# Soil sample collection and analysis procedures

## Petroleum Remediation Program

This document describes the procedures for field screening of petroleum-contaminated soil and collection of soil samples for laboratory analysis.

The Minnesota Pollution Control Agency (MPCA) Petroleum Remediation Program (PRP) conducts random on-site audits of fieldwork. Give the PRPat least 48 hours' notice prior to conducting fieldwork at sites under program oversight. Find information on the fieldwork notification process at https://www.pca.state.mn.us/waste/field-work-notifications. Prior notification of fieldwork is mandatory and will be verified upon submittal of the results.

To assure data quality, the U.S. Environmental Protection Agency (EPA) has required the MPCA to develop a Quality Assurance Program Plan (QAPP) for the PRP. The objective of the QAPP is to define the Quality Assurance/Quality Control (QA/QC) procedures to be followed for the collection and analysis of environmental samples. This ensures sufficient precision and accuracy of samples used in the PRP.

# I. Field screening procedures

## A. Soil headspace screening

Use the polyethylene bag headspace method described below to characterize soil contamination at petroleum release sites.

1. Use photoionization detectors (PIDs) with a 10.2 eV (+/-) or greater lamp source. Perform PID instrument calibration on site and at least daily to yield "total organic vapors" in parts per million by volume of a benzene equivalent. Follow the manufacturer's instructions for operation, maintenance, and calibration of the instrument. Keep calibration records in a bound book. MPCA staff reserve the right to request these records.
2. Use a resealable one-quart polyethylene freezer bag. Half-fill the bag with sample (the volume ratio of soil to air is equal), then immediately seal it. Manually break up the soil clumps within the bag. Note: Immediately after opening the split spoon sampler or soil sample liner, transfer soil to field screening bags. Collect soil samples from excavations or soil stockpiles from freshly exposed surfaces.
3. Allow headspace development for at least 10 minutes at approximate room temperature. Vigorously shake bags for 15 seconds at the beginning and end of the headspace development period. Headspace development decreases with temperature. When temperatures are below the operating range of the instrument, perform headspace development and screening in a heated vehicle or building. Record the ambient temperature during headspace screening. Complete headspace screening within approximately 20 minutes of sample collection.
4. After headspace development, introduce the instrument sampling probe through a small opening in the bag to a point about one-half of the headspace depth. Keep the probe free of water droplets and soil particles.
5. Record the highest meter response on a sampling form. Maximum response usually occurs within about two seconds. Erratic meter response may occur if high organic vapor concentrations or moisture is present. Note any erratic headspace data in the sampling form. Do not collect analytical samples from the polyethylene bag.

## B. Petroleum sheen test

The petroleum sheen test is a quick and easy field method used to determine if a soil sample is considered petroleum saturated. A soil is defined as petroleum-saturated when pore spaces contain some petroleum light non-aqueous phase liquid (LNAPL), with the remainder occupied by air and/or water. Detection of sheen or droplets of product when conducting this test is direct evidence that LNAPL is present and the soil sample is petroleum saturated.

1. Place a small quantity of petroleum-contaminated soil in a jar or on a large spoon. Alternatively, use the soil in the polyethylene bag after measuring the soil headspace described above.
2. Add enough water to break apart and submerge the soil particles. Add water directly to the polyethylene bag if using soil from headspace screening. Let the sample rest for a minimum of 10 minutes prior to examining for sheen or droplets of product.
3. If droplets of product or sheen are present on the water surface, the test result is positive and the soil is considered petroleum saturated.

