LABORATORY ANALYSIS OF SAMPLES TAKEN FROM PETROLEUM RELEASE SITES
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Field screening for hydrocarbon vapors at a petroleum release site provides enough information to determine the presence of contamination and the relative concentration. In many cases, screening information may be sufficient for decision-making purposes, for example, when there is no uncertainty about the type of contaminant (i.e., the source is from a known spill or identified leaking tank). There are instances, however, when laboratory analyses may be desired during an investigation. For example, analytical data from a laboratory may be used for the following reasons:

1. To determine if a potable water supply has been affected and the need for an alternative water source;

2. To identify contaminants that cannot be detected (or differentiated) by field screening techniques; and

3. To meet state and local requirements (e.g., verification of a cleanup, site investigation activities, etc.).

Given the cost and time associated with laboratory analyses, it is important that certain measures are taken to ensure accurate results. In order to avoid resampling (which results in unnecessary delays and expenses), consultants should be aware of the following common mistakes and possible solutions:

Mistakes	Solutions
Selection of inappropriate type of analysis	Consult with the laboratory and existing guidelines for recommended analyses. See Table 1 for a summary of some common analytical options.
Use of improper container and preservative	Consult with laboratory and use containers that they have approved and/or provided.
Samples unusable due to breakage or cross- contamination	Collect duplicates. Place field blank in storage container with samples.
Samples unusable due to improper storage and excessive holding time	Arrange with laboratory ahead of time for analysis to be run as soon as possible after delivery. Store samples in ice-filled cooler immediately following collection.
Analytical results indicate improper labeling or sample misidentification	Label samples very carefully in the field and carefully fill out chain-of-custody forms prior to delivery to the laboratory.

I. Analytical Procedures

Analytical procedures should be consistent with federal and state guidelines. A summary of potential analytical parameters to be performed on samples taken at petroleum release sites and the corresponding methodology for each parameter are provided in Table 1. The required containers, preservation techniques, and holding times are listed in Table 2.

The following are suggested procedures for the analysis of samples (soil and water) at petroleum release sites. While the use of these procedures is not mandatory, the North Dakota Department of Environmental Quality (NDDEQ) staff has the option of rejecting any analytical results which are considered incomplete, inadequate, or inaccurate. Proper sampling should include the following elements:

Table 1. Summary of Analytical Procedures for Soil and Water Matrices

	Matrix to be Analyzed	Analytical Method	Instrumentation
1.	**Water**		
	a. Benzene, Toluene, Ethylbenzene and Xylene (BTEX), Purgeable Aromatics	5030A/8020A 602	GC GC
	b. All Purgeables	8240B 8260A 624	GC/MS GC/MS GC/MS
	c. Purgeable Halocarbons	5030A/8010B 601	GC/ELCD GC/ELCD
	d. Gasoline Range Organics	8015B	GC/FID
	e. Diesel Range Organics	8015B	GC/FID
	f. Semivolatile Organic Compounds	8270B	GC/MS
	g. Oil & Grease	1664	Gravimetric
	h. Lead – Total	3010A/7420 3010A/6010A 3020A/7421	Flame AA ICP GFAA
	i. Chromium – Total	3010A/7190 3010A/6010A 3020A/7191	Flame AA ICP GFAA
	j. Ignitability Flash Point	1010 1020A	Pensky-Martens Closed Cup Setaflash Closed Cup
	k. Total Organic Halogens (TOX)	9020B 9022	 Neutron Activation
2.	**Soil**		
	a. All Purgeables	5035/5030B/8260B	GC/MS
	b. Gasoline Range Organics	8015B	GC/FID
	c. Diesel Range Organics	8015B	GC/FID
	d. Semivolatile Organic Compounds	8270B	GC/MS
	e. Oil & Grease	9071A	
	f. Lead – Total	3050A/6010A 3050A/7420 3050A/7421	ICP Flame AA GF AA
	g. Chromium – Total	3050A/6010A 3050A/7190 3050A/7191	ICP Flame AA GFAA

