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Appendix A

Laboratory Analytical Methods

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1.0 Laboratory Analytical Methods

Laboratory analytical methods must be selected to accurately reflect types and concentrations of substances present, or suspected to be present, at a release site. This section presents guidance on selecting the appropriate analytical methods to provide OIS technical staff with the necessary data to evaluate the nature, extent, and magnitude of contaminants at petroleum storage tank sites. A discussion of each hydrocarbon compound class is included below.

Table 1 provides guidance on the appropriate analytical methods to use. Methods must be selected based on the need to measure contaminants at or below the lowest of the applicable cleanup levels. The target method detection limits are necessary for measurement of the most restrictive cleanup criteria; however, other method detection limits may be acceptable for other criteria. In some situations, a number of analytical methods are listed for those cases where more than one method is appropriate to allow flexibility in the selection and use of analytical methods.

Methods listed below are from EPA SW-846, *Test Methods for Evaluating Solid Waste*, Third Update to the Third Edition. Methods from previous versions of SW-846 may be accepted and other methods not listed in SW-846 may be used, however they should be preapproved by the OIS project manager.

Table 1: Acceptable Analytical Methods for Common Petroleum Products.

Petroleum Product	Soil	Water
Gasoline	8015B modified (TVPH) ¹	8015B modified (TVPH) ¹
Diesel, Jet Fuel, Kerosene, Fuel Oils	8015B modified (TEPH) ¹ , 418.1	8015B modified (TEPH) ¹ , 418.1
BTEX & MTBE	8021, 8020, 8260	8021, 8020, 8260
Waste/Lubricating Oils	8015B modified (TEPH) ¹ , 1664, 418.1, 413.1	8015B modified (TEPH) ¹ , 1664, 418.1, 413.1
Polynuclear Aromatic Hydrocarbons (PAHs)	8270, 8310, 8100	8270, 8310, 8100
Glycols	8015 modified (Glycols) ¹	8015 modified (Glycols) ¹

1. When utilizing the 8015 modified TEPH method for extractable hydrocarbons, the GC column must be standardized for the particular hydrocarbon range of concern (e.g., diesel, kerosene, motor oil, etc.)

1.1 Gasoline Range Organics

Parameters for investigation and cleanup verification of releases from tanks known to contain gasoline range organics (gasoline, stoddard solvent, etc.) include BTEX, total volatile petroleum hydrocarbons (TVPH), and polynuclear aromatic hydrocarbons (PAHs). Characterization and cleanup of releases from gasoline tanks are based primarily on the aromatic volatiles benzene, toluene, ethylbenzene, and xylene (BTEX). BTEX is particularly well suited for evaluating gasoline releases to groundwater since these compounds are among the most soluble gasoline constituents. PAH compounds are occasionally present at low concentrations in soils associated with gasoline releases, however, site closure is nearly always based on other compounds such as BTEX.

1.2 Fuel and Diesel Oils

Parameters for investigation and cleanup verification of light distillate fuel and diesel oil releases (kerosene, fuel oils 1 and 2, diesel oils 1-D and 2-D, and jet fuels JP-4, JP-7, JP-8, and Jet A) include BTEX, total extractable hydrocarbons (TEPH), and PAHs. BTEX compounds (with the exception of JP-4) are not particularly good monitoring parameters for these light distillate oils since they are present in only trace amounts or absent. PAHs are better monitoring compounds since they range from low levels to approximately 5%. If target analytes are not present and further cleanup is necessary to eliminate aesthetic impacts in groundwater or soil, the TEPH target method detection limits may be used in an evaluation to achieve closure.

1.3 New and Used Lubricating Oils

Parameters for investigation and cleanup verification of new or used oil releases initially include BTEX, Oil & Grease, and PAHs. Additionally, for waste oil tanks, a minimum of one sample (the sample with the highest observable or measured contamination) should be analyzed for volatile organics, TCLP cadmium, chromium, and lead, and PCBs. Since nearly any type of waste could conceivably be mixed with "waste oil", this parameter list is by no means fixed. Other parameters can and should be added consistent with information about the source of the waste. Other metals, cyanide, sulfide, chlorinated hydrocarbons (extractable), and other compounds may be present. Table 2 presents a listing of additional analytical methods for samples related used oil tanks.