# II. Soil sampling procedures

## A. Soil sampling - site characterization for investigation purposes

1. Minimize the possibility of cross-contamination by using disposable sampling equipment that is certified as clean for each sample collected. If disposable sampling tools are not available, specify the cleaning procedures used. Wear clean sampling gloves at each sampling point. When using a split-spoon or similar sampler, wash it with a detergent solution (e.g., Liquinox or equivalent), and rinse before each use.
2. When sampling excavation sidewalls or floors, remove at least one foot of exposed soil prior to collecting the sample to ensure collection of a fresh sample. See Excavation of petroleum contaminated soil for sampling requirements.
3. Collect samples from split-spoon samplers or soil sample liners using a procedure that will minimize losses due to volatilization. Collect samples as soon as possible after the surface of the soil has been exposed to the atmosphere. Do not collect analytical samples from soil cores that have been exposed for more than a few minutes. In order to allow for sample selection based on field screening results, immediately transfer an undisturbed portion of the soil coreto a resealable one-quart polyethylene freezer bag, sealed with no headspace, and placed on ice in a cooler. After the sampling interval has been determined, collect the analytical samplefrom the undisturbed bagged core sample. Complete sample containerization and preservation within two hours of retrieving the core from the subsurface. Document the procedure in the Investigation report. See Soil and groundwater assessments performed during site investigations for sampling requirements.
4. EPA Method 5035 is required when sampling soil for volatile contaminants per EPA SW-846. Collect soil samples using coring devices (e.g., cut syringe, EnCore™ , or US Analytical's Eazydraw Syringe™ sampler, or other approved coring device) and either put the "cored" soil directly into containers provided by the analytical laboratory (verify that the laboratory has pre-weighed these containers) or place the sealed coring device (for an EnCore™ type sampler) containing the soil in a cooler containing ice. The correct volume of soil to use in the coring device is established by weighing a similar soil sample before coring the analytical sample. Do not weigh analytical sample into the sample container because doing so can undesirably aerate the soil sample. The holding time is 14 days for soil samples preserved by methanol or frozen in an approved coring device. Samples in a coring device that are not frozen must be extracted within 48 hours. **Do not retain soil previously used for field screening or soil classification for analytical samples.**
    a) Gasoline range organics (GRO) and volatile organic compound (VOC) sampling: Collect GRO and VOC samples according to the Wisconsin Department of Natural Resources Modified GRO method and EPA Method 5035, respectively. These methods require the use of methanol as a preservative for most

sampling. When methanol is used as the preservative, GRO and VOC results can be obtained from the same sample. Preserve approximately 25 grams of soilwith 25 ml of methanol in a tared 60-ml vial. A maximum of 35 grams of soil is recommended to enable a 1:1 ratio of soil to extraction solvent in the sample container. Other sample sizes, such as 5 grams of soil and 5 mL of methanol in a 40-mL VOC vial, can be utilized if the 1:1 ratio is maintained. An approved sampler (e.g., EnCore™ or similar certified sampler) can also be used to hold the samples for 48 hours from the time of collection when held at 4 ± 2 degrees Celsius, or 14 days from the collection date when frozen below -12 degrees Celsius, before methanol preservation. A dry weight vial without methanol preservation is also required for every sample. Clean the vial threads to assure a good seal with the cap provided.

b) Diesel range organics (DRO) sampling: Collect DRO samples according to the Wisconsin Department of Natural Resources Modified DRO method. Place approximately 25 grams of soil in a tared 60-ml vial without preservative. A maximum of 35 grams of soil is recommended to enable a 1:1 ratio of soil to extraction solvent in the sample container. An approved sampler can also be used to hold the sample until it is extracted following the Wisconsin DRO protocols. Collect another vial for dry weight determination on the sample. Clean the vial threads to assure a good seal with the cap provided. Add methylene chloride to the DRO samples within 10 days. The laboratory must complete the extraction process and sample analysis within 47 days of collection.

5. Label all vials, place in a covered cooler with ice, and transport to the laboratory for analysis. Samples should be kept at a stable temperature of 4 ± 2 degrees Celsius. Include a container of water for sample temperature verification (temperature blank). The labels should indicate:
   a) Type of analysis
   b) Name of facility
   c) Monitoring point identification
   d) Name of person collecting sample
   e) Time and date the sample was collected
   f) Name of preservative added, if applicable
6. Collect, transport, and deliver samples under chain of custody.
7. Samples not transported or analyzed within the accepted holding time are invalid.

## B. Soil sampling – characterization for off-site treatment/disposal

1. Analysis for VOCs, GRO, and DRO requires collection of grab samples from representative portions of the stockpile or from soil borings conducted in locations that are representative of soil contaminated by the release. Use the sampling procedures described in subsection A above. Base the number of soil samples on Table A.

**Table A. Number of grab samples required from contaminated soil stockpiles.**

| Cubic yards of soil in pile | Number of grab samples |
|---|---|
| Less than 50 | 1 |
| 51-500 | 2 |
| 501-1,000 | 3 |
| 1,001-2,000 | 4 |
| 2,001-4,000 | 5 |
| Each additional 2,000 | One additional sample |

Analysis of soil samples is not normally necessary if less than 10 cubic yards of contaminated soil will be land treated, unless the soil could potentially be considered a hazardous waste.

