SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

Standard Operating Procedure

Total Volatile Petroleum Hydrocarbons (TPH) by Gas Chromatography

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TOTAL VOLATILE PETROLEUM HYDROCARBONS (TPH) BY GAS CHROMATOGRAPHY

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1.0 SCOPE AND APPLICATION

1.1 This SOP is applicable to the chromatographic analysis of solid and liquid samples for total volatile petroleum hydrocarbons. This method is used for the analysis of samples containing hydrocarbon fuels including gasoline and Stoddard solvent. Concentration levels from 20ppb to 1000ppb (undiluted) are obtainable for volatile fuels (gasoline) using purge and trap techniques.

2.0 METHOD SUMMARY

2.1 Samples are analyzed for total petroleum hydrocarbons by gas chromatography (GC) using a flame ionization detector (FID) under conditions similar to those described in SW-846 Method 8015. Preparation for GC analysis is by purge-and-trap by SW-846 Method 5030. Volatile fractions must be analyzed within 14 days from the time of sample collection.

Note: See attached appendices for specific state requirements and information.

3.0 HEALTH AND SAFETY

3.1 Good laboratory practices dictate the careful handling of all laboratory samples and reagents. Refer to the current version of the "Laboratory Safety Plan", SWL-GA-111. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals is reduced to a minimum.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Soil Sample Handling and Storage

4.1.1 Soil samples should be collected and stored in airtight glass containers with Teflon lined lids or brass sleeves. Containers should have minimal or no headspace after filling with the sample. A minimum of 100 grams of sample should be collected. Samples should be maintained at 39°F ± 4° (4°C ± 2°) during shipment and storage prior to analysis.

4.2 Aqueous Sample Handling and Storage

4.2.1 Aqueous samples should be collected in airtight glass containers with Teflon lined lids. Containers should contain no headspace after filling with the sample. A minimum of two 40 mL aliquots (VOA vials) should be collected for purgeable analyses. Samples should be maintained at 39°F ± 4° (4°C ± 2°) during shipment and storage prior to analysis.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Solvents, reagents, and processing hardware may yield artifacts and/or elevated baselines leading to misinterpretation of chromatograms. The system must demonstrate that it is free of interference under the conditions of the analysis by running method blanks. Specific selection of reagents and purification of solvents may be required.

5.2 Many major sources of contamination are avoided by using stainless steel, nickel, PFTE, glass, and other inert materials for all equipment and materials that in any way affect the detection and quantitation of volatile contaminants in samples. However, other areas of contamination do exist, and are avoided as follows:

5.2.1 Samples can sometimes be contaminated by diffusion of volatile organics through the septum seal during shipment and storage. This can be handled by the use of a trip blank prepared from organic-free filtered tap water, which is carried through the entire sampling and shipping process with all samples.

5.2.2 Interfering contamination can occur when a sample is analyzed immediately following a sample containing high concentrations of volatile organic compounds. All such samples are reviewed carefully for any indication of carryover and reanalyzed when there is any question of whether contaminants found in the sample are the result of true contamination or originate from the previous analysis.

5.2.3 Common laboratory solvents present in the air of most analytical laboratories, notably methylene chloride, acetone, and chloroform, can cause contamination of the analytical instruments themselves. This can be checked by analyzing an instrument blank, where the presence of volatile contaminants can alert the analyst of the need to perform any cleaning measures necessary to achieve an analytical run which is as contaminant-free as possible.

6.0 EQUIPMENT/APPARATUS

6.1 Gas Chromatograph – Hewlett-Packard model 5890 equipped with FID, or equivalent.

6.2 Purge-and-trap – Tekmar ALS 2016 and ALS 2032 automatic liquid samplers and Tekmar LSC 2000 liquid sample concentrator, or equivalent. Heating blankets are added to the Tekmar ALS when low level soils are analyzed.

