



ANALYTICAL METHODOLOGY FOR GROUNDWATER AND SOIL ASSESSMENT GUIDELINES

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ANALYTICAL METHODOLOGY FOR GROUNDWATER AND SOIL ASSESSMENT GUIDELINES

The analytical methodologies discussed in this document pertain to groundwater and soil analyses for petroleum related sites regulated under RCRA Subtitle I, the SUPERB Site Rehabilitation and Fund Access Regulations (R. 61-98), and the Pollution Control Act. All required analyses are to be performed by a laboratory certified by the South Carolina Department of Health and Environmental Control (SCDHEC) per State Regulation 61-81 for the appropriate EPA methodology. Deviations from the analytical methodology presented must be approved prior to use for compliance purposes. Questions concerning appropriate analytical methodology should be directed to the Underground Storage Tank Management Division. The laboratory report for the analyses must include the S.C. Laboratory Identification Number for the laboratory performing the analyses. If analyses are subcontracted to another laboratory for analysis, the laboratory's S.C. Laboratory Identification Number must be documented beside the analyses performed by that laboratory.

Sampling Methodology

Sample containers, preservation techniques, and holding times for the required analyses for water and soil are addressed in Tables 1 and 2. Additional information is provided below concerning the collection and the analysis of volatile organic compounds (VOCs).

It is very important that you contact the analytical laboratory for the proper sample containers, chain-of-custody forms, and sample collection instructions prior to sampling for volatile organics. The low-level volatile analysis will require increased coordination between the field personnel and laboratory personnel. When collecting the samples, the appropriate tools and sample collection devices must be available. For more information on sample collection procedures, refer to EPA Publication SW-846, Third Edition, Updates I, II, IIA, IIB, III, IIIA, IIIB, IVA, IVB, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods". The document is available on-line at <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>.

Volatile Organic Compounds (VOCs)

Method 5030B incorporates the analysis of aqueous samples, soils, and other solid samples with a high VOC concentration (greater than 200 µg/kg) or a high concentration oily waste sample (greater than 200 µg/kg) using the conventional purge and trap apparatus. This procedure is used in conjunction with EPA Method 8015B and 8260B.

Method 5035 describes the collection and analysis of low-level VOC solid samples (soils, sediment, and solid waste with VOC concentrations in the range of 0.5 to 200 µg/kg). The analysis consists of a closed system purge-and-trap method. Method 5035 utilizes a hermetically sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible. The method also describes the procedures for collecting and preparing solid samples and oily wastes containing high concentrations of VOCs. This procedure is used in conjunction with EPA Method 8015B and 8260B.

Method 5030B Ground-Water Sample Collection

Standard 40 mL glass screw-cap VOA vials with Teflon-lined silicone septa are to be used for collecting water samples for volatile analyses. Samples must always be collected in duplicate after correctly purging the ground water monitoring wells. The sample container needs to contain the necessary preservative and the water should be introduced into the vials slowly without introducing any air bubbles within the vial. The vials should be completely filled at the time of sampling, so that when the septum cap is fitted and sealed, and the vial inverted, no headspace is visible. The vial must not be opened prior to analysis to preserve its integrity. Immediately after collection, the sample vials must be labeled and stored at 4°C.

Method 5035 Solid Sample Collection

There are various options that can be used when collecting soil samples for low-level VOCs. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of volatile components. Always wear gloves whenever handling the tared sample vials. Several techniques may be used to perform the transfer of the sample to the relatively narrow opening of the low concentration soil vial such as the ENCORE™ sampler, a cut off disposable plastic syringe, or a stainless steel spatula.

(1) ENCORE™ Sample Collection:

The ENCORE™ sampler is both a sampler and a container for low-level soils. It is designed to collect an average weight with the exact weight to be determined in the lab. It is disposable and is also designed to have zero headspace. If the ENCORE™ sampler is used, the field personnel must get the sample to the laboratory within 48 hours of collection to preserve the sample.

A separate sample may need to be collected to enable the laboratory to perform a pretest on the soil to determine if the soil sample contains carbonate minerals that will effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the sample cannot be preserved with sodium bisulfate, the sample is then transferred to a pre-weighed vial with 5 mL of reagent water added to it. The unpreserved sample in the vial must either be analyzed immediately or frozen within the 48-hour time frame. It still must be analyzed within the 14-day holding time. Extra samples must still be collected for high concentration analysis, screening, and moisture content.