2. Analysis for metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver) and polychlorinated biphenyls (PCBs) requires collection of a separate composite sample. To take a composite sample from a stockpile, collect 15 samples from randomly selected locations within the stockpile, place samples in a clean container, mix thoroughly, and remove a single subsample. To take a composite sample from soil borings, collect 15 samples from randomly selected locations in borings that represent soil that will be excavated. Place samples in a clean container, mix thoroughly, and remove a single subsample. In each case, send the single subsample to the laboratory for analysis.

# III. Soil analysis

The MPCA requires laboratory certification. See the MPCA quality system webpage and Methods and analytes requiring laboratory certification. Laboratories reporting data are required to include the name of the certifying organization on analytical reports. Certification is currently required for fixed-base and mobile laboratories analyzing VOCs. Certification for mobile labs must meet the same requirements established for fixed-base laboratories.

If a noncertified mobile laboratory is used for sample screening, split ten percent of samples with a certified fixed-base laboratory. An MPCA-recognized certified laboratory must generate data supporting site closure. In appendices and tables, clearly label data generated by mobile laboratories and fixed-base laboratories. For all sample analyses, unless otherwise noted in this document, use an EPA-approved method or equivalent. Provice chromatograms for positive GRO and DRO analyses. These chromatograms must be properly scaled in order to show enough detail to allow for interpretation and identification.

Table B lists the analyses for the different types of petroleum products.

**Table B. Required analyses and laboratory procedures.**

| Petroleum product | Parameters |
|---|---|
| Leaded gasoline, aviation gasoline | A, B, D |
| Unleaded gasoline, ethanol-blended fuel | A, B |
| Unused petroleum products: fuel Oil, motor Oil, diesel Fuel, kerosene, jet fuels | A, C, G |
| Used oil (e.g., motor oil, other used petroleum products). See notes 1, 2, and the section on used oil special considerations below. | A, C, E, F, G |
| Unknown petroleum or hydrocarbons mixture | A, B, C, E, F, G |
| Other petroleum products | Site specific |
| Hydraulic fluids | A, C, F, G |

A. Volatile organic compounds (VOCs) by the most recent version of EPA Method 8260 (see Note 6). Find the VOC target analyte list in Appendix A.

   If groundwater at the site will be analyzed for VOCs, then analyze soil for only petroleum VOCs (PVOCs) by the most recent version of EPA Method 8260 or Method 8021. PVOC analysis QA/QC procedures are in Section IV. The PVOC target analyte list is in Appendix B.

B. Wisconsin Department of Natural Resources Modified Gasoline Range Organics (GRO) Method (see Note 3)

C. Wisconsin Department of Natural Resources Modified Diesel Range Organics (DRO) Method (see Notes 4 and 5)

D. Lead (see Note 7)

E. Resource Conservation and Recovery Act (RCRA) metals - arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver (see Note 2)

F. Polychlorinated biphenyls (PCBs) using the most recent version of EPA Method 8082 by the Aroclors method (see Note 2). See Appendix C for specific Aroclors. Complete analysis for PCBs for hydraulic fluids used in elevators and other hydraulic fluids subject to high heat prior to the 1980s.

G. Polycyclic aromatic hydrocarbons (PAHs) by the most recent version of EPA Method 8270. PAH analysis QA/QC procedures are found in Section IV. The PAH target analyte list is found in Appendix D.

Note that the MPCA project manager will determine the need for PAH analysis. If a drinking water aquifer is impacted by fuel oil or higher boiling point petroleum product contact the project manager.