Methods listed below are from EPA SW-846, *Test Methods for Evaluating Solid Waste*, unless otherwise noted. The most current version of each method listed shall be utilized. Other methods may be used, however they shall be preapproved by the OIS project manager

Table 2: Supplemental Analytical Methods for Used Oil Tank Samples.

Parameter	Water	Soil
Volatile Organics	8260	8260
Semi-volatile Organics/PAHs	8270	8270
Cadmium	6010, 6020, 7131	6010, 6020, 7131
Chromium	6010, 6020, 7191	6010, 6020, 7191
Lead	6010, 6020, 7421	6010, 6020, 7421
PCBs	8082, 1656	8082, 1656

1.4 Petroleum Solvents

Parameters for investigation and cleanup of releases from tanks known to contain petroleum solvents include TVPH, Volatile Organic Compounds (VOCs), and PAHs. Petroleum solvents do not usually contain sufficient quantities of target analytes (BTEX and PAHs) to allow their measurement in soil and water samples. If, after initial analysis, the target analytes are not detected, the measurement of petroleum solvents may be based on the total volatile petroleum hydrocarbons (TVPH) procedure, which quantifies all chromatographic peaks between 2-methyl pentane and 1,2,4-trimethylbenzene.

1.5 Unknown Petroleum Products

In the case of developing a monitoring strategy for unknown petroleum products, a phased sampling/analytical approach is often valuable. It is recommended that the sample with the highest apparent contamination, as evaluated in the field, be analyzed first for TVPH and TEPH to determine the range of organics. The second phase would include Volatile organics, PAH compounds, volatile halocarbons, and TCLP cadmium, chromium, and lead.

For sites where the above compounds are not detected, and aesthetic impacts to soil or groundwater are present, both TVPH and TEPH methods should be performed. These non-specific test procedures are useful for those petroleum products which may not contain measurable quantities of BTEX or PAH compounds. Target MDL criteria or aesthetic criteria are acceptable for demonstrating cleanup.

1.6 Glycols

The analytical parameter for investigation and cleanup of releases from tanks known to contain glycols is TVPH by EPA Method 8015 modified (glycols).

1.7 Other Products

In the case of other petroleum products or additives where the product name or chemical name is known, the parameters should be selected based on the information about the product such as might be contained in product labeling or Material Safety Data Sheets. Material Safety Data Sheets often provide useful information for selecting the appropriate

analytical method.

2.0 Laboratory Quality Control

The responsibility for conducting a valid investigation and completing an appropriate site closure rests with the consultant or project manager and ultimately with the responsible party. The validity of the investigation hinges on having data of known and acceptable quality. Data should be evaluated periodically throughout the course of the investigation and cleanup to assure validity. In addition, data should be evaluated whenever the need to review data is suggested by inconsistent split sample data, suspected contamination or carry over, poor agreement between field and lab data, unusual data, apparent outliers or data which is otherwise inconsistent with other existing site data.

Analytical quality control procedures include the routine use of method blanks as a check on contamination, calibration checks or control samples (preferably at two concentrations) to verify calibration in each run, the use of sample spikes and duplicates to verify sample recovery and reproducibility, and surrogate spikes in every sample to verify the entire analytical procedure from preparation through analysis. With every batch (a group of samples analyzed sequentially) of 20 or fewer samples analyzed, the lab must also analyze certain quality control samples, and meet specific performance requirements as outlined below.

2.1 Method Blanks

A method blank must be analyzed with every batch of samples. All target analytes must be below method detection limits. When detection limits are exceeded in the method blank, sample analysis should be discontinued until contamination is brought back into control. Affected samples should be qualified or reanalyzed. Method blank results (including surrogate spike results for the method blank) must be reported with their associated sample results.

2.2 Matrix Spike Duplicates

A matrix spike and its duplicate must be analyzed in every batch of samples. The spike concentration should fall in the upper half of the calibration range. The spike recovery should be between 60 and 140% unless another method performance recovery is specified in the method. In all cases, recoveries must be greater than 50%.