6.3 GC column – JandW Scientific DB-1, #125-1034, megabore capillary column, 30 meters long x 0.53 mm ID, or equivalent.

6.4 Volatiles-specific column – J&W DB-624 #125-1334

6.5 Data System – Hewlett-Packard Chemstation/Environment G1701AA , or equivalent.

6.6 Toploader Balances – Ohaus Galaxy 400 and Precision Plus, or equivalent.

6.7 Micro Pipettor – Drummond, various sizes.

6.8 Graduated Cylinder – 1000 mL.

6.9 Volumetric Flasks – Various sizes.

6.10 Micro Syringes – 10 µL and larger.

6.11 VOA Vials – 40 mL with teflon septa.

7.0 REAGENTS

7.1 The following standards of known purity as neat material or solutions of certified concentration:

7.1.1 Trifluorotoluene (TFT)– Surrogate

7.1.2 Commercially Prepared Standard Solutions---Commercially prepared solutions are purchased and stored at –10 to –20°C.

7.1.3 Reagent Water—Produced by filtering tap water through an organic filtering system containing activated charcoal prior to use.

7.1.4 Methanol – Pesticide quality or equivalent

8.0 PROCEDURE

8.1 Recommended operating conditions for the gas chromatograph and column conditions: Column temperature is isothermal at 45°C for four minutes, then programmed at 4°C per minute to 105°C. further programmed at 12°C/min. to 155°C and 27°C/min. to 255°C for 5 minutes.

8.2 Recommended purge-and-trap parameters include a purge time of 11 ± 1.0 minutes. All other parameters are trap dependent and will vary accordingly.

8.3 Adjust the purge gas (helium) flow rate to 35 ± 3 mL/min on the purge and trap. Variations from this flow rate may be necessary to achieve better purging and collection efficiencies.

8.4 Initial Calibration

8.4.1 Initial calibration of the GC is established and validated by the analysis of traceable standards at five concentration levels which are 20, 100, 250, 500,and 1000 ppb for the water and soil methods. These standards are made up in 5mL of filtered tap water (as shown in Table 1 on the next page) and purged on the Tekmar before analysis by the GC.

Table 1
Soil - 5 mL Purge/Tekmar ALS2032/ALS2016

100 (ng/mL) Surrogate (µL)	5uL of Stock Std. Conc.	Standard Final Conc. (ng/mL)	Surrogate Final Conc. (ng/mL)
5	20	20	100
5	100	100	100
5	250	250	100
5	500	500	100
5	1000	1000	100

8.4.2 When analyzing on the Tekmar 2032/2016, the syringe is attached to the syringe valve assembly on the Tekmar. The syringe valve is opened, the sample is injected into the purging chamber, and the purging device valve is closed. Water samples are purged at ambient temperature. Low-level soils are purged at 40° +/- 1°C. The sample is purged at ambient temperature. At the conclusion of the purge time, the trapped materials are introduced to the GC column by the Desorb Mode where the trap is rapidly heated for four minutes.

8.4.3 After the sample desorbs onto the GC, the trap is baked for a minimum of four minutes, then it is allowed to cool to less than 35°C before being ready to purge the next standard or sample.

8.4.4 The standard will be quantitated into a quant report in which concentrations and responses. For the identification of gasoline range organics by the purge and trap technique, several characteristic peaks are chosen and integrated individually. Gasoline is integrated to the baseline. Each characteristic peak is calibrated and the peaks are summed for a total concentration reported as gasoline. Integration technique may be specified in particular methods, and those should be consulted for specific guidelines.

8.4.5 The results of the initial calibration are used to prepare a calibration curve. Average calibration factors are calculated for each compound. The relative standard deviation (RSD) should be less than 20% over the range. If the %RSD of the calibration factor is greater than 20%, then a linear regression equation may be employed. The correlation coefficient of the curve should be greater than 0.99.

