 6 hours. The final digest is diluted up to a volume of 10 mL with distilled deionized water. A set of standards, duplicate samples, triplicate blanks, and a reference control solution are digested with each total mercury analysis event.

(ii) Mercury in soil samples are similarly digested (0.25 g dry soil, 195ºC for 6 hours, 5 mL of 1:4 concentrated nitric acid: concentrated sulphuric acid). After the digestion, the samples are diluted up to 25 mL with distilled deionized water. A set of standards, a duplicate, a blank, and a soil reference are digested with each soil run.

The samples are analyzed using cold vapour generated mercury hydride atomic absorption spectrophotometry. Water samples and digests 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% tin chloride ($SnCl_2$) 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. Soil and dissolved mercury data are not background corrected. Total mercury data are corrected for mean background absorbance. The sample results are reported to detection limits of 0.1 µg/g (ppm) for mercury in soil and 0.0004 mg/L (ppm) for total mercury in water.

4.4 pH Measurement

Maxxam Analytics Inc in Mississauga, Ontario, will conduct the laboratory analyses.

Water samples are measured directly using a 50-mL sample; the pH of soil is determined by mixing 10 g of soil with 10 mL of distilled water, allowing the mixture to settle, and measuring the pH of the supernatant. The pH is measured with a Denver Instruments model 220/300729.1 pH meter and probe, relative to buffered reference standards and control.

4.5 Polychlorinated Biphenyls in Soil

Maxxam Analytics Inc in Mississauga, Ontario, will conduct the laboratory analyses. Each sample is clearly labeled and stored at low temperatures in a secured area prior to and following analysis.

All samples are thoroughly homogenized before being sampled for the analysis. Soil samples are subsampled for determination of wet/dry weight ratio. Accurately weighed samples of wet soil (10 g) to which the surrogate standard DCBP, sodium sulphate (40 g) and Ottawa sand (20 g) are added, are extracted three times for 20 minutes with 50 mL of dichloromethane on an orbital shaker. Accurately weighed samples of soil that can not be extracted by shaker are instead extracted by Soxhlet for 4 hours at 4 - 6 cycles per hour with 250 mL of dichloromethane. In both cases, the extract is then concentrated by rotoevaporation to approximately 1 mL, and 5 mL of hexane are added and again evaporated to 1mL. This is repeated twice more, resulting in 1 mL of hexane solvent, which is then applied to a Cu-Florisil (Diagnostix) column for cleanup. The column was thoroughly rinsed with hexane and the eluate containing the PCBs diluted to 10.0 mL. A 2-mL GC vial is then filled in preparation for analysis.

An alternative extraction method used for solid samples is pressurized solvent extraction (PSE); such extractions are performed according to ASG Procedure 02-38 ‘Extraction by PSE.’ An Applied Separations PSE system capable of six simultaneous extractions is used in conjunction with 33- mL stainless steel extraction vessels, frits, and filters. Each vessel is filled to a depth of approximately 1 cm with Cu-Florisil (Diagnostix) cleanup reagent, to which a mixture of 5-10 g of sodium sulphate, 10 g of soil, and appropriate surrogate spikes was added. The remaining void was filled with Ottawa sand and glass wool. Six extraction vessels containing samples, blanks, or controls are placed in the automated PSE system. Extraction proceeds according to the following parameters: solvent hexane, static temperature 100ºC, static time 5 minutes, 1 extraction cycle. The extracts are flushed to 45-mL collection vessels using a 3-minute solvent flush and 2-minute nitrogen gas flush.

The samples are analyzed by gas chromatography (GC) with electron capture detection (ECD), using an HP/Agilent 6890 Plus Gas Chromatograph equipped with a $_{63}$Ni Electron Capture Detector (GC/ECD), a SGE HT-8 fused silica capillary column (10 m, 0.1 mm i.d. x 0.1 µm film thickness) and HP ChemStation software. The conditions are as follows: Sample volume 0.5 µL, splitless injection, temperature programmed ramp and constant helium carrier gas pressure. Nitrogen was used as a makeup gas for the ECD. All values were reported as µg/g dry weight (ppm).

Samples requiring ultra-low detection limit analysis are handled similarly, using Soxhlet or PSE extraction techniques. In each case, the samples were concentrated to a known volume of approximately. 0.5 mL. A 0.5-µL injection volume is used for GC analysis. All values were reported as µg/g dry weight (ppb).

4.6 Polychlorinated Biphenyls in Water

Maxxam Analytics Inc in Mississauga, Ontario, will conduct the laboratory analyses. Each sample is clearly labeled and stored at low temperatures in a secured area before and after analysis.

A 500-mL sample is placed in a 1-L separatory funnel and spiked with decachlorobiphenyl (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 then pipetted onto a Cu-Florisil (Diagonstix) solid phase extraction tube (Supelco) and eluted with hexane. The column was thoroughly rinsed with hexane and the eluate containing the PCBs is diluted to 10 mL. A GC vial (2 mL) is then filled in preparation for analysis. All samples are analyzed by gas chromatography (GC) with electron capture detection (ECD), using an HP/Agilent 6890 Plus Gas Chromatograph equipped with a 63Ni Electron Capture Detector (GC/ECD), an SGE HT-8 column (10 m, 0.1 mm i.d. x 0.1 µm film thickness) and HP ChemStation software. Typical conditions are as follows: Sample volume 0.5 µL, splitless injection, temperature programmed ramp, and constant helium carrier gas pressure. Nitrogen is used as a makeup gas for the ECD. All values are reported as µg/g dry weight (ppm). Analyses requiring ultra-low detection limits are similarly treated, but are extracted from 800 mL water and concentrated to a known volume of approximately 0.5 mL after Cu-Florisil (Diagnostix) elution.