2.3 Surrogate Spikes

Surrogate spikes must be analyzed with every organic analysis; the surrogate data must be submitted with each sample; and the surrogate data must be within the appropriate windows (identified below). In those cases where the acceptance window is not met, and the problem cannot be attributed to a correctable cause, the sample must be reanalyzed. If the surrogate(s) sample is still not within the acceptance window in the repeat analysis, the sample results must be reported with a qualifying statement explaining the sample matrix or analytical problems and providing guidance concerning the usability of the sample results.

2.4 Laboratory Documentation and Reporting

2.4.1 Content of Lab Reports

Laboratory results submitted to support closure must be submitted on the original laboratory data sheets. This data may also be tabulated, summarized, and presented in other formats to meet the needs of the project report. The original laboratory data reports must be submitted to OIS and include the following information:

- Sample Field ID.
- Laboratory sample identification numbers and corresponding field sample identification numbers.
- Method of analysis as specified in this guidance (e.g., Method 8021 or Method 8310). If methods other than those specified in this guidance are used, the complete method number, title, and source must be identified.
- Obvious sample odor must be reported. Consultants or lab analysts are not encouraged to sniff samples. However, sample odor must be reported if it is obvious in handling the sample. Odors should be reported in the comment field or section of the lab report.
- Specific parameter or analyte(s).
- Report soil results as ug/kg on a dry-weight basis. Report groundwater results in ug/l.
- Report the percent moisture of soil samples.
- Report the dilution factor for samples or extracts that required dilution to bring the sample concentration within the analytical range of the method.
- Report the lab MDL or TMDL used to report data for the specific method or analyte.
- Date sample was collected.
- Date sample was extracted.
- Date sample was analyzed.
- Report the pH of preserved water samples submitted for analyses of BTEX compounds, volatile halocarbons, TVPH, TEPH, lead, cadmium, and chromium. The pH of volatile samples may be determined after sample analysis has been done so as not to compromise the sample.
- Questionable data should be flagged. Examples of questionable data include

exceedance of sample holding times; failure to meet QC requirements; instrument failure during analysis; improper sample preservation; or any other relevant factors.

- Report the presence of unknown or non-target peaks in the comment section of the lab report when such peaks appear on the sample chromatogram. Examples of this are:
 - A peak on a GC chromatograph for which no standard has been run.
 - Diesel peaks tailing the end of a gasoline range chromatograph.
- Analyst's signature (or the signature of the analyst's supervisor or QC manager).
- QC data associated with the sample result. With every batch of 20 samples or less the results of either:
 - Two replicate spikes (accuracy and precision).
 - One matrix spike.
 - Surrogate spikes. These results must be reported as percent relative standard deviation or percent recovery as appropriate.
- Labs should have Standard Operating Procedures for all methods and procedures. Any deviations from laboratory SOPs must be reported in the comment section of the lab report.

3.0 Characteristics of Petroleum Products

A basic understanding of the major components of petroleum products is necessary to properly select appropriate analytical methods for monitoring or measurement of various petroleum products and to follow them as they move, partition or degrade in the environment. The following section provides information concerning the major components of these products.

3.1 Petroleum Fractions

Petroleum is separated by distillation into various fractions. Because of the relationship between boiling point and molecular weight, this amounts to a rough separation according to carbon number. Each fraction is a complicated mixture containing a range of carbon numbers and molecular structures. The use that each fraction is put to depends on its volatility or viscosity. Examples of fractions separated by distillation are listed as follows.

Table 3 Petroleum Fractions.

Fraction	Dist. Temp. (°C)	Carbon Number/Main Constituents
Gasoline	40 - 205 °C	C ₅ -C ₁₀ , cycloalkanes
Diesel Oils	160 - 400 °C	C ₁₂ -C ₂₀ , cycloalkanes
Lubricating Oils	345 - 540 °C	C ₂₀ -C ₃₄ paraffinic and cycloparaffinic based oils
Kerosenes, Jet Fuels	175 - 325 °C	C ₁₂ -C ₁₈ , and aromatics
Fuel Oils	275 - 345 °C	C ₁₂ -C ₂₀ and higher alkanes
Stoddard Solvents, Naphthas, Thinners	100 - 200 °C	C ₅ -C ₁₃ , cycloalkanes and aromatics
Glycols	175 - 200 °C	C ₅ -C ₇