8.4.6 If the five-point does not meet criteria, each standard is checked for the need of a possible rerun, in case the standards have been added at the wrong amount, or the purge may have been bad. If reanalyzing a standard will not solve the problem, a supervisor is contacted. Some problems could be found to be related to purge chambers, flows, etc.

8.4.7 The retention times are updated with each initial calibration run. The Chemstation Software Program performs this function. The software requires the window width to be the same for all analytes. e.g. ± 0.1 minute. A separate window is established for surrogates.

8.5 Calibration Verification

8.5.1 A calibration verification of the GC uses the same traceable standards, but at only one level: 500 ppb. The analysis is performed in the same manner as stated before in the initial calibration and is analyzed each day prior to analysis of samples, after every ten samples, and at the end of a sequence.

8.5.2 The calibration verification must meet all criteria before any other analysis continues. If the criteria are not met, the standard must be rerun, as it is possible, the standard was not prepared correctly or that there was a bad purge. However, if the second standard still doesn't meet criteria, an initial calibration must be run or corrective action performed.

8.5.3 If the calibration verification does meet the criteria as stated in the Appendices, samples may be run. Samples are quantitated based on the criteria stated above in Section 8.4.4.

8.6 Water Samples

8.6.1 Any headspace in the bottle is noted before opening. If analyzing on the Tekmar 2032 or 2016, the sample bottle (which has been allowed to come to ambient temperature) is opened and is carefully poured into the syringe barrel to just short of overflowing, then adjusted to a volume of 5 mL. This process of taking an aliquot destroys the validity of the sample for future analysis. Care must also be taken to prevent air from leaking into the syringe.

8.6.2 When spiking samples on the Tekmar 2032 or 2016, 5 µL of the Surrogate Spiking Solution is added through the valve bore of the syringe, which is equivalent to a concentration of 100 µg/L of each standard.

8.6.3 Water samples are loaded in the following manner to ensure the proper sample is placed into its designated purge chamber on the Tekmar 2032/2016 Autosampler:

NOTE: All sample purge tubes are labeled with the SWLO ID Number and Autosampler position. Each analyst verifies as he/she loads each sample into the purge chambers the syringe matches the ID on the purge tube.

8.6.4 For water samples analyzed on the Tekmar 2032 or 2016 requiring matrix spike and matrix spike duplicates, 5 µL of the matrix spike solution is added to the sample to be purged. Disregarding any dilutions, this is equivalent to a concentration of 500 µg/L of each matrix spike standard.

8.6.5 With all matrices, all dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

8.6.6 The formula for calculating concentrations for target compounds is found in Section 9.

8.6.7 After analysis is complete, the pH is recorded for all water samples on a pH Log (See Figure 1). If a sample has a pH reading >2, the project officer associated with that particular client's samples is notified.

8.7 Soil Sample Analysis - Low Level

8.7.1 The low-level method is based on purging a heated sediment/soil mixed with filtered tap water containing the surrogate and internal standards.

8.7.2 The sample (for volatile organics) consists of an equal representation of the entire contents of the sample container (i.e., no supernatant liquids are discarded.) The contents of the sample container are mixed with a narrow spatula, then the sample is weighed into a disposable test tube if analyzing on the Tekmar 2032 or 2016.

8.7.3 Immediately after weighing the sample into the labeled test tube, it is loaded onto the Tekmar ALS. If analyzing on the Tekmar 2032/2016, the plunger from a 5mL Luerlock syringe is removed and the syringe is filled to overflowing with filtered tap water. The plunger is replaced to vent trapped air, then the volume is adjusted to 5.0mL. 5 µL of the Surrogate Spiking Solution is added to the syringe. This addition to the soil is equivalent of 100µg/Kg of each standard. This spiked filtered tap water is added to the test tube, the sample is heated to 40°C, and purging is begun. The samples follow the same purge method as stated in the calibration.

8.7.4 For soil samples requiring matrix spike and matrix spike duplicates, 5µL of the matrix spike solution is introduced into the 5 mL of the filtered tap water being added to the soil if analyzing on the Tekmar 2032/2016. Disregarding any dilutions, this is equivalent to 500 µg/Kg of each matrix spike standard.