(2) Coring Device/Drive Tube with Field Preservation:

If using a coring device or drive tube device, collect several trial samples with the device and weigh each trial sample, noting the length of the soil column in the device. Use the data to determine the length of soil in the device that corresponds to 5.0 g. This can be done prior to going into the field to collect the samples, or if a portable balance is available this can be performed in the field. The sample is collected using the device and placed into a pre-weighed vial, provided by the laboratory, with the sample preservative and a stir bar. It is very important that the transfer of the sample be made as quickly as possible with very little disturbance to the soil to avoid loss of volatiles. The weight of the sample can be determined in the field and also determined in the laboratory.

A portable balance (capable of weighing to 0.01g) can be used to weigh the sealed vial containing the sample to ensure that approximately 5.0 g of sample has been added to the vial, but this is optional. This weight determination, if made in the field, must be documented on the sample container and in the field records. If a portable balance is used, the balance calibration must be verified in the field using an appropriate weight for the sample containers employed. The appropriate reference weight must be used at least once daily prior to weighing any samples and records must be kept for the balance checks.

For Any Sample Collection Method:

All low-level soil samples must be collected at least in duplicate to allow the laboratory an additional sample for reanalysis. The additional soil sample(s) must be collected from the same soil stratum or the same section of solid waste being sampled and within close proximity to the location from which the original sample was collected.

Additional samples must be collected for screening, dry weight determination, and high concentration analysis (if necessary) without the preservative. If high concentration samples are collected in vials containing methanol, an additional sample should be collected for screening and dry weight determination in a vial without preservative.

The laboratory performing the analysis needs to be contacted prior to sample collection to ensure that all necessary containers (with or without preservative) are available and that the proper sampling technique is used.

Options for sample collection appear below:

Option 1 - Encore

- 1) Core with the Encore™ device in the field.
- 2) Collect two Encore™ samples/sample location.
- 3) Collect one glass container (2 oz.) with septum lid for the high level and to determine moisture content.
- 4) Collect one headspace vial for screening.
- 5) Laboratory preservation/preparation within 48 hours of collection for the Encore™ samples and high level samples.

Option 2 - Coring Device/Drive Tube with Field Preservation:

- 1) Core and weigh samples in field. (Balance required).
- 2) Collect two low-level vials (preserved). Vials must be obtained from the laboratory performing the analysis.
- 3) Collect one glass container (2 oz.) with septum lid for high level and moisture determination. High-level sample must be transferred to methanol within 48 hours of collection or the high level sample can be collected in methanol.
- 4) Collect one headspace vial for screening.

Option 3 - Field Screening

- 1) Field Screening.
- 2) Core and weigh in field.
- 3) Collect 2 low-level vials (preserved) Or One high-level vial.

Method 5035 Sample Preservation

Method 5035 addresses the preservation of the low concentration soil samples with sodium bisulfate to ensure a sample pH of 2. If using option 1, two pre-weighed sample vials with the sodium bisulfate preservative solution must be obtained from the laboratory along with two pre-weighed sample vials with 5 mL of reagent grade water (used if vigorous effervescence). The laboratory will also provide a sample vial to check the reaction of the soil with the sodium bisulfate preservative solution. Soil samples that contain carbonate minerals (limestone) may effervesce upon contact with the acidic preservative solution in the low-level concentration sample vial. If the amount of gas generated is very small, any loss of volatiles as a result of such effervescence may be minimal, if the vial is sealed quickly. If at all possible the sample should be preserved. A test sample should be collected, added to a vial with the preservative and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and collect low concentration samples in vials that contain 5 mL of reagent water. Soil samples in reagent water with no preservative must be analyzed within 48 hours of collection or frozen within 48 hours of collection. Records must be maintained on the chain-of-custody documenting the necessary actions taken by the laboratory.

Soil samples for volatile analysis that are preserved with the sodium bisulfate preservative solution are to be cooled to approximately 4°C, packed in appropriate containers, and delivered to the laboratory on ice. These are to be analyzed within 14 days of sample collection. Samples receiving no preservation in the field other than cooling to 4°C must be delivered to the laboratory the same day as collected. Once in the laboratory, the samples must be analyzed or frozen within 48 hours of sample collection. The laboratory sample storage area must be free of organic solvent vapors. All samples need to be analyzed as soon as possible, not to exceed the designated sample holding time of 14 days from sample collection for preserved samples or 48 hours for unpreserved samples.