3.2 Major Petroleum Products

3.2.1 Gasoline and Gasoline Additives

Gasoline is a variable and complex blend of distillation temperature fractions to meet a wide variety of engine designs and operating conditions. Gasoline is blended for the monthly temperature and altitude of the area where it is used. It is also made in different grades or octane ratings to meet the needs of different internal combustion engines. Gasoline differs between companies, refineries, and even between batches from the same refinery. Gasoline contains 11-25% C₃-C₁₂ straight chain alkanes, 18-57% C₄-C₁₀ branched alkanes, 0.7-2.6% C₅-C₈ cycloalkanes, 2.3-3.5% C₄-C₇ straight chain alkenes, 3.3-4.0% C₅-C₇ branched alkenes, 11-56% C₆-C₁₂ alkylbenzenes, and 0.1 to 0.5% C₁₀-C₁₂ polynuclear aromatic hydrocarbons (PAHs).

Gasoline additives include various antiknock compounds, dyes, corrosion inhibitors, antioxidants, octane boosters, and icing inhibitors. The most common octane boosting additives are the aromatics benzene, toluene, ethylbenzene, and xylenes (BTEX). BTEX compounds are added to gasoline as an octane booster and are typically found in the following proportions: benzene, 0.1-3.5%; toluene 2.7-21.8%; ethylbenzene, 0.4-2.9%; and xylenes, 3.2-8.5%. BTEX compounds are useful for evaluating gasoline releases to soil and groundwater. These compounds (especially benzene) tend to move into the aqueous phase more rapidly and are more susceptible to microbial degradation than most other gasoline components. As a result, historically contaminated soil may not contain measurable quantities of benzene and certain other BTEX compounds. Leachate testing of soils can be used, in conjunction with total BTEX concentrations, for determining the allowable soil concentrations for the soil leaching to groundwater migration pathway.

Methanol, ethanol, tertiary-butyl-alcohol (TBA), methyl-tertiary-butyl-ether (MTBE), ethylbenzene-tertiary-butyl-ether (ETBE), and tertiary-amyl-methyl-ether (TAME) are added as blending agents to increase octane ratings of unleaded gasoline. These oxygenated compounds may also be required in Clean Air Act carbon monoxide non-attainment areas to reduce carbon monoxide emissions. MTBE is added to gasoline at concentrations from 2-7% and is restricted to 15% by volume. MTBE is blended into regular, unleaded, and unleaded premium grades to increase octane ratings. The highest levels are found in the higher octane unleaded gasolines (premium unleaded). At 4.3% solubility in water, it is among the most water soluble compounds present in gasoline. Since adsorption to soils is inversely proportional to solubility, MTBE is one of the first gasoline constituents to leach out of soil. It also separates in groundwater, and moves ahead of other gasoline components in groundwater. MTBE is therefore a potentially valuable monitoring parameter, and precursor of gasoline contamination since it may form a "halo" along the leading edge of the plume.

The presence and concentration of MTBE may also be important for design of cleanup remedies. For these reasons, MTBE is a parameter to be evaluated in the investigation and cleanup of unleaded gasoline contaminated groundwater and soil. The value of MTBE as a monitoring parameter is significantly reduced by analytical detection limits of 50 mg/l in water and 100 mg/kg soil. Leachate testing of soil, accompanied by total MTBE concentrations, is an acceptable option for establishing site-specific concentrations that are protective of the soil leaching to groundwater pathway.

Prior to 1996, tetraethyllead was the primary organolead additive to leaded gasoline and was permitted under EPA regulations up to 1,100 mg/l as lead, to increase octane ratings. Other organolead compounds used within the last 10 years include tetramethyllead, trimethyllead, dimethyllead, and methyltriethyllead. Organolead compounds in unleaded gasoline are restricted to 13 mg/l as lead. The use of organolead compounds in gasoline has been greatly reduced in recent years. The last producer of organolead additives in the United States stopped making them in early 1991. 1,2-dichloroethane (1,2-DCA) and Ethylene Dibromide (EDB) were added to leaded gasoline to keep organolead combustion products volatile for discharge with exhaust gases instead of being deposited in the engine combustion chamber. The amount of added 1,2-DCA or EDB is proportional to the amount of organolead.

