



TABS ON CONTAMINATED SITES

Contaminated Sites Program - Federal Sites

This is one in a series of Technical Assistance Bulletins (TABs) prepared by Environment Canada-Ontario Region for Federal Facilities operating in Ontario.

TAB #4



Sampling & Analysis of Hydrocarbon Contaminated Soil

DESCRIPTION:

Soil samples must be collected for laboratory analysis at all petroleum release sites to document the type, location and degree of soil contamination. In order to properly characterize the soil, samples must adequately reflect the properties of the site being sampled. Proper sampling and analytical procedures of petroleum contaminated sites are outlined in this TAB.

CONTENTS

Page

1. Site-Screening Process	1
2. Additional Sampling and Analytical Requirements	1
3. Collection Methods	2
(3a) Surface Sampling	2
(3b) Test Pits	3
(3c) Borehole Installation	3
(3d) Soil Pile	3
(3e) Tank Pit	3
4. Sampling QA/QC Protocol	3
5. Quality Control Samples	4
(5a) Control Site Sample	4
(5b) Sample Blanks	4
(5c) Sample Spikes	4
(5d) Quality Control Duplicates	5
6. Record Keeping and Documentation	5
(6a) Sample Identification	5
(6b) Sample Log Record	5
(6c) Chain of Custody	5
7. Equipment Cleaning and Decontamination	6
8. Sampling Procedures	6
(8a) Free Product Sampling	6
(8b) Soil Sampling	6
(8c) Sample Handling and Transport	7
9. Analytical Procedures	7
10. Laboratory QA/QC	10

This TAB deals with the sampling and analysis of hydrocarbon contamination, but the same procedures can be used for other types of contaminants, although the analysis criteria will differ.

1. SITE-SCREENING PROCESS

Before the sampling program is initiated, site-screening techniques such as soil gas and geophysical surveys should be conducted so that soil sampling can be targeted at areas of greatest contamination.

2. ADDITIONAL SAMPLING AND ANALYTICAL REQUIREMENTS

The scope of this TAB is limited to the sampling for hydrocarbon contamination in soil. Soil characteristics, such as soil type and porosity, should also be determined in order to further define subsurface conditions and to assess chemical movement along various pathways.

For an outline of soil characteristics, as well as geology, groundwater, and other information which may be needed, refer to **TAB #2**.

If there are plans to transport the soil to a waste disposal site, provincial regulations must be followed (e.g. for Ontario, refer to provincial disposal requirements).

3. COLLECTION METHODS

There are several different methods which can be used to collect soil samples, depending upon the depth of the samples to be taken and the soil characteristics of the site. **Table 1** outlines the collection methods and indicates their appropriate usage.

It has become common practice to use organic vapour detectors (OVDs) to sample for soil contamination. Although this technique is a useful method for identifying the presence of contamination, it is not good for quantitative measurements. The results are often quite different from those obtained from laboratory analysis. OVDs may be used as site-screening tools to determine the general location and degree of contamination, but **sample collection for laboratory analysis is absolutely necessary**.

All sampling equipment should be either stainless steel or polytetrafluoroethylene (e.g. Teflon®). The equipment used for sample collection should not be the same as that used to advance the hole. Clean gloves should be worn and should be changed before each new sample is collected. When possible, a different set of equipment should be used for each sample collection. When this is not possible, the equipment should be cleaned

TABLE 1: SOIL COLLECTION METHODS AND THEIR APPLICATIONS

Collection Method	Depth of Collection	Applications
Surface Sampling	0-15 cm	<ul style="list-style-type: none"> Recent spills Low migration rates (e.g. clay soils)
Test Pit	0-5 m	<ul style="list-style-type: none"> Shallow contamination Complex stratigraphy Heterogeneous fill
Borehole	0 m - Bedrock	<ul style="list-style-type: none"> Deep contamination Dispersed spill Prevents cross-contamination Best method for identifying volatile contaminants
OTHER: Soil Pile	N.A.	<ul style="list-style-type: none"> To determine contaminant levels in a mound of excavated soil that is potentially contaminated
Tank Pit	N.A.	<ul style="list-style-type: none"> After removal of underground storage tank

between each sampling event (cleaning procedures are outlined in **Section 7**).