Field Notes for Sample Collection

Field notes must be maintained by the sample collector to document the collection of the samples. These field notes must be attached to the chain-of-custody form submitted to the laboratory and the laboratory must attach the copy of the completed chain-of-custody with the attached field notes. If the field notes do not accurately reflect how the sample was collected, the results for the testing may not be accepted by the Department.

Analytical Methodology

Tables 3 and 4 address the analytical methodology approved for the analysis of groundwater and soil samples pertaining to gasoline, diesel, fuel oil, and kerosene. Tables 5 and 6 address the analytical methodology approved for the analysis of groundwater and soil samples pertaining to waste oil. Other sample extraction procedures can be used provided the laboratory has the necessary certification.

Sample screening needs to be performed by the laboratory to determine the appropriate sample preparation procedure for the particular sample, such as the low-concentration closed-system direct purge-and-trap method, the high concentration method, or the nonaqueous liquid (oily waste) methanol or PEG dilution procedure. Calibration standards used for instrument calibration must also contain approximately the same amount of sodium bisulfate preservative as the sample, as the presence of preservative may affect the purging efficiencies of the analytes. If sodium bisulfate preservative is not used for the samples, then the standards used for instrument calibration must also not contain sodium bisulfate. Therefore laboratories will possibly have two different calibration curves for soil analysis. Analysis records must document the referenced calibration curve used for the quantification of sample results.

Chain-of-Custody and Sample Analysis Documentation

Soil and groundwater samples collected for petroleum-related sites must be handled in a manner that is consistent with the analytical testing method and that preserves the integrity of the sample. All environmental sample results submitted to the Department for work performed at these sites must include adequate documentation of proper sample collection and analysis. The details of soil collection must be documented on the laboratory chain-of-custody form, field notes, laboratory results, and/or the environmental report. Sufficient details should be included to address collection and preservation methods employed in the field and in the laboratory. The Responsible Party (RP), usually the tank owner or operator or other entity responsible for the release, must ensure that the environmental contractor gathers this information. The RP is also responsible for ensuring that the laboratory is certified for the analyses in question and that this information is submitted to the Department with the sample results.

The following information must be included in the documentation submitted to the Department:

- 1) South Carolina Laboratory Identification Number: This number is assigned to a laboratory once it is granted certification by the South Carolina Department of Health and Environmental Control. **Certification is granted on an analyte-by-analyte basis; therefore, it is imperative that the laboratory's certification status for a particular analyte or method be verified through the Department (Call (803) 896-0970).** Analyses performed by a laboratory without certification for the analyte or method will not be accepted by the Department.
- 2) Sample type, description, location, and UST Permit #: Describe the sample (soil, groundwater, surface water, etc.) and the location where the sample was collected. The field/laboratory identification must clearly correlate the sample with sample locations shown on a map.
- 3) Type of sample preservation: Indicate type of sample preservation performed on the sample. The laboratory must verify preservation of the water and soil samples for volatile analyses.
- 4) Sample integrity upon receipt in the lab: Describe if the sample container was broken or cracked, if the sample cap was loose or poorly sealed (e.g. due to sediment on the cap threads), or if the sample integrity was otherwise questionable. For Method 5035, the laboratory is responsible for ensuring that these samples have been collected properly. Any discrepancies must be addressed on the chain-of-custody form and on the sample results. Preservation steps taken by the laboratory must be sufficiently documented on the chain-of-custody form or laboratory record.
- 5) The temperature of the sample when received: If the sample was received in ice and solid ice is still present, report the sample as "received on ice." Exact sample temperature need not be reported for samples received on ice. If the sample is cooled using "blue ice" packs or the ice used in shipping is

melted, then the temperature of a "temperature blank" must be reported. If the ice used to ship the sample has melted, the temperature of the melt water may be substituted for a temperature blank.

- 6) The analytical methods used to analyze the samples: When using an EPA method, specify the number and when analyzing for a specific petroleum product, list the petroleum product with the method for sample preparation or extraction and then list the determinative method. For example, BTEX in soil - EPA Method 5035/8260B.

Initial lead or other RCRA metal samples should not be filtered. If elevated levels of metals are detected, the UST project manager will direct the number and type of additional metal samples (filtered lead, organic lead).