Leaded aviation gasoline, because of its higher octane rating, will contain up to double the amount of lead and lead scavengers (1,2-DCA or EDB) contained in leaded automotive gasoline. In 1990, six percent of all gasoline produced in the U.S. was leaded gasoline. After December 31, 1995, use of leaded gasoline as a motor vehicle fuel was prohibited as per Section 211 of the Clean Air Act Amendments of 1990.

3.2.2 Diesel Fuels

Diesel fuels contain predominantly a mixture of C₁₀-C₁₉ hydrocarbons and are available in three grades based on volatility, ignition quality, viscosity, and other characteristics. Grade No. 1-D comprises the class of volatile fuel oils from kerosene to intermediate distillates. Fuels within this grade are applicable for use in high speed engines in services involving frequent and relatively wide variations in loads and speeds as well as abnormally low temperatures. Grade No. 2-D includes the class of distillate gas oils of lower volatility. These fuels are applicable for use in high-speed engines in services involving relatively high loads and uniform speeds or engines not requiring high volatility or properties of Grade No. 1-D. Grade No. 4-D covers the class of more viscous distillates and blends of these distillates with residual fuel oils. These fuel oils are applicable for use in low and medium speed engines employed in services involving sustained loads at substantially constant speed. Octyl nitrate is added to some diesel fuels as an ignition improver (as measured by cetane number) at 0.1% to 0.2% by volume.

3.2.3 Lubricating Oils

Lubricating oils contain predominantly a mixture of C₂₀-C₃₄ hydrocarbons. They are made from the more viscous portion of the crude oil which remains after removal of gas oil and lighter fractions by distillation. Many petroleum derived hydraulic oils are similar, but are discussed separately as hydraulic fluids. These generally range from low viscosity oils with molecular weights as low as 250 to very viscous lubricants with molecular weights as high as 1000. The types of hydrocarbon compounds present in lubricating oils include normal (n-), and iso- alkanes, cycloalkanes, aromatics, and mixed alkane, cycloalkane, and aromatic ring compounds. These include SAE viscosity engine oil grades 5W through 50 and gear, axle, and manual transmission lubricant grades 75W through 250.

To minimize variation and produce lubricating oils that provide consistent performance, crude oils are selected and segregated according to principal type. The oil is distilled to achieve the appropriate boiling point range; undesirable constituents are removed; and oils are blended with other oils to attain the appropriate physical characteristics. Crude oil stocks are categorized as paraffinic and cycloparaffinic. Cycloparaffinic crude oil contains no wax, has a low pour point (-46°C), and is most desirable for the range of temperatures in automotive engines. Paraffinic stock requires dewaxing to reduce its pour point from approximately 27 to -18°C, and is less desirable for use in automotive engines. Lubricating oils may contain up to 30% additives including pour point depressants, viscosity index improvers, defoamers, oxidation inhibitors, antiwear compounds, and friction reducing compounds. A discussion of the various refining processes; cracking, upgrading, hydrotreating, chemical treatments, and lube oil additives necessary to yield the final product is beyond the scope of this document.

PAHs are formed in the combustion chambers of internal combustion engines and concentrated in the motor oil. Levels of various individual PAHs found in used motor oil are commonly in the 10-20 mg/kg range and have been measured up to 500 mg/kg. These levels in used oil contrast levels in new lubricating oil which are often near the range of detectability up to about 0.3 mg/kg. If target analytes are not present and further cleanup is necessary to eliminate aesthetic impacts in groundwater or soil, the TEPH target MDLs may be used in conjunction with a risk based corrective action (RBCA) evaluation to achieve closure.

3.2.4 Kerosene and Jet Fuels

Kerosene is used in heating and as an aviation turbine fuel (Jet A and B). Kerosene for heating purposes is available as a special low sulfur grade for use in non-flue connected kerosene burner appliances, and as a regular grade for use in flue connected burner appliances.