For test pits, boreholes, and surface sampling, the location and number of samples required are site specific, and will depend on the type of contaminant, its mobility in the environment, and the physical features of the site. The preliminary site assessment and site-screening procedures should be used to determine the appropriate number and location of samples to be taken.

An adequate number of sampling locations should be established in order to determine the horizontal and vertical extent of soil contamination. The sampling density should be increased in areas of anomalies. If no information is available for predicting the location of hot spots, a grid pattern can be used to identify sampling locations.

A sufficient number of samples should be collected from each sampling location to analyze for all parameters as well as soil characteristics. **Table 2** outlines the recommended number of samples that are needed. The soil sample should consist of soil particles not greater than 2 mm.

Additional Procedures for each Method

(3a) Surface Sampling

To retrieve the sample, follow the general collection procedures. Alternatively, a soil punch

General Collection Procedures for all Methods

A clean stainless steel trowel, scoop, or gloved hands should be used to sample the soil. If this is not possible, a backhoe may be used. **To prevent loss of volatiles**, samples should be gathered from freshly exposed soil and preserved as soon as possible, after the excavation.

may be used. The advantage of a soil punch is that it retains the sample with the soil core intact.

For slightly deeper samples, a bucket auger may be used, but the sample gathered will be a combination of soil at the surface and at greater depths.

TABLE 2: SAMPLING REQUIREMENTS FOR EXCAVATED SOIL

SOIL VOLUME (Cubic Metres)	# OF SAMPLING LOCATIONS
0-375	4
376-750	5
751-1500	6
1501-3000	7
Each Additional 1500	One Additional Sample

(3b) Test Pits

A clean backhoe should be used to excavate the pit. All excavated material should be placed on a tarp. If free product (free product is a layer of hydrocarbon contamination floating on the water table is present in the pit at a thickness greater than 2 mm follow the Free Product Sampling Procedures outlined in Section 8a. **All free product should be pumped out before sampling the soil.**

To retrieve the sample, **follow the general collection procedures.** Representative samples should be collected each time a different soil type is encountered. Samples should be taken from the areas where it is likely that the highest degree of contamination is present. The bottom sampling depth will be dependent upon the characteristics of the particular site and determined through pre-screening methods.

(3c) Borehole Installation

Soil borings should be advanced using a power auger drill. Borings should extend to the water table, or a least 1.5 m below the base of the contamination.

Soil samples often exhibit geological variability. A soil sample is generally not a homogeneous mass, but rather a heterogeneous body of material. Samples should therefore be collected at least every 1.5 m and at changes in lithology. Special consideration should be given to the sampling of the **vadose zone**, as it is an important transition area between the soil and the groundwater.

Note: A Split-spoon sampler should be employed to obtain depth specific samples.

(3d) Soil Pile (Excavated Soil)

Samples should be taken from the areas where it is likely that the highest degree of contamination is present. To retrieve the sample, **follow the general collection procedures.** If the pile has been left to sit, dig 15 to 30 cm into the pile before

taking the sample. Refer to **TABLE 2** for a listing of the number of sampling locations required.

(3e) Tank Pit

If free product is present in the pit at a level greater than 2 mm, follow the Free Product Sampling Procedures outlined in **Section 8a**. All free product must be pumped out before sampling the soil.

Samples should be taken from the areas where it is likely that the highest degree of contamination is present; the “Jar Headspace Test” outlined in **TAB #1**, may be used to help determine the areas of greatest contamination.

To retrieve the sample, **follow the general collection procedures.** Refer to **TABLE 3** for a listing of the number of samples required.

TABLE 3: SAMPLING REQUIREMENTS FOR TANK PITS

FACILITY	SAMPLING AMOUNT AND LOCATION
One Tank	Two samples below tank; and one on the lower half of each wall.
Each Additional Tank < 40,000 litres	One sample below each tank.
Each Additional Tank > 40,000 litres	Two samples below each tank.
Leaking Lines	One sample from below each suspected point of release.
Dispensers	One sample from below each dispenser which is removed.
Note: Additional samples may be needed to adequately characterize the excavation.	