- 7) Sample results: The analytical result should be reported in the appropriate units for the sample matrix analyzed and corrected for any dilutions performed on the sample or extract. Report soil results as $\mu\text{g}/\text{kg}$ or mg/kg on a dry-weight basis with percent moisture and groundwater results as $\mu\text{g}/\text{L}$ or mg/L . For each result reported, indicate any dilutions performed on the sample or extract. **If the specified action levels for particular contaminants cannot be achieved due to matrix interferences, the laboratory must explain what steps were taken to overcome these interferences. The laboratory can use antifoam reagents and appropriate cleanup procedures to eliminate these matrix interferences.**
- 8) Laboratory reporting limit: The laboratory's reporting limit for the specific method or analyte must be indicated with the analytical result. The reporting limit is the concentration in a sample that is equivalent to the concentration of the lowest calibration standard. This is the concentration at which the entire analytical system must give a recognizable signal and an acceptable calibration point.
- 9) Reporting Limit: The reporting limit is based on the lowest non-zero standard in the calibration curve.
- 10) Surrogate recovery data: Surrogate recovery data for each sample must be reported for each organic determinative method employed.
- 11) Date and time sampled, received, extracted and analyzed: The laboratory's certificate of analysis must include the date and time the sample was collected, date and time the sample was extracted by the lab (if applicable), date and time the sample analysis was completed, and the analyst. The date and time the sample was received in the lab must be included on the chain-of-custody. For EPA Method 5035, if using the Encore™ sampling device, the laboratory must also document the date of transfer from the sampling device to the analysis vial and storage conditions. If the date of analysis is past the acceptable holding time, the lab needs to ask for a resample to avoid rejection of results by the Department.
- 12) Product type identification: If product type identification is made for a petroleum hydrocarbon, it needs to be identified and quantified. Other helpful information such as the type of site or the results of field screenings are to be reported.
- 13) Flagged data: Any questionable data is to be flagged (such as exceeding holding times, improper sample collection, QC requirement failures, instrument failure during analysis, improper preservation of sample, or any other relevant factors).
- 14) Sample odor: Any obvious sample odor must be reported.
- 15) Unidentified but detected contaminants: Any detected but unidentified contaminants or peaks must be reported.

A complete chain-of-custody form must be maintained and submitted with the results for all samples reported to SCDHEC. The chain-of-custody forms must document all transfers and receipts of the samples. Field notes must be attached to the chain-of-custody to accurately document how the samples are collected. Certificates of analysis from each laboratory performing the analyses must be also be submitted.

If you have questions concerning the methodology requirements and/or questions concerning a laboratory's certification status, please contact the Office of Environmental Laboratory Certification of SCDHEC at (803) 896-0970.

If you have questions or would like a copy of the South Carolina UST Control Regulations or other UST related documents, please contact the Underground Storage Tank Management Division at (803) 898-2544. Copies can also be obtained at <http://www.scdhec.gov/Agency/RegulationsAndUpdates/LawsAndRegulations/LWM> .

Table 1
Required Containers, Preservation Techniques, Holding Times
Ground-Water Samples¹

Contaminant	Container ²	Preservation ³	Holding Time ⁴
Volatiles ⁷ (BTEX, MTBE, Naphthalene, & 1,2-DCA)	2 x 40-mL glass vials with Teflon-lined septum caps	Cool to 4°C and adjust pH to less than 2 with H ₂ SO ₄ , HCl, or solid NaHSO ₄	14 days
1,2-dibromoethane (EDB)	2 x 40-mL glass vials with Teflon-lined septum caps	Cool to 4°C and adjust pH to less than 2 with HCL, Residual chlorine present: add Na ₂ S ₂ O ₃ to make 0.008% concentration	14 days
Polynuclear Aromatic Hydrocarbons (PAH)	Amber glass container with Teflon-lined lid	Cool to 4°C	7 days until extraction 40 days after extraction
Oxygenates ⁸	2 x 40-mL glass vials with Teflon-lined septum caps	Cool to 4°C and adjust pH to less than 2 with H ₂ SO ₄ , HCl, or solid NaHSO ₄	14 days
Total Petroleum Hydrocarbons (TPH) Waste Oil	1-Liter Glass	Cool to 4°C and adjust pH to less than 2 with H ₂ SO ₄ or HCl	28 days
Total Petroleum Hydrocarbons (TPH) (High Boiling Point Fuels)	1-Liter Amber Glass container with Teflon-lined lid	Cool to 4°C	7 days until extraction 40 days after extraction
Methane	2 x 40-mL glass vials with Teflon-lined septum caps	Cool to 4°C and adjust pH to less than 2 with 1:1 HCl	14 days
Dissolved Oxygen	G (bottle and top)	None required	Analyze immediately ⁵
Ferrous Iron	G	None required	24-48 hours
Nitrate	P, G	Cool to 4°C	48 hours
Sulfate	P, G	Cool to 4°C	28 days
Total Organic Carbon	P, G	Cool to 4°C H ₂ SO ₄ or HCl to pH 2	28 days
Mercury	P, G	HNO ₃ to pH < 2	28 days
Metals (except mercury) ⁶	P, G	HNO ₃ to pH < 2	6 months