**Notes:**

1. Do not confuse used oil with waste oil. **Used oil** means any oil that (because of use) is contaminated by physical or chemical impurities. Examples of used oil include, but are not limited to, motor oils, metal cutting oils, and hydraulic fluids. **Waste oil** means oil that is discarded or spilled before use.
2. During investigation at used oil sites, collect samples for all of the parameters listed (VOCs, DRO, RCRA metals, and PCBs), but direct your laboratory to analyze only the VOC and DRO samples initially. If any of these compounds are detected, proceed with analysis of the metals and PCB samples. Note: If you are sampling to fulfill soil disposal requirements, analyze samples for all required parameters (VOCs, DRO, metals, and PCBs). See below for special considerations for soil contaminated with used oil.
3. This is a purge-and-trap, gas chromatography (GC) procedure that uses a 10-component blend of gasoline compounds for the quantification standard. Collect approximately 25 grams of soil using a zero headspace method and placed into a tared 60-mL vial containing 25 mL of methanol followed by measuring the total weight in the laboratory. The sample must be cooled to 4 ± 2 degrees Celsius and must be received by the laboratory within four days (EnCore™ samplers can be held for 48 hours) and analyzed within 21 days of collection. To ensure the extraction of the volatiles, the soil must be sonicated for 20 minutes (ensure the temperature does not exceed room temperature) before samples are analyzed. Use a methanol trip blank when using methanol as a preservative as this ensures no contamination is present in the methanol. The method quantitation limit shall be no more than 10 mg/kg dry weight, assuming 100% solids.
4. This is a solvent extraction, direct injection, GC procedure that uses a ten-component blend of typical diesel oil components for a quantification standard. Collect approximately 25 grams of soil using a zero headspace method and placed into a tared 60-mL vial followed by measuring the total weight in the laboratory. The samples must be cooled to 4 ± 2 degrees Celsius, received by the laboratory and preserved in solvent within 10 days, and analyzed within 47 days of collection. The method quantitation limit shall be no more than 10 mg/kg dry weight, assuming 100% solids. Separate samples are required for GRO and DRO analyses.
5. The DRO analysis method can have false positives that lead to elevated results, which are not from petroleum compounds. The DRO analysis method is a very useful screening method, but may not provide an accurate determination of petroleum compounds for making site decisions. When false positives are suspected, cleanup of extracts to remove non-petroleum compounds may be necessary. See Section IV for laboratory QA/QC procedures for DRO cleanup.
6. This is a purge-and-trap, gas chromatography method. Sampling is similar to GRO (see Note 3). Base the reporting limits and quality assurance for all parameters on the most recent version of EPA Method 8260. Note that PVOC holding time is 14 days and must be sampled using EPA Method 5035. If a mobile laboratory is being used for the analysis of soil samples, the mobile laboratory must follow the method specified calibration and quality control procedures required by the referenced method.
7. Sampling for lead is required for fulfilling soil disposal requirements. Specifically, soil that will be treated after its removal that is actually or potentially contaminated with leaded gasoline must be analyzed to determine if it exhibits the toxicity characteristic for lead. If a total lead analysis indicates a level equal to or greater than 100 mg/kg, perform a Toxicity Characteristic Leaching Procedure (TCLP). If the TCLP level exceeds 5 mg/L, manage the soil as a hazardous waste in accordance with Minn. R. ch. 7045.

**Special considerations for soil contaminated with used oil:**
This section outlines additional procedures for testing used oil contaminated soil to determine if the soil is considered a hazardous waste. If the soil is considered a hazardous waste, manage the soil in accordance with Minn. R. ch. 7045.

1. Soil with a PCB concentration above 50 mg/kg is considered a hazardous waste.
2. Soil is considered a hazardous waste if it is contaminated with any hazardous waste listed in Minn. R. 7045.0135. If the generator of the soil can certify to the satisfaction of MPCA staff, by previous knowledge or chemical analysis, that the soil is not contaminated with waste listed in Minn. R. 7045.0135 the soil need not be managed as a hazardous waste.
3. If the concentration of total halogenated compounds determined by VOC analysis is greater than 1,000 mg/kg, the soil is presumed to be hazardous waste unless the generator can rebut this presumption to the satisfaction of MPCA staff, through previous knowledge or chemical analysis.
4. Soil is considered hazardous if it exhibits the toxicity characteristic of any of the following contaminants: arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver, endrin, lindane, methoxychlor, toxaphene, 2,4-dichlorophenoxyacetic acid, and 2,4,5-trichlorophenoxypropionic acid. Do not test for contaminants known not to be present, such as pesticides and herbicides, if the generator certifies to the satisfaction of the MPCA that the contaminants are not present. Contaminants exceeding levels in Table C are considered to potentially exhibit the toxicity characteristic and a complete TCLP must be performed:

**Table C. Levels at which TCLP will be required**

| Contaminant | Soil concentration (mg/kg) |
|---|---|
| Arsenic | 100 |
| Barium | 2,000 |
| Cadmium | 20 |
| Chromium | 100 |
| Lead | 100 |
| Mercury | 4.0 |
| Selenium | 20 |
| Silver | 100 |
| Endrin | 0.40 |
| Lindane | 8.0 |
| Methoxychlor | 200 |
| Toxaphene | 10 |
| 2,4-Dichlorophenoxyacetic Acid | 200 |
| 2,4,5-Trichlorophenoxypropionic Acid | 20 |

If soil exhibits the toxicity characteristic, it is considered a hazardous waste.