4. SAMPLING QA/QC PROTOCOL

A proper Quality Assurance/Quality Control (QA/QC) program should be established in order to ensure that data obtained are **accurate and representative** of actual soil conditions. Quality control is the set of procedures used to measure and, when necessary, to correct data quality. Quality assurance is the set of procedures used to provide documentary assurance of the proper application of quality control and the resulting data quality.

The first step that should be undertaken in a sampling program is the development of a sampling protocol, a written description of the detailed procedures that should be followed. It should contain the following elements, which are described later in detail:

- Sample collection method(s) to be used.
- Sample Locations (use a map).
- Equipment (calibration and maintenance).
- The number of field samples required.
- Quality control samples (number and type).
- The laboratory to be used, as well as their QA/QC procedures (see Section 10).
- Order of sampling.
- Type, number, and size of containers (Laboratories should be able to provide containers).
- Preservation instructions (The laboratory will provide the necessary preservatives, if needed).
- Chain of custody procedures.
- Transportation plans.
- Field preparations.
- Field measurements.

5. QUALITY CONTROL SAMPLES

(5a) Control Site Sample

This sample is used to determine background levels of chemicals in the clean soil for comparison with the contaminated soil. Two control site samples (duplicate sample blanks) should be obtained and analyzed for each site that is being assessed.

The control site should have common soil characteristics with the contaminated site, but obviously should not be contaminated. It should be located near the contaminated site, and upgradient from the contamination. If there is no suitable sample blank location nearby, a site in the general region should be chosen. The control site sample should be taken just prior to the field samples.

(5b) Sample Blanks

Three types of quality control blanks are used during sampling:

Trip Blanks: Used to verify if sample contamination occurred in the sample containers and/or as a result of sample cross contamination during sample transport and storage.

Field Blanks: Used to verify if sample contamination occurred as a result of reagent and/or environmental contamination, such as from contaminated air at the sampling location.

Equipment Blanks: These are designed to check for contamination from sampling equipment (e.g. pumps and bailers) Equipment blanks are useful for evaluating the effectiveness of equipment decontamination procedures.

Blank Preparation

Use the same sampling containers for blanks as you would use to collect field samples. However, the blanks are prepared using contaminant-free (blank) water. To sample for metals and other parameters, use water that meets ASTM (American Society for Testing and Materials) Type II reagent water standards. Most laboratories supply Type II water.

Trip Blanks: Prepare by filling sample containers prior to going into the field. These blanks are carried with the field samples in the sample cooler, and are not opened in the field.

Field Blanks: Prepare by pouring blank water from a clean container into a clean sample container in the field at the same time of sample collection.

Equipment Blanks: Prepare in the field by pouring blank water into the bailer of the well, and process as if they were field samples.

Prepare a blank of each type for every **20 samples**.

(5c) Sample Spikes

Spiked samples which are prepared in the field at the time of sample collection, provide a quantitative measure of analyte loss by microbial degradation, volatilization, adsorption by sampling equipment, and other mechanisms. To avoid cross-contamination, spiked samples should not be carried in the same cooler as the field samples.

Spiked Samples are prepared by adding a known mass of the target compounds or elements of interest to a known volume of contaminant-free water or soil. The added concentration levels of target compounds should be about 20 times the analytical detection limit. The concentration of target compounds in spiking solutions and their stability must be verified independently. Both blanks and spikes should be available from the analytical laboratory.

(5d) Quality Control Duplicates

The collection of duplicate samples provides for the evaluation of the laboratory's performance by

comparing analytical results of two samples from the same location. A minimum of one set of duplicates should be collected for every 20 samples.

Obtaining duplicate samples in soil requires homogenization of the sample aliquot prior to filling sample containers. This should be accomplished by filling a properly de-contaminated stainless steel tray or bowl and mixing it with a clean instrument. Once mixing is completed, the sample should be divided in half, and containers should be filled by scooping material alternatively from each half.