¹ SW846, Third Edition, Updates I, II, IIA, III and IIIA, Table 4-1 Sample Containers, Preservation, Techniques, and Holding Times. 40 CFR Part 136, Table II, Required Containers, Preservation Techniques, and Holding Times.

² Polyethylene (P) or Glass (G).

³ Sample preservation must be performed during or immediately after sample collection.

⁴ Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.

⁵ Samples must be analyzed in the field at the time of collection.

⁶ Initial lead or other RCRA metal samples should not be filtered. If elevated levels of metals are detected, the UST project manager will direct the number and type of additional metal samples (filtered lead, organic lead).

⁷ BTEX (Benzene, toluene, ethylbenzene, and xylenes), MTBE (Methyl tert-butyl ether), Naphthalene, and 1,2-dichloroethane.

⁸ Oxygenates: ETBE(Ethyl tert-butyl ether), ETBA (ethyl tert-butyl alcohol), TAME (tertiary-amyl methyl ether), DIPE (diisopropyl ether), TBF (tert-butyl formate), TBA (tert-butyl alcohol), TAA (tert-amyl alcohol), and ethanol

Table 2
Required Containers, Preservation Techniques, Holding Times
Soil Samples¹

Contaminant	Container ²	Preservation ³	Holding Time ⁴
Volatiles ⁵ (BTEX and MTBE)	Amber glass container with Teflon-lined lid	See Method 5035 Cool to 4° C	14 days
Polynuclear Aromatic Hydrocarbons (PAH)	Amber glass container with Teflon-lined lid	Cool to 4° C Store in dark	14 days until extraction 40 days after extraction
Total Petroleum Hydrocarbons (TPH) Waste Oil	Glass wide-mouth container with Teflon-lined lid	Cool to 4° C HCl to pH < 2, when practical	Analyze as soon as possible
Total Petroleum Hydrocarbons (TPH) (High Boiling Point Fuels)	Amber Glass container with Teflon-lined lid	Cool to 4° C	14 days until extraction 40 days after extraction
Total Organic Carbon	P, G	Cool to 4° C	28 days
Mercury	P, G	None	28 days
Metals (except mercury)	P, G	None	6 months

¹ SW846, Third Edition, Updates I, II, IIA, and III, Table 4-1 Sample Containers, Preservation, Techniques, and Holding Times.

² Polyethylene (P) or Glass (G).

³ Sample preservation must be performed immediately after sample collection.

⁴ Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.

⁵ BTEX (Benzene, toluene, ethylbenzene, and xylenes) and MTBE (Methyl-tert-butyl ether).

Table 3
Analytical Methodology for Ground-Water Samples
Gasoline, Diesel, Fuel Oil, Kerosene

Contaminant	Analytical Method	Reference	Reporting Limit ⁶
Volatiles ⁵ (BTEX, MTBE, Naphthalene, and 1,2 DCA)	EPA Method 5030B with EPA Method 8260B	SW846 ¹	5 µg/L
1,2-dibromoethane (EDB)	EPA Method 8011	SW846 ¹	0.05 µg/L
Polynuclear Aromatic Hydrocarbons (PAH)	EPA Method 3510C ⁴ with EPA Method 8270C or EPA Method 8310	SW846 ¹	10 µg/L
Oxygenates ⁹	EPA Method 5030B with EPA Method 8260B	SW846 ¹	10 µg/L
Methane	Kerr Method	RSKSOP-175 ⁷	10,000 µg/L
Dissolved Oxygen	SM4500-O G	Standard Methods ²	1,000 µg/L
Ferrous Iron	SM3500-Fe D EPA Method 6010B ⁸	Standard Methods ² SW846 ¹	10 µg/L 20 µg/L
Lead ³	EPA Method 3005A or EPA Method 3010A	SW846 ¹	5 µg/L
Nitrate	EPA Method 9056A or EPA Method 9210	SW846 ¹	100 µg/L
Sulfate	EPA Method 9056A or EPA Method 9038	SW846 ¹	1000 µg/L
Total Organic Carbon (TOC)	EPA Method 9060	SW846 ¹	1 mg/L

¹ SW-846-3rd Edition, Updates I, II, IIA, and III, Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods. Superintendent of Documents, U.S. Government Printing Office, Washington, D.C.