8.7.5 With all matrices, all dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

8.7.6 The formula for calculating concentrations for target compounds is found in Section 9.

8.7.8 All soil samples must have their percent moisture determined so that compound contamination can be reported for "dry weight." This is performed by the Extractions Department and submitted to the Volatiles Data Clerk for reporting.

8.8 Soil Sample Analysis — Medium Level

8.8.1 Medium level extraction/analysis is required if analysis of a sample at a 1-gram level produces peak saturation, or compounds out of linear range. The medium level method is based on extracting the sediment soil sample with methanol. An aliquot of the methanol extract is added to filtered tap water containing the internal standards. The surrogate is spiked directly into the extract, thus making the addition directly into the filtered tap water unnecessary. The extract is purged at ambient temperature.

8.8.2 The medium level extract is prepared by weighing 4 grams of the sample into the scintillation vial. Using a top-loading balance, the actual weight is noted to the nearest 0.1 g, then 9900 µL of methanol and 100 µL of the medium concentration Surrogate Standard are added. The mixture is then capped and shaken by hand for 2 minutes. The extract should be made up quickly in order to avoid the loss of volatile organics.

8.8.3 The sample is loaded in the following manner: if analyzing on the Tekmar 2032/2016, a syringe is filled just short of overflowing using filtered tap water, then the volume is adjusted to 4.9 mL. The plunger is then adjusted to 5 mL to allow for the addition of the extract. Finally, the volume of the methanol extract and the volume of methanol, if needed, is added to total 100 µL.

8.8.4 The sample is loaded into the purging device (Tekmar) and the same purging method as stated during calibration is followed.

8.8.5 For samples requiring matrix spike and matrix spike duplicates, an extract is prepared in the same manner as stated above in Section 8.9.2, using 9800 µL of methanol, 100 µL of medium concentration surrogate, and 100 µL of medium concentration matrix spike solution. These samples are loaded in the same manner as other samples.

8.8.6 The method blank must be analyzed in the same manner as with low-level soils and waters, but it is prepared differently using 4 gram of a purified solid matrix. Surrogate Spiking Solution and methanol are then added to make a total volume of 10 mL, and the blank is purged at ambient temperature. An acceptable blank must contain less than or equal to the reporting limit of any volatile target compound, or else corrective action will be taken immediately.

8.8.7 With all matrices, all dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

8.8.8 The formula for calculating concentrations for target compounds is found in Section 9.

8.8.9 All soil samples must have their percent moisture determined so that compound contamination can be reported for "dry weight." This is performed by the Extractions Department and submitted to the Volatiles Data Clerk for reporting. The values may also be obtained from the LIMs database by episode.

8.9 Analysis Report Criteria--A sample is acceptable when it meets the following criteria:

8.9.1 Analysis Sequence

8.9.1.1 Calibration Verification standard (or initial calibration if Ccal fails twice)

8.9.1.2 Method Blank

8.9.1.3 Laboratory Control Spike

8.9.1.4 Up to 10 samples including an MS/MSD

8.9.1.5 Calibration Standard

8.9.1.6 Repeat steps 8.3.1.4 and 8.3.1.5, ending with 8.3.1.5

8.9.2 Surrogate recoveries are within the limits stated in Table 3.

8.9.3 Any compound found in the sample is within the linearity range, with any dilutions showing compounds in an acceptable range of linearity. If a sample analysis contains any target compounds above the linear range, all subsequent samples will be reviewed for those target compounds. The target compounds must be below the reporting limits to be acceptable; otherwise they must be reanalyzed.

8.9.4 Chromatogram shows no signs of carryover or instrument deficiencies.

8.9.5 Confirmation. Second column confirmation is not feasible for petroleum fuels. Rather than determining specific analytes, this method determines the presence of petroleum hydrocarbons over a wide range of elution times.