# IV. Required laboratory quality assurance/quality control

**A. PVOC analysis:** Analyze samples for the target analytes listed in Appendix B using the most recent version of EPA Method 8260 or Method 8021. Follow all quality control (QC) elements defined in the method. Laboratories should also incorporate the QC procedures listed below.

1. **Initial calibration**: The initial calibration curve must contain at least five calibration points. For Methods 8260 and 8021, the $r^2$ for each curve must be greater than or equal to 0.990, or the r must be greater than or equal to 0.995. If the ratio of response to concentration is constant (<15% relative standard deviation for Method 8260 and <20% relative standard deviation for Method 8021), linearity can be assumed and the average response factor can be used in place of a calibration curve. The recovery (accuracy) for each point in

the curve must be 70% to 130% except for the lowest point in the curve, which must be 60% to 140%. The lowest calibration point in the curves shall be at or below the analyte report level. If a sample concentration exceeds the highest calibration standard from the initial calibration, dilute the sample into the calibration range and re-analyzed.

2. **Continuing calibration verification**: Analyze one low-level standard at the report level (RL) and one mid-level calibration verification standard prior to the samples. Bracket the batch of samples with a second mid-level calibration verification standard (in each 12-hour period, up to 20 environmental samples can be analyzed between standards). The percent recovery (%R) for the target analytes in the low-level standard should be between 60% and 140% of the true value. The %R for the target analytes in the mid-level standards should be between 70% and 130% of the true value (with a %D of less than or equal to 30%).
3. **Initial demonstration of capability**: Analyze 4-7 replicate mid-level check standards. Percent recovery (%R) must be equal to 70-130%. The percent relative standard deviation (%RSD) must be less than 20%.
4. **Method detection limit/report level**: Method detection limits (MDLs) and RLs are determined annually or after a major change to the instrument conditions. The MDLs are determined per the procedure defined in 40 CFR 136, Appendix B. Analyze a minimum of seven standards. The RL should be approximately three to five times the MDL. The lowest calibration point in the curves shall be at or below the analyte report level. If the accuracy of the RL standard does not meet the 60% to 140% criteria, a new RL standard is chosen and analyzed until the accuracy criteria are met.
5. **Batch quality control:** A batch is defined as up to 20 environmental samples analyzed in a 12-hour analytical sequence. At a minimum, each batch must contain a method blank, a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate (MS/MSD) pair. If there is not enough sample to prepare and analyze a MS/MSD pair, a laboratory control sample duplicate (LCSD) is prepared and analyzed.
6. **Method blanks**: Analyze one method blank per QC batch of 20 samples or less. The concentration of PVOCs in the method blank must be less than the associated report level. If the method blank is contaminated, take measures to eliminate the problem. Re-extract and re-analyze affected samples. If the contamination cannot be eliminated, the results must be qualified to indicate the problem. All concentration levels for the affected target analyte that are less than ten times the concentration in the blank should be qualified with a "B" to indicate that the sample results may contain a bias related to the blank contamination. Concentrations of the affected analyte that are above ten times the blank contamination will not need to be qualified.
7. **Accuracy/precision**: One LCS is required per batch. The %R must be between 70% and 130%. One MS and MSD is required per batch. The %R for each analyte in the MS/MSD must be between 70% and 130% with a relative percent difference (RPD) of less than or equal to 30%.
8. **Holding time**: Analyze the samples within 14 days of sample collection.
9. **Confirmation analysis**: For Method 8021, confirmation analysis using a dissimilar detector or a column of different polarity must be performed, or by GC/mass spectrometry analysis. Evaluate the agreement between the quantitative results by calculating the relative percent difference (RPD) between the two results. The formula is:

   RPD = (ABS(R1 – R2)/((R1 + R2)*2))*100

   The RPD should be ≤40%. If one result is significantly higher, check the chromatograms to see if an obviously overlapping peak is causing the high result. If no overlapping peaks are noted, examine the baseline to determine if there were any data system problems during peak integration.