² *Standard Methods for the Examination of Water and Wastewater*, 18th edition, 1992, American Public Health Association.

³ Initial lead or other RCRA metal samples should not be filtered. If elevated levels of metals are detected, the UST project manager will direct the number and type of additional metal samples (filtered lead, organic lead).

⁴ Other extractions methods may be used if the laboratory is certified for the extraction and determinative method.

⁵ BTEX (Benzene, toluene, ethylbenzene, and xylenes), MTBE (Methyl-tert-butyl ether), Naphthalene, and 1,2-dichloroethane.

⁶ Reporting Limit (RL): The Reporting Limit is the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. For many analytes, the Reporting Limit analyte concentration is selected as the lowest non-zero standard in the calibration curve. Sample Reporting Limits are highly matrix dependent.

⁷ Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples using a GC Headspace Equilibration Technique, EPA, Ada, OK, RSKSOP-175.

⁸ If field analysis of ferrous iron is not performed, laboratory analysis can be performed if sample is analyzed within 24 hours of sample collection. The method for laboratory analysis is SW-846 Method 6010B.

⁹ Oxygenates: ETBE(Ethyl tert-butyl ether) CAS #637-92-3, ETBA (ethyl tert-butyl alcohol) CAS #624-95-3, TAME (tertiary-amyl methyl ether) CAS #994-05-8, DIPE (diisopropyl ether) CAS #108-20-3, TBF (tert-butyl formate) CAS #762-75-4, TBA (tert-butyl alcohol) CAS #75-65-0, TAA (tert-amyl alcohol) CAS #75-85-4, and ethanol CAS #64-17-5

Table 4
Analytical Methodology for Soil Samples
Gasoline, Diesel, Fuel Oil, Kerosene

Contaminant	Analytical Method	Reference	Reporting Limit ⁴
Volatiles (BTEX and MTBE)	EPA Method 5035 with EPA Method 8260B	SW846 ¹	5 µg/kg
Polynuclear Aromatic Hydrocarbons (PAH)	EPA Method 3550C ³ with EPA Method 8270C or EPA Method 8310	SW846 ¹	660 µg/kg
Total Petroleum Hydrocarbons ² (TPH)-DROs (Diesel Range Organics)	EPA Method 3550C ³ with EPA Method 8015C	SW846 ¹	10 mg/kg
Lead	EPA Method 3050B with EPA Method 6010C or 6020A	SW846 ¹	250 µg/kg
Total Organic Carbon (TOC)	EPA Method 9060	SW846 ¹	0.1 mg/Kg ⁵

¹ SW-846-3rd Edition, Updates I, II, IIA, and III, Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods. Superintendent of Documents, U.S. Government Printing Office, Washington, D.C.

² Used for Soil Leachability Model only.

³ Other extractions methods may be used if the laboratory is certified for the extraction and determinative method.

⁴ Reporting Limit (RL): The Reporting Limit is the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. For many analytes, the Reporting Limit analyte concentration is selected as the lowest non-zero standard in the calibration curve. Sample Reporting Limits are highly matrix dependent.

⁵ The laboratory must use TOC instrumentation equipped for the analysis of soil samples.