Table 2. Required Containers, Preservation Techniques, and Holding Times			
Name	**Container**	**Maximum Preservation**	**Holding Time**
Metals:			
Chromium VI	P,G	Cool, 4°C	24 hours
Mercury	P,G	HNO_3 to pH <2	28 days
Metals, except Chromium VI	P,G	HNO_3 to pH <2	6 months
Oil and grease	G	Cool, 4°C, H_2SO_4 to pH <2	28 days
Volatile Organics:			
Concentrated waste samples	8 oz. Widemouth glass with Teflon liner	None	14 days
Liquid samples			
No residual chlorine present	Two 40 ml vials with Teflon-lined septum caps	4 drops conc. HCl, cool, 4°C	14 days
Residual chlorine present	Two 40 ml vials with Teflon-lined septum caps	Collect sample in a 4 oz. Soil VOA container which contains 4 drops of 10% sodium thiosulfate. Gently mix sample and transfer to a 40 ml VOA vial that contains 4 drops conc. HCl, cool to 4°C.	14 days
Soil/sediments and sludges	4 oz. (120 ml) Widemouth glass with Teflon liner	Cool, 4°C	14 days
Semivolatile Organics:			
Concentrated waste samples	8 oz. Widemouth glass with Teflon liner	None	14 days
Liquid samples			
No residual chlorine present	1-gallon or 2 1/2-gallon amber glass with Teflon liner	Cool, 4°C	Samples extracted within 7 days & extracts analyzed within 40 days.
Residual chlorine present	1-gallon or 2 1/2-gallon amber glass with Teflon liner	Add 3 ml 10% sodium thiosulfate per gallon, cool, 4°C	Samples must be extracted within 7 days and extracts analyzed within 40 days.
Soil/sediments and sludges	8 oz. Widemouth glass with Teflon liner	Cool, 4°C	14 days

II. Internal Quality Control Checks

In order to provide the necessary quality control of samples taken at petroleum release sites, duplicate samples, blind spikes, field blanks, split samples, trip blanks, and background samples should be collected and submitted to a quality assurance laboratory. The work/sample plans should state the type of sample and frequency with which a sample will be taken. A suggested frequency for the different sample types is noted in the following table:

Table 3. Internal QA Sampling Frequency

Type of Sample	Frequency	Comments
Duplicate*	10% of samples collected, if possible	Aqueous samples
Blind Spike**	When possible, in coordination with the laboratory.	
Field Blank***	One per sampling incident, if appropriate	Aqueous samples
Split Sample****	As appropriate.	
Trip Blank	Two 40-ml vials filled with deionized water/ cooler.	Aqueous samples; only if samples are analyzed for organic volatiles.
Background Sample	As appropriate.	Primarily associated with on-site soil samples.

Every effort should be made to assure that representative samples are collected.

* Duplicate samples (these are independent samples collected at the same sampling location during the same sampling event).

** Blind spike samples (samples resulting from the addition of compounds to samples).

*** Field blanks (these are obtained by running analyte-free deionized water through sample equipment after decontamination, and collecting the water in appropriate containers for analysis).

**** Split sample (the sample is divided into more than one sample container for separate analyses, usually by different laboratories).

Guidance Documents: The following Division of Waste Management's *Underground Storage Tank Program* guidance documents should be used and referenced while conducting site investigations or corrective actions (see https://deq.nd.gov/wm for UST rules and other guidelines referenced below):

1. Groundwater Monitoring Well Design & Installation
2. Decommissioning of Monitoring Wells and Boreholes
3. Procedures for Headspace Analysis of Gasoline Contaminated Soils
4. Procedures for the Collection of Soil Samples at UST Sites
5. North Dakota Underground Storage Tank Rules
6. Guidelines for the Disposal of Tank Sludge
7. Guidelines for Proper Land Treatment of Petroleum Product Contaminated Soils
8. Land Treatment of Petroleum Contaminated Soil: Single Application Sites
9. Guidelines on Report Format for Site Investigations