   If no anomalies are noted, report the higher result and add a flag that alerts the data user of the disparity between the results on the two detectors or columns.

**B. PAH analysis:** Analyze samples for these target analytes in Appendix D using the most recent version of EPA Method 8270. 1-Methylnaphthalene and 2-methylnaphthalene are included in the list of PAH target analytes for the PRP. Follow all QC elements defined in the method. Laboratories should also incorporate the QC procedures listed below.

1. **Initial calibration**: The initial calibration curve should contain at least five calibration points. The r for each curve must be greater than or equal to 0.995. If the ratio of response to concentration is constant (<20% relative standard deviation), linearity can be assumed and the average response factor can be used in place of a calibration curve. The recovery (accuracy) for each point in the curve must be 70% to 130% except for the lowest point in the curve, which must be 60% to 140%. The lowest calibration point in the curves shall be at or below the analyte report level. If a sample concentration exceeds the highest calibration standard from the initial calibration, dilute the sample into the calibration range and re-analyze.
2. **Continuing calibration verification**: Analyze one low-level standard at the report level (RL) and one mid-level calibration verification standard prior to the samples. Bracket the batch of samples with a second mid-level calibration verification standard (in each 12-hour period, up to 20 environmental samples can be analyzed between standards). The percent recovery (%R) for the target analytes in the low-level standard should be between 60% and 140% of the true value. The %R for the target analytes in the mid-level standards should be between 80% and 120% of the true value (with a %D of less than or equal to 20%).
3. **Initial demonstration of capability**: Analyze 4-7 replicate mid-level check standards. Percent recovery (%R) must be equal to 70-130%. The percent relative standard deviation (%RSD) must be less than 20%.
4. **Method detection limit/Report level**: Method detection limits (MDLs) and Ls are determined annually or after a major change to the instrument conditions. The MDLs are determined per the procedure defined in 40 CFR 136, Appendix B. Analyze a minimum of seven standards. The RL should be approximately three to five times the MDL. The lowest calibration point in the curves shall be at or below the analyte report level. If the accuracy of the RL standard does not meet the 60% to 140% criteria, a new RL standard is chosen and analyzed until the accuracy criteria are met.
5. **Batch QC**: A batch is defined as up to 20 environmental samples prepared in the same 24-hour period. At a minimum, each batch must contain a method blank, a LCS, and a MS/MSD pair. If there is not enough sample to prepare and analyze a MS/MSD pair, prepare and analyze an LCSD.
6. **Method blanks**: Analyze one method blank per QC batch of 20 samples or less. The concentration of PAHs in the method blank must be less than the associated report level. If the method blank is contaminated, take measures to eliminate the problem. Re-extract and re-analyze affected samples. If the contamination cannot be eliminated, the results must be qualified to indicate the problem. All concentration levels for the affected target analyte that are less than ten times the concentration in the blank should be qualified with a “B” to indicate that the sample results may contain a bias related to the blank contamination. Concentrations of the affected analyte that are above 10 times the blank contamination will not need to be qualified.
7. **Accuracy/precision**: One LCS is required per batch. The laboratory should generate in-house limits for accuracy. The % recoveries should be between 50% and 150%. One MS and MSD is required per batch. The laboratory should generate in-house limits for accuracy and precision. The % recoveries for each analyte in the MS/MSD should be between 50% and 150% with a RPD of less than or equal to 30%.
8. **Surrogates**: Surrogates are added to all environmental and QC samples. The laboratory should generate in-house limits for surrogate recoveries. The % recoveries should be between 50% and 150%.
9. **Holding time**: The samples should be extracted within 7 days of collection and analyzed within 40 days of extraction.

**C. DRO cleanup:** Clean up soil extracts prior to DRO analysis using three methods, either separately or in combination. The methods include: 1) EPA Method 3630C, silica gel cleanup, 2) EPA Method 3650B, acid/base partitioning, and 3) EPA Method 3611B, alumina column cleanup. When DRO cleanup is requested, pre-cleanup analysis should be completed prior to conducting the cleanup. If there are no detections in the pre-cleanup analysis, the laboratory should not conduct the cleanup and post-cleanup analysis.