8.10 Reanalysis Criteria: If sample does not meet surrogate criteria, it must be reanalyzed. If the reanalysis meets all criteria, it is submitted. If the reanalysis duplicates the results from the first analysis, both analyses are submitted. This information is recorded in the analyst's automated logbook.

8.11 A sample report is complete when it contains all of the following in order:

8.11.1 Chromatogram

8.11.2 Quant Report

8.11.3 Manual Integrations

8.11.4 Surrogates

9.0 CALCULATIONS.

9.1 Concentration of a compound is determined from the calibration curve multiplied by any dilution factor. The type curve fit is specified and the software calculates the concentration. If a linear curve fit is used, concentration is calculated from the following formula:

$$\text{Conc. } (\mu g/kg \text{ or } \mu g/l) = (Rs \times A \times D) \times \frac{V}{W}$$

Where:

Rs = Average response factor
A = Peak area
D = Dilution factor
W = Weight (in kg) or volume (in liters) of sample used (extractables only)

Note: This is an As Reviewed Value, not corrected for moisture.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Method Blank (Laboratory Reagent Blank)

10.1.1 The method blank is analyzed at a minimum of one per 20 samples or per batch once the calibration has been met, whether it be an initial calibration or calibration verification. The method blank, called the "BLANK", consists of loading a syringe with 5mL of filtered tap water spiked to yield a concentration of 20ppb surrogate. The BLANK is used to establish the analytical system is free of contaminants. It is also used to check for carryover contamination after a standard run.

10.1.2 An acceptable blank must meet the surrogate recoveries found in Table 3. The concentration of the target compounds in the blank must be less than the method reporting limit for each target compound (see Table 4).

10.1.3 If a blank exceeds these criteria, corrective measures must be taken immediately, beginning with immediately reanalyzing the blank. If all criteria are still not met, the supervisor is then consulted.

10.2 Laboratory Control Spike (LCS and LCSD)

10.2.1 The Laboratory Control Spike (LCS and LCSD) are internal laboratory QC samples designed to show the capability of our laboratory to be in compliance with the protocol. They consist of filtered tap water spiked with an LCS solution, which contains Gasoline from a source other than daily calibration standards at a 500ppb level.

10.2.2 An acceptable LCS and LCSD must meet the surrogate recoveries in Table 3. The percent recovery for each of the compounds in the LCS and LCSD should be within recovery limits shown in the QC Method Reference "Blue Book". The LCS limits are set forth by our laboratory showing our internal control limits, therefore, operators should put forth every effort to meet these limits. Limits are derived using a control chart program as per the Control Chart SOP (SWL-GA-112).

10.2.3 If the compounds are outside QC limits, corrective action is needed immediately, beginning with rerunning the LCS to verify that it was prepared correctly and that it was not a bad purge. If reanalysis does not solve the problem, a new standard may need to be made.

10.3 Matrix Spikes - MS/MSDs will be analyzed at a rate of 1 MS/MSD per batch of 20 or fewer samples. The recovery limits will be the same as the LCS/LCSD and will be advisory since they are dependent upon the matrix.

10.4 Control Limits

10.4.1 Maximum %RSD for linearity calculation of initial calibration standards is 20%. For curve fit, the minimum correlation coefficient is 0.99. If neither condition is met, a new five point initial calibration must be established.

10.4.2 Calibration verification standards may not differ by more than 15% from the value established in the initial calibration. If this difference is > 15%, repeat calibration verification. If the difference is still > 15%, a new initial calibration is performed. Subsequent mid level standards in a sequence must be within 15% of the calibration verification for that day. Otherwise, all samples analyzed after an out-of- limit standard must be reanalyzed for any analyte, which was out of control and present in the sample.

10.4.3 If a sample contains an analyte at > twice times the highest concentration of the linear range and the subsequent sample contains low levels of this analyte, the subsequent sample will be reviewed for contamination and/or reanalyzed to determine if analyte presence was due to carryover.