6. RECORD KEEPING AND DOCUMENTATION

Record keeping is an integral part of the sampling procedure. If the sampling is not adequately documented, the sample results may well be meaningless. The documentation can be grouped into three areas: **sample identification, sample log record, and the chain of custody record.**

(6a) Sample Identification

Each sample should be assigned a unique number in the same sequence as the field samples were taken. The details of each sample should be recorded in a sample log record. To avoid laboratory bias during the analysis, the only information that the laboratory should be provided with, is the sample number.

(6b) Sample Log Record

A detailed record should be kept of the sampling procedures and should contain the following:

- Sampling site name.
- Signature of sample collector(s).
- Pit, surface, or borehole identification number.
- Sample number.
- Identification of the sample (e.g. a field sample, a control site sample, a QC duplicate, a blank, or a spike).
- Sampling date and time.
- Type of analysis required for the sample.
- If the sample is a composite or grab.
- If a preservative was added to the sample, and the type of preservation.
- The temperature at which the sample was preserved.
- Sampling equipment.

- Sampling conditions (e.g. weather).
- Relevant sample site observations.

(6c) Chain of Custody Record

The Chain of Custody form provides a record of all the personnel responsible for handling the samples. It must accompany the samples at all times. Samples should change hands as few times as possible on their way to the analyzing laboratory. The Chain of custody process acts as a mechanism for ensuring that data from the analysis of a sample are credible and defensible.

Chain of Custody Procedures:

1. Chain of custody begins at the time and point of sample collection.

2. After both field and blank sample containers have been filled, affix a signed and dated chain of custody seal, if available, across the sample cap, overlapping the joints between cap and container.

3. Record the sample number and other appropriate information on the chain of custody form. Always record transfer of custody with your signature, the signature of the person receiving the sample, the date, and time. Retain an original of this record with site sample files.

***Note:** Protect the label from water or soil marks by the use of a plastic covering.*

4. Affix signed and dated chain of custody labels to shipping containers overlapping container cover joints.

5. Chain of custody must be maintained until completion of the analysis of a sample and the reporting and acceptance of the analytical results.

7. EQUIPMENT CLEANING AND DECONTAMINATION

The cleaning and decontamination of equipment is extremely important in order to prevent the contamination of the samples that are to be collected. All excavation and boring devices should be cleaned prior to the initial excavation and before each new site is excavated.

All sampling equipment should be cleaned before each new sample is collected. Do not place the sampling equipment on the ground; instead a clean drop cloth should be used.

All cleaning should be done in one central location, and all wash/rinse solutions should be collected and contained on site. The fate of this material will be determined after review of analytical data generated from samples on site.

In the absence of specific recommended procedures for the **decontamination of equipment**, the following procedures should be adopted:

- Disassemble equipment into parts.
- Submerge and scrub equipment with a **non-phosphate detergent** and rinse with water.
- Steam cleaning is a viable alternative.
- Rinse with tap water, followed by 3 separate rinses with distilled water.
- Seal and label clean and dried equipment in new polyethylene bags, noting the date, equipment contained, decontamination procedure, and person performing the decontamination.
- **No lubricants** should be used on the drill bits or rods that are used to excavate boreholes. Only non-petroleum vegetable oil based lubricants should be used on the external surfaces of the drilling equipment that may come into contact with the drill rods. After each borehole is extracted, the augers, bits, and rods used during the drilling should be washed with lab detergent, then rinsed thoroughly with **deionized rinse water**.

8. SAMPLING PROCEDURES

(8a) Free Product Sampling

Clean gloves should be worn and should be changed before each new sample is collected. Transfer the free product (using a pipet and a pipet bulb) to a 40 ml septum vial. Filling it half full with product. Do not completely fill the vial as thermal expansion could cause it to burst. Collect all samples in duplicates. Seal, wipe, and label the vial, and store in a separate sample cooler to avoid cross-contamination.