4.7 CCME Method of Petroleum Hydrocarbons in Soil and Water

Maxxam Analytics Inc in Mississauga, Ontario, will conduct the laboratory analyses. Each sample is stored in an appropriate, clearly labeled container and kept at low temperatures in a secured area before and after analysis. Soil analysis was performed as prescribed in the CCME Reference Method for Canada Wide Standards for Petroleum Hydrocarbons in Soil, 2001.

(a) Soil Fraction F1

Accurately weighted samples of wet soil (5 g) are extracted on a shaker for 1 hour with 10 mL of methanol. The methanol layer is then transferred into a vial and refrigerated until analysis. A 50-µL aliquot of the extract, made up to 5 mL with water, is directly syringed into a Tekmar autosampler/purge-and-trap apparatus. The sample is then purged with high purity helium gas for 11 minutes. The trapped components are desorbed from the trap in the unit by heating to 225ºC and holding for 4 minutes. A Hewlett Packard 6890 GC-FID system is used for analysis in conjunction with an SPB-1 fused silica capillary column (30 m, 0.25 mm i.d. x 0.25 µm film thickness). The retention time is marked using nC6 and nC10 hydrocarbons, and calibration is performed with toluene. Blanks, control samples and duplicates are run at a frequency of approximately 20%. A wet/dry ratio for the sample is determined using a sub-sample. The final result is calculated using the dry weight of the sample (µg/g). Where analyses for benzene, toluene, ethylbenzene and xylenes (BTEX) are requested, the BTEX results are subtracted from the F1 fraction results.

(b) Soil Fraction F2 to F4

Soil samples are homogenized and sub-samples are dried for moisture determination. Wet soil samples (exactly 10 g) are extracted by Soxhlet for 6 hours at 4-6 cycles per hour with 250 mL of hexane/acetone (1:1). The extract is then filtered through sodium sulphate and 3 mL of toluene are added. The extract is then concentrated by roto-evaporation. A column of 15 mm diameter containing 5.0 g activated silica below 1 cm of sodium sulphate is prepared and eluted with 10 mL 50:50 dichloromethane: hexane. The concentrated extracted is added to the head of the column and eluted with 20 mL 50:50 dichloromethane: hexane. Toluene (1 mL) is added and the collected eluent is concentrated to approximately 1 mL using rotary evaporation.

Analysis is performed by GC-FID using an Agilent 6890 instrument fitted with a cool on-column injection system. A DB-1 capillary column (15 m, 0.53 mm diameter, 0.15µm film) is used to achieve separation. Blanks, control samples, and duplicates are run at a frequency of approximately 20%. Calibrations are performed and the retention time is marked using nC10, nC16, and nC34 hydrocarbons. NC50 is analyzed as the performance standard, with a required response of less than 70% of that obtained using nC10, nC16, and nC34 response factors. The final result is reported as mg/kg for each fraction. In any samples that undergo analysis for polycyclic aromatic hydrocarbons (PAH), naphthalene is subtracted from fraction F2, and the other 15 priority pollutant PAHs are subtracted from fraction F3.

(c) Water Fraction F1

For water samples, 5 mL of sample are directly syringed into the water purge-and-trap apparatus. Chromatography and calibration are performed as described for soil samples above.

(d) Water Fraction F2-F4

A 200-mL portion of the water sample is accurately measured and transferred to a clean 125-mL glass separatory funnel. Hexane (5 mL) is added and the mixture is shaken vigorously and allowed to separate. If emulsions form, the funnel is briefly sonicated to ensure adequate phase separation. Some of the hexane phase is then transferred to a GC vial and the extract is analyzed by GC under the same conditions as for soil samples, using the same calibration and retention time markers. The final result is reported in mg/L for each fraction.

5.0 SAMPLE HANDLING

All samples are to be sent to an accredited laboratory for chemical analyses and will be stored in coolers with ice to maintain the temperature below 4°C. Those samples requiring preservation will be completed as per the laboratory instructions.

5.1 Sample Identification

Sample bottles will contain a label of the accredited laboratory where the sample will be sent for chemical analyses. Just prior to collecting each individual sample, each label of the container/s will be marked with the appropriate site location, date, chemical analysis required and the name and company of the sampler. A permanent water resistant marker will be used to label the container.

5.2 Sample Transportation

Transportation of the samples will be in a ice chilled cooler to ensure the temperature of the samples does not exceed 4°C. Each chemical analysis will have holding times that they cannot exceed. These holding times will differ depending on the chemical analysis being conducted on the samples. Transportation of the samples will be via air to the accredited laboratory for chemical analysis. Please note that some of the analytical holding times can not be met (ie. pH) and will be conducted at the site using pH pens or alternatively, meters that will enable on site field testing with some accuracy.

6.0 REPORTING REQUIREMENTS

The following types of QA/QC samples will also be collected as part of the landfill monitoring sampling program. Note that if more than one type of bottle is used for a 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 were collected as field duplicates. That is, two samples were 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 consisted of distilled water and were collected to ensure that there is no corruption of samples from the sampling method. The distilled water was 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 were filled at ESG prior to leaving for the field. They were 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.

APPENDIX A

CHEMICAL ANALYSIS SPREADSHEET

APPENDIX B

SITE MAPS

APPENDIX C

WASTE WATER MANAGEMENT PLAN