11.0 WASTE DISPOSAL

11.1 All samples shall be disposed of following the guidelines set forth in SOP, SWL-GA-114 "Hazardous Waste Management Plan".

12.0 METHOD PERFORMANCE. Not Applicable.

13.0 REFERENCES In addition to modifications of method 8015, this procedure is based on the following procedures:

13.1 "Proposed Modified EPA Method 8015" from the U.S. Corps of Engineers (CENPD-EN-G-L). 1989.

13.2 "Method for Total Petroleum Hydrocarbons and Total Organic Lead" from the Hazardous Materials Laboratory, California Department of Health Services. February, 1988.

13.3 "OA-1" and "OA-2" from the University Hygienic Laboratory, Iowa City, Iowa. July, 1991.

13.4 USEPA SW-846 "Test Methods for Evaluating Solid Waste Physical/Chemical, Revision 4, December 1996, Methods 8000B & 8015B.

14.0 DEFINITIONS. Not Applicable.

15.0 ATTACHMENT

15.1 Table 2: Purgeable TPH Calibration Standards

15.2 Table 3: Surrogate Recovery Limits

15.3 Table 4: Compound and Method Recovery Limits

15.4 Volatiles pH Log

Table 2
Purgeable TPH Calibration Standards

Petroleum Product	Level 1 conc. (ppb)	Level 2 conc. (ppb)	Level 3 conc. (ppb)	Level 4 conc. (ppb)	Level 5 conc. (ppb)
Gasoline	20	100	250	500	1000

The above extractable standards are for California and Iowa (also Alaska, Tennessee, Wisconsin, etc.) which require separate analyses for purgeable (i.e. gasoline) and extractable (i.e. all other) TPH.

Note: The QC acceptance criteria for spikes and surrogates are derived statistically per SW846 and are used to determine if analytical batches are in control. The recoveries are monitored by the use of Control Charts and may be found in the SWL Blue Book.