DRO clean-up results are accepted only if quality assurance documentation shows results that meet acceptance criteria, i.e. no significant loss of petroleum compounds. The quality control acceptance criteria for the clean-up results will be as follows:

1. At least one method blank, one laboratory duplicate, one matrix spike, and one laboratory control sample must be run through the complete clean-up and analysis process for up to 20 samples (1/20).
2. The laboratory must provide a narrative of the entire clean up and analysis process. They must also provide pre- and post-cleanup results and compare the pre-cleanup chromatogram with that of the post-cleanup chromatogram for every sample when cleanup is completed.
3. The RPD between sample duplicates, matrix spike pairs, or laboratory control sample pairs must be less than or equal to 20%. However, use the difference between duplicates to measure precision if the concentration of the target analyte in the sample/sample duplicate is less than five times the report level. The difference must then be less than or equal to the report level.

# Appendix A: Target analyte list for volatile organic compounds

| Chemical name | CAS # | Report level (mg/kg dry weight)* |
|---|---|---|
| 1,1,1,2-Tetrachloroethane | 630-20-6 | 1.0 |
| 1,1,1-Trichloroethane | 71-55-6 | 1.0 |
| 1,1,2,2-Tetrachloroethane | 79-34-5 | 1.0 |
| 1,1,2-Trichloroethane | 79-00-5 | 1.0 |
| 1,1,2-Trichlorotrifluoroethane | 76-13-1 | 1.0 |
| 1,1-Dichloroethane | 75-34-3 | 1.0 |
| 1,1-Dichloroethene | 75-35-4 | 1.0 |
| 1,1-Dichloropropene | 563-58-6 | 1.0 |
| 1,2,3-Trichlorobenzene | 87-61-6 | 1.0 |
| 1,2,3-Trichloropropane | 96-18-4 | 1.0 |
| 1,2,4-Trichlorobenzene | 120-82-1 | 1.0 |
| 1,2,4-Trimethylbenzene | 95-63-6 | 1.0 |
| 1,2-Dibromo-3-chloropropane | 96-12-8 | 5.0 |
| 1,2-Dibromoethane | 106-93-4 | 1.0 |
| 1,2-Dichlorobenzene | 95-50-1 | 1.0 |
| 1,2-Dichloroethane | 107-06-2 | 1.0 |
| 1,2-Dichloropropane | 78-87-5 | 1.0 |
| 1,3,5-Trimethylbenzene | 108-67-8 | 1.0 |
| 1,3-Dichlorobenzene | 541-73-1 | 1.0 |
| 1,3-Dichloropropane | 142-28-9 | 1.0 |
| 1,4-Dichlorobenzene | 106-46-7 | 1.0 |
| 2,2-Dichloropropane | 594-20-7 | 1.0 |
| 2-Chlorotoluene | 95-49-8 | 1.0 |
| 4-Chlorotoluene | 106-43-4 | 1.0 |
| Acetone | 67-64-1 | 20 |
| Allyl chloride | 107-05-1 | 1.0 |
| Benzene | 71-43-2 | 1.0 |
| Bromobenzene | 108-86-1 | 1.0 |
| Bromochloromethane | 74-97-5 | 1.0 |
| Bromodichloromethane | 75-27-4 | 1.0 |
| Bromoform | 75-25-2 | 1.0 |