Assign the samples a unique sample number. The details of each sample should be recorded on the sample log record. Free product samples should

be kept in a different transportation container than the other samples.

(8b) Soil Sampling

There are two common methods of soil sampling: grab samples and composite samples. A grab sample is a sample taken from one specific location, at one time. A composite sample is a combination of smaller samples taken at different locations or at different times. For the identification of hydrocarbon contamination, grab samples should be taken. They are required for the analysis of volatiles.

The sampling should proceed from the least contaminated to the most contaminated site.

The number of field samples that are required, as well as the type and size of the sample vial, is dependent upon the type of contaminant that is being sampled. **TABLE 4** provides this information.

Procedures:

- Clean gloves should be worn and should be changed before each new sample is collected.
- Completely fill each sample vial so that no headspace exists. **Minimize aeration and air contact.**
- Clean threads of jar thoroughly with a disposable wipe.
- Cap the vial and affix a chain of custody sticker.
- Label the vial.
- Wrap vial in aluminum foil.
- Place on ice in a covered cooler.
- Complete the necessary documentation.

(8c) Sample Handling and Transport

Keep samples cool (4 degrees C) and in the dark. Use ice cubes or crushed ice to chill samples as soon as they are collected, and ice packs to maintain internal temperatures in shipping containers. Deliver samples to the laboratory as soon as possible after collection, noting the recommended maximum holding times (**TABLE 5**).

Samples should be secured during storage and transport.

9. ANALYTICAL PROCEDURES

Depending on the type of contaminant that is being sampled, different parameters may be analyzed. Parameters that are suggested for analysis are shown in **TABLE 4**. Refer to **TABLE 5** for the suggested analytical method, the preservation temperature, the maximum holding time, the container type and volume for each parameter.

Don't forget to properly document the analytical requirements.

TABLE 6 outlines the clean-up criteria to be met for all relevant parameters.

10. LABORATORY QA/QC

The facility conducting the laboratory analysis should follow proper QA/QC procedures. Prior to the sampling, the laboratory should provide the following information:

1. Laboratory storage and sampling procedures.
2. Turnaround times from receipt of samples to analysis of samples to reporting of data.
3. Maximum holding times of samples according to approved methodologies.
4. Laboratory analytical methods, including accuracy of data in terms of precision, completeness, and comparability:
 - a) Identify method for each specific parameter.
 - b) If other methods are proposed, provide

justification.

5. Preparation of reagents and de-ionized water.
6. Complete itemization of laboratory equipment:
 - a) Specific name and model.
 - b) Maintenance schedules.
 - c) Calibration procedures in terms of accuracy, precision and references, and the frequency of calibration of equipment.
7. Detection limits achievable according to analytical methods and equipment used.
8. Preventative maintenance.
9. Corrective action.
10. Personnel;
 - a) Credentials.
 - b) Sample custodian(s).
11. Building or cooler security.
12. Routine assessment of data precision, representativeness, comparability, accuracy, and completeness of specific measurement of parameters involved.

After the laboratory has conducted the analysis, their report should provide the following information:

- Method of analysis.
- Date of analysis.
- Analyst's name, laboratory calibration charts and other measurement charts.
- Method detection levels; confidence limits.
- Records of calculation.
- Actual analytical results.
- Assessment of data..

TABLE 4: SOIL HYDROCARBON ANALYSIS

Petroleum Product	Analysis Type	Sample Containers
• Unleaded Gasoline	A, B	1 (120 ml)
• Regular Gasoline, Aviation Gasoline	A, B, D	1 (500 ml); 1 (120 ml)
• Fuel Oil, Diesel Fuel, Kerosene, Jet Fuels, Mineral Oil/ Spirits, Motor Oil	A, C, H	2 (250 ml); 1 (120 ml)
• Petroleum Solvents	A, C, G	2 (250 ml)
• Waste Oil, Any Waste Petroleum Product	A, C, E, F, G, H	3 (250 ml); 1 (500 ml)
• Unknown Petroleum or Hydrocarbon Mixture	A, B, C, E, G, H	1 (500 ml); 2 (250 ml)
• Crude Oils, Hydraulic Fluids	C, H	2 (250 ml)
• Coal Gasification	H	1 (250 ml)
A - Benzene, Ethylbenzene, Toluene, Xylenes (BETX) C - Total Hydrocarbons (TPH) as Fuel Oil E - Lead, Chromium, Cadmium G - Phenols B - Total hydrocarbons (TPH) as Gasoline D - Lead F - Polychlorinated Biphenyls (PCBs) H - Polycyclic Aromatic Hydrocarbons (PAHs)		