15.2 Table 3: Surrogate Recovery Limits

Table 3
Surrogate Recovery Limits*

Surrogate Compound	Water	Low/Med Soil Sediment
Trifluorotoluene	86-115	74-121

*Based upon laboratory limits

15.3 Table 4: Compound and Method Reporting Limit

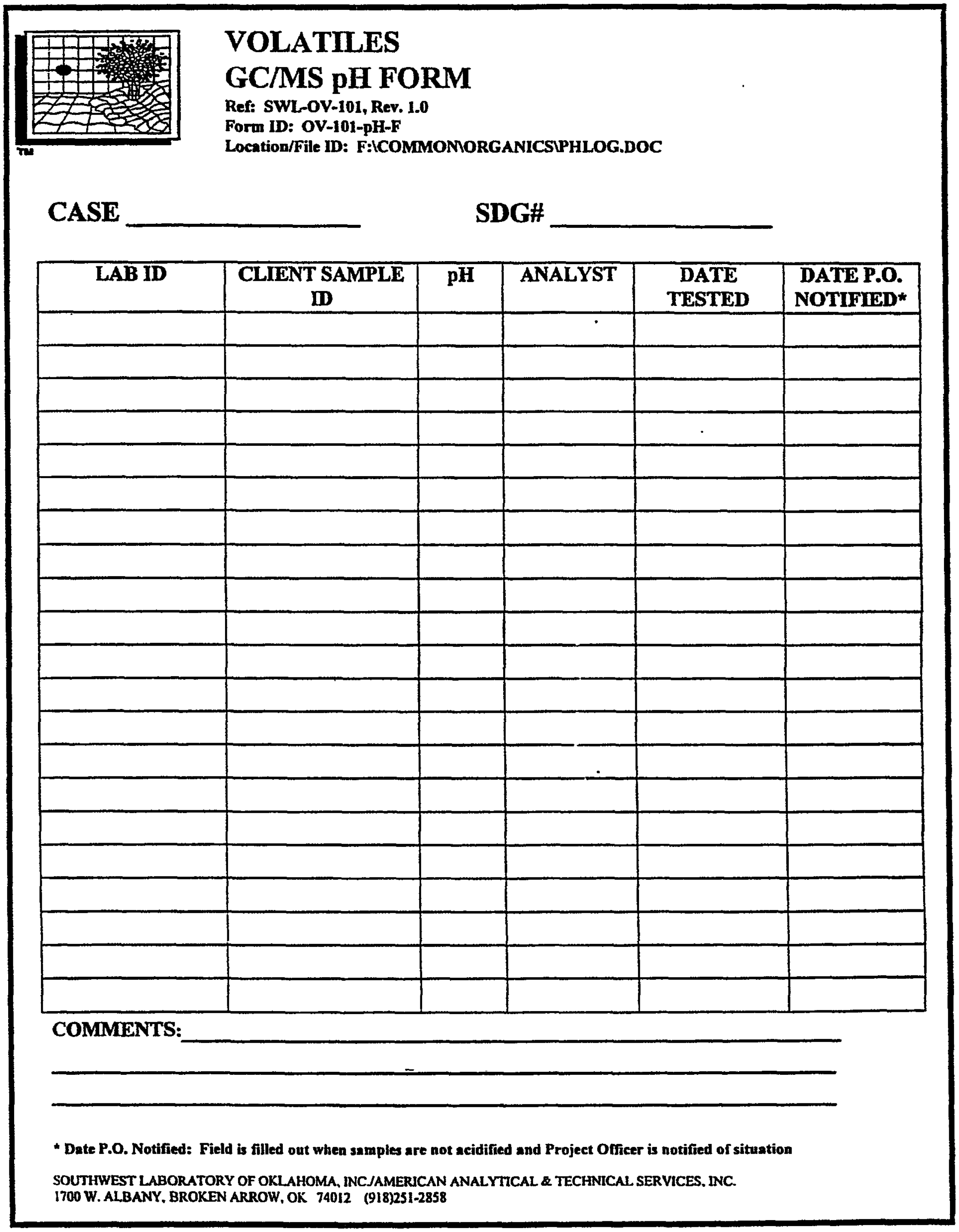
Table 4

Compound and Method Reporting Limits

Volatile Compound	5mLWater or 5g Soil*
Gasoline	20

NOTE: Concentration Units: Water=µg/L Soil=µg/Kg
*Reporting Limits derived from method.

15.4 Volatiles GC pH Log



VOLATILES
GC/MS pH FORM
Ref: SWL-OV-101, Rev. 1.0
Form ID: OV-101-pH-F
Location/File ID: F:\COMMON\ORGANICS\PHLOG.DOC

CASE ____________ **SDG#** ____________

LAB ID	CLIENT SAMPLE ID	pH	ANALYST	DATE TESTED	DATE P.O. NOTIFIED*

COMMENTS: ____________

*** Date P.O. Notified: Field is filled out when samples are not acidified and Project Officer is notified of situation**

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Appendices:

Note: These appendices contain specific information pertaining to various state requirements and method information. The following method information will take precedence when there is variation from method procedures that were established previously in SOP. The establishment of specific state requirements does not limit nor preclude the ability of the laboratory to generate changes to methodology for client specific needs or further development of new methods.

Appendix A

Summary of California method :

A flame ionization detector (FID) that is in series with a photo ionization detector (PID), in conjuction with purge and trap methods, is used for detection and quantitation of certain volatile petroleum fractions. Benzene, Toluene , Ethylbenzene, and Xylenes may require a PID in highly contaminated soils in which their determination would otherwise be masked. Determination of volatile hydrocarbons will be done by purge and trap method as per "Method for Total Petroleum Hydrocarbons and Total Organic Lead" from the Hazardous Materials Laboratory, California Department of Health Services. February, 1988.