| Chemical name | CAS # | Report level (mg/kg dry weight)* |
|---|---|---|
| Bromomethane | 74-83-9 | 2.0 |
| n-Butylbenzene | 104-51-8 | 1.0 |
| sec-Butylbenzene | 135-98-8 | 1.0 |
| tert-Butylbenzene | 98-06-6 | 1.0 |
| Carbon tetrachloride | 56-23-5 | 1.0 |
| Chlorobenzene | 108-90-7 | 1.0 |
| Chlorodibromomethane | 124-48-1 | 1.0 |
| Chloroethane | 75-00-3 | 1.0 |
| Chloroform | 67-66-3 | 1.0 |
| Chloromethane | 74-87-3 | 1.0 |
| cis-1,2-Dichloroethene | 156-59-2 | 1.0 |
| cis-1,3-Dichloropropene | 10061-01-5 | 1.0 |
| Dibromomethane | 74-95-3 | 1.0 |
| Dichlorodifluoromethane | 75-71-8 | 1.0 |
| Dichlorofluoromethane | 75-43-4 | 1.0 |
| Ethylbenzene | 100-41-4 | 1.0 |
| Ethyl ether | 60-29-7 | 1.0 |
| Hexachlorobutadiene | 87-68-3 | 1.0 |
| Isopropylbenzene | 98-82-8 | 1.0 |
| p-Isopropyltoluene | 99-87-6 | 1.0 |
| Methyl ethyl ketone (2-butanone) | 78-93-3 | 10 |
| Methyl isobutyl ketone (4-methyl-2-pentanone) | 108-10-1 | 5.0 |
| Methyl *tertiary*-butyl ether | 1634-04-4 | 2.0 |
| Methylene chloride | 75-09-2 | 2.0 |
| Naphthalene | 91-20-3 | 1.0 |
| n-Propylbenzene | 103-65-1 | 1.0 |
| Styrene | 100-42-5 | 1.0 |
| Tetrachloroethene | 127-18-4 | 1.0 |
| Tetrahydrofuran | 109-99-9 | 10 |
| Toluene | 108-88-3 | 1.0 |
| trans-1,2-Dichloroethene | 156-60-5 | 1.0 |
| trans-1,3-Dichloropropene | 10061-02-6 | 1.0 |
| Trichloroethene | 79-01-6 | 1.0 |
| Trichlorofluoromethane | 75-69-4 | 1.0 |
| Vinyl chloride | 75-01-4 | 1.0 |
| m&p-Xylene | 179601-23-1 | 1.0 |
| o-Xylene | 95-47-6 | 1.0 |

*Assuming 100% solids

# Appendix B: Target analyte list for petroleum volatile organic compounds

| Chemical name | CAS # | Report level (mg/kg dry weight)* |
|---|---|---|
| 1,2,4-Trimethylbenzene | 95-63-6 | 1.0 |
| 1,3,5-Trimethylbenzene | 108-67-8 | 1.0 |
| Benzene | 71-43-2 | 1.0 |
| Ethylbenzene | 100-41-4 | 1.0 |
| Methyl *tertiary*-butyl ether | 1634-04-4 | 2.0 |

| Chemical name | CAS # | Report level (mg/kg dry weight)* |
|---|---|---|
| Naphthalene | 91-20-3 | 1.0 |
| Toluene | 108-88-3 | 1.0 |
| m&p-Xylene | 179601-23-1 | 1.0 |
| o-Xylene | 95-47-6 | 1.0 |

*Assuming 100% solids

# Appendix C: Target analyte list for polychlorinated biphenyls

| Chemical name | CAS # | Report level (mg/kg dry weight)* |
|---|---|---|
| Aroclor 1016 | 12674-11-2 | 0.050 |
| Aroclor 1221 | 11104-28-2 | 0.100 |
| Aroclor 1232 | 11141-16-5 | 0.050 |
| Aroclor 1242 | 53469-21-9 | 0.050 |
| Aroclor 1248 | 12672-29-6 | 0.050 |
| Aroclor 1254 | 11097-69-1 | 0.050 |
| Aroclor 1260 | 11096-82-5 | 0.050 |

*Assuming 100% solids

# Appendix D: Target analyte list for polycyclic aromatic hydrocarbons

| Chemical name | CAS # | Report level (mg/kg dry weight)* |
|---|---|---|
| Acenaphthene | 83-32-9 | 0.17 |
| Acenaphthylene | 208-96-8 | 0.17 |
| Anthracene | 120-12-7 | 0.17 |
| Benzo(a)anthracene | 56-55-3 | 0.17 |
| Benzo(b)fluoranthene | 205-99-2 | 0.17 |
| Benzo(k)fluoranthene | 207-08-9 | 0.17 |
| Benzo(g,h,i)perylene | 191-24-2 | 0.17 |
| Benzo(a)pyrene | 50-32-8 | 0.17 |
| Chrysene | 218-01-9 | 0.17 |
| Dibenz(a,h)anthracene | 53-70-3 | 0.17 |
| Fluoranthene | 206-44-0 | 0.17 |
| Fluorene | 86-73-7 | 0.17 |
| Indeno(1,2,3-cd)pyrene | 193-39-5 | 0.17 |

| Chemical name | CAS # | Report level (mg/kg dry weight)* |
|---|---|---|
| 2-Methylnaphthalene | 91-57-6 | 0.17 |
| Naphthalene | 91-20-3 | 0.17 |
| Phenanthrene | 85-01-8 | 0.17 |
| Pyrene | 129-00-0 | 0.17 |
| 1-Methylnaphthalene | 90-12-0 | 0.17 |

*Assuming 100% solids