TABLE 5: SAMPLE COLLECTION AND ANALYTICAL PROCEDURES

PARAMETER	BTEX	TPH- GASOLINE	TPH-FUEL OIL	PHENOLS (non- chlorinated)	METALS	PAHs	PCBs	FREE PRODUCT
Container Volume	40 ml	40 ml	250 ml	40 ml	500 ml	250 ml	250 ml	40 ml
Container/Cap Material	Widemout h Glass Jar/Teflon Lined Cap	Widemouth Glass Jar/Teflon Lined Cap	Widemouth Glass Jar/Teflon Lined Cap	Widemouth Glass Jar/Teflon Lined Cap	Glass Jar	Widemouth Glass Jar/Teflon Lined Cap	Widemouth Glass Jar/Teflon Lined Cap	Widemouth Glass Jar/Teflon Lined Cap
Preservation (temp.)	Cool 4°C	Cool 4°C	Cool 4°C	Cool 4°C	Cool 4°C	Cool 4°C	Cool 4°C	Cool 4°C
Max. Holding Time	14 days	14 days	Extract within 7 days / Analyse within 40 days	Extract within 14 days/ Analyse within 40 days	6 months	Extract within 14 days/ Analyse within 40 days	Extract within 14 days/ Analyse within 40 days	Extract within 14 days/ Analyse within 40 days
Recommended Analytical Method(s)	Purge and Trap GC/PID	Purge and Trap GC/FID	Solvent Extraction with Freon 113 IR Analysis	Solvent Extraction GC/MS	ICP Scan	HPLC/ Fluorescenc e & UV detector	Solvent Extraction GC/ECD	GC/PID
Recommended EPA Method	EPA 5030/802 0	EPA 5030/8015	EPA 9073	EPA 8270	EPA 6010	EPA 8310	EPA 8080	NA.

ECD : Electrolytic Conductivity Detector

FID : Flame Ionization Detector

GC : Gas Chromatography

HPLC: High Pressure Liquid Chromatography

CFM : Containers for metals

ICP : Inductively Coupled Plasma

IR : Infra-red

MS : Mass Spectrometry

PID : Photoionization Detector

CFO : Containers for Organics

TABLE 6: SOIL CLEAN-UP LEVELS (IN PPM-DRY WEIGHT)*

Parameter	Agricultural	Residential / Parkland	Commercial / Industrial
Benzene	0.05	0.5	5
Ethylbenzene	0.1	5	50
Toluene	0.1	3	30
Xylene	0.1	5	50
TPH as Gasoline **	40	400	2000
TPH as Fuel Oil	40	400	2000
PCBs	0.5	5	50
Phenols	0.1	1	10
Cadmium	3	5	20
Chromium (total)	750	250	800
Lead	375	500	1000
Parameter ***	A	B	C
PAHs (group1)	0.1	1	10
PAHs (group 2)	0.1	5	50
PAHs (Pyrene)	0.1	10	100

* Guidelines taken from the CCME Interim Canadian Environmental Quality Criteria for Contaminated Sites. The document contains a list of guidelines for all parameters.

** Guidelines taken from Alberta MUST Regulations

*** CCME Interim Guidelines from PAH contamination at abandoned coal tar sites (refer to this document for specific chemical parameters found in group 1 and Group 2 and for the application of the "A, B, C" Guidelines

Note: The Petroleum Hydrocarbon Canada Wide Standards (PHC CWS - 2000/2001) should be consulted for additional guidance.

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