Table 5
Analytical Methodology for Ground-Water Samples
Waste Oil

Contaminant	Analytical Method	Reference	Reporting Limit ⁶
Volatiles (BTEX)	EPA Method 5030B with EPA Method 8260B	SW846 ¹	5 µg/L
Total Petroleum Hydrocarbons (TPH)	EPA Method 9070A ⁵ with silica cleanup	SW846 ¹	5 mg/L
Polynuclear Aromatic Hydrocarbons (PAH)	EPA Method 3510C ³ with EPA Method 8270C or EPA Method 8310C	SW846 ¹	10 µg/L
Nitrate	EPA Method 9056 or EPA Method 9210	SW846 ¹	100 µg/L 1 mg/L
Sulfate	EPA Method 9056 or EPA Method 9038	SW846 ¹	1 mg/L 5 mg/L
Total Organic Carbon (TOC)	EPA Method 9060	SW846 ¹	1 mg/L
Dissolved Oxygen	SM4500-O G	Standard Methods ²	Not available
Ferrous Iron	SM3500-Fe D EPA Method 6010B ⁷	Standard Methods ² SW846 ¹	Not available 20 µg/L
Lead ⁴	EPA Method 7421	SW846 ¹	2 µg/L
Mercury	EPA Method 7470A	SW846 ¹	0.02 µg/L
Trace Metals: ⁴			
Arsenic	EPA Method 7060A	SW846 ¹	5 µg/L
Barium	EPA Method 6010B		50 µg/L
Cadmium	EPA Method 7131A		0.1 µg/L
Chromium	EPA Method 7191		5 µg/L
Selenium	EPA Method 7740		5 µg/L
Silver	EPA Method 7761		5 µg/L

¹SW-846-3rd Edition, Updates I, II, IIA, and III, Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods. Superintendent of Documents, U.S. Government Printing Office, Washington, D.C.

²*Standard Methods for the Examination of Water and Wastewater*, 18th edition, 1992, American Public Health Association.

³Other extractions methods may be used if the laboratory is certified for the extraction and determinative method.

⁴Initial lead or other RCRA metal samples should not be filtered. If elevated levels of metals are detected, the UST project manager will direct the number and type of additional metal samples (filtered lead, organic lead).

⁵This is the hexane extraction method. Please refer to 40 CFR Parts 136 and 260, "Guidelines Establishing Test Procedures for the Analysis of Oil & Grease and Non-Polar Material under the Clean Water Act and Resource Conservation and Recovery Act: Final Rule", dated May 14, 1999.

⁶ Reporting Limit (RL): The Reporting Limit is the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. For many analytes, the Reporting Limit analyte concentration is selected as the lowest non-zero standard in the calibration curve. Sample Reporting Limits are highly matrix dependent.

⁷ If field analysis of ferrous iron is not performed, laboratory analysis can be performed if sample is analyzed within 24 hours of sample collection. The method for laboratory analysis is SW-846 Method 6010B.

Table 6
Analytical Methodology for Soil Samples/Oil Samples
Waste Oil

Contaminant	Analytical Method	Reference	Reporting Limit ⁴
Volatiles (BTEX)	EPA Method 5035 with EPA Method 8260B	SW846 ¹	5 µg/kg
Polynuclear Aromatic Hydrocarbons (PAH)	EPA Method 3550B ³ with EPA Method 8270C or EPA Method 8310	SW846 ¹	330 µg/kg
Total Petroleum Hydrocarbons ² (Waste Oil)	EPA Method 9071B with Silica cleanup	SW846 ¹	250 mg/kg
Total Organic Carbon (TOC)	EPA Method 9060	SW846 ¹	----- ⁵
Mercury	EPA Method 7471B	SW846 ¹	10 µg/kg
Trace Metals: ⁴		SW846 ¹	
Arsenic	EPA Method 7060A		0.25 mg/kg
Barium	EPA Method 6010B		2.5 mg/kg
Cadmium	EPA Method 7131A		0.25 mg/kg
Chromium	EPA Method 7191		0.25 mg/kg
Lead	EPA Method 7421		0.25 mg/kg
Selenium	EPA Method 7740		0.25 mg/kg
Silver	EPA Method 7761		0.25 mg/kg

¹ SW-846-3rd Edition, Updates I, II, IIA, and III, Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods. Superintendent of Documents, U.S. Government Printing Office, Washington, D.C.

² Other extractions methods may be used if the laboratory is certified for the extraction and determinative method.

³ This is the hexane extraction method. Please refer to 40 CFR Parts 136 and 260, "Guidelines Establishing Test Procedures for the Analysis of Oil & Grease and Non-Polar Material under the Clean Water Act and Resource Conservation and Recovery Act: Final Rule", dated May 14, 1999.

⁴ Reporting Limit (RL): The Reporting Limit is the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. For many analytes, the Reporting Limit analyte concentration is selected as the lowest non-zero standard in the calibration curve. Sample Reporting Limits are highly matrix dependent.

⁵ The laboratory must use TOC instrumentation equipped for the analysis of soil samples.