Jet fuels are made by blending and refining crude oil petroleum products such as naphtha, gasoline, or kerosene to meet specific military or commercial specifications. JP-4 and JP-7 are military specifications; Jet A and Jet A-1 are commercial or civilian specifications. JP-4 is intended for use in advanced supersonic aircraft and is a naphtha-type fuel made by blending straight-run kerosene streams and lower boiling streams to fit the composition containing approximately (by weight) 43% C₄-C₁₄ alkanes, 11% C₆-C₁₀ cycloalkanes, 12% C₆-C₁₀ alkylbenzenes, and 2% C₁₀-C₁₂ naphthalenes. It is known as a wide-cut fuel made from distillation products obtained over a wide range of temperatures, and has a broad spectrum of individual compounds with chain lengths ranging from C₄-C₁₆.

JP-7 is made by blending kerosene distillates to achieve a product containing a maximum of 5% aromatics by volume and a maximum of 0.1% sulfur by weight. One example of JP-7 contained approximately (by volume) 97.4% C₁₀-C₁₇ alkanes, 0.4% C₁₀-C₁₁ cycloalkanes, 1.6% C₁₀-C₁₂ alkylbenzenes, and 0.6% C₁₀-C₁₁ naphthalenes.

Jet A and Jet A-1 are kerosene type fuels, used in commercial jet aircraft. Jet A-1 is similar to Jet A, except that the freezing point is lower for long duration international flights. Jet A-1 is the military equivalent of JP-8.

3.2.5 Fuel Oils

Fuel oils are classified as distillates or residuals based on production methods. The distillates consist of overhead or distilled fractions. The residuals are bottoms remaining from distillation. Grades No. 1 and 2 are distillates, and grades No. 4 to 6 are usually residuals (Grade No. 3 is no longer produced). Grade No. 1 is a light distillate, consisting of hydrocarbons in the C₉-C₁₆ range, and intended for use in burners of the vaporizing type in which the oil is converted to a vapor by contact with a heated surface or by radiation. High volatility is necessary to ensure that evaporation proceeds with a minimum of residue. Grade No. 2 is a heavier distillate, usually blended, consisting of hydrocarbons in the C₁₁-C₂₀ range, and intended for use in atomizing type burners which spray the oil into a combustion chamber where the tiny droplets burn while in suspension. This grade is used in most domestic burners and in many medium capacity commercial-industrial burners. Grade numbers 4, 5, and 6 (Bunker C) are progressively heavier residuals intended for burners designed to handle their associated viscosity ranges. In the heavier residuals, preheating may be necessary to atomize the fuel or pump the fuel from the storage tank.

The individual PAH compounds and levels present in fuel oil grades are variable. For comparison, PAHs present in fuel oil No. 1 include naphthalenes (2.8%) and acenaphthenes (0.4%); PAHs present in No. 2 fuel oil include naphthalenes (8.2%), acenaphthenes (2.6%), fluorenes and acenaphthylenes (1.4%), and phenanthrenes (1.4%).

3.2.6 Stoddard Solvent and Other Petroleum Solvents

Stoddard solvent, also called white spirits, is a mixture containing hydrocarbons from C₅ to C₁₃ with the majority of hydrocarbons in the C₉-C₁₁ range, and with a boiling point range of between 155-200 °C. There are several different formulations, but in general the hydrocarbons composing Stoddard solvent are 30-50% alkanes, 30-40% cycloalkanes, and 10-20% aromatics. Specific compounds and amounts measured in some formulations and appearing on EPA regulatory lists, or listed in analytical methods include: acenaphthenes (0.4%), n-propylbenzene (2.0%), 1,2,4-trimethylbenzene (0.9%), naphthalene (0.2%), and acenaphthalenes (0.3%). Stoddard solvent is a multipurpose petroleum solvent. Industrial uses include paint vehicles; thinning agents for paints, coatings, and waxes; printing inks; adhesives; as a solvent in liquid photocopier toners; and as a degreaser in machine and automotive repair shops.

Other petroleum solvents include petroleum spirits, mineral spirits, varnish maker's and painter's (VM&P) naphthas. These products are general purpose thinners and solvents for paint and varnish. The chemical composition is similar to stoddard solvent, but the range of boiling points in this category of products is broader.