Specific information relating to method:

A.1 STOCK STANDARDS:

a.1.1 Unleaded Gasoline Composite Standard

a.1.2 Surrogate- Triflourotoluene.

A.2 CALIBRATION INFORMATION:

a.2.1 A correlation coefficient of 0.995 or higher should be obtained. Alternatively, the average response factor can be used if a 20% or less percent relative standard deviation is achieved.

a.2.2 A daily continuing calibration standard response factor must be within +/- 10% of the initial calibration average response factor or within +/-10 % drift for linear regression curves. Otherwise, another initial calibration will need to run.

A.3 QUALITY CONTROL:

Note: The QC acceptance criteria for spikes and surrogates are derived statistically per SW846 and are used to determine if analytical batches are in control. The recoveries are monitored by the use of Control Charts and may be found in the SWL Blue Book.

a.3.1 Calculated control limits

Table a.1
TPH Control limits

Matrix	Surrogate TFT	Laboratory Control Spike	Relative Percent Difference	Matrix Spike	Relative Percent Difference
Water	49-133	64-110	19%	55-111	21%
Soil	21-153	64-110	19%	41-117	22%

A.4 COMPOUND AND METHOD RECOVERY LIMIT:

Table a.2
Detection Limits

Compounds	Water (ug/l)	Soil(ug/kg)
Total Gasoline Range Organics	20	20

A.5 STANDARD PREPARATION:

Table a.3

Standards Preparation

Compound or Standard Mix	Begin Conc. (µg/mL)	Aliquot (µL)	Total Vol. (mL)	Final Conc. (µg/mL)
Surrogate: RESTEK: a,a,a-TFT	2,000	1250	25	100
L4-500STD RESTEK: Unleaded Gas Composite Standard	50,000	100	10	500

Appendix B

Summary of Iowa method (OA-1):

A flame ionization detector (FID) that is in series with a photo ionization detector (PID), in conjuction with purge and trap methods, is used for detection and quantitation of certain volatile petroleum fractions. Benzene, Toluene , Ethylbenzene, and Xylenes may require a PID in highly contaminated soils in which their determination would otherwise be masked.

Specific information relating to method:

B.1 STOCK STANDARDS:

b.1.1 Unleaded Gasoline Composite Standard

b.1.2 Individual mix-Revised WISC PVOC/GRO mix.

b.1.3 Surrogate- Triflourotoluene.

B.2 CALIBRATION INFORMATION:

b.2.1 A correlation coefficient of 0.995 or higher should be obtained. Alternatively, the average response factor can be used if a 20% or less percent relative standard deviation is achieved.

b.2.2 A daily continuing calibration standard response factor must be within +/- 20% of the initial calibration average response factor or within +/-20 % drift for linear regression curves. Otherwise, another initial calibration will need to run.

B.3 QUALITY CONTROL:

Note: The QC acceptance criteria for spikes and surrogates are derived statistically per SW846 and are used to determine if analytical batches are in control. The recoveries are monitored by the use of Control Charts and may be found in the SWL Blue Book.

b.3.3 Calculated control limits

Table b.1
TPH Control limits

Matrix	Surrogate TFT	Laboratory Control Spike	Relative Percent Difference	Matrix Spike	Relative Percent Difference
Water	49-133	64-110	19%	55-111	21%
Soil	21-153	64-110	19%	41-117	22%

B.4 COMPOUND AND METHOD RECOVERY LIMIT:

Table b.2
Detection Limits

Compounds	Water (ug/l)	Soil(ug/kg)
Total Gasoline Range Organics	20	20

B.5 STANDARD PREPARATION:

Table b.3

Standards Preparation

Compound or Standard Mix	Begin Conc. (µg/mL)	Aliquot (µL)	Total Vol. (mL)	Final Conc. (µg/mL)
Surrogate: RESTEK: a,a,a-TFT	2,000	1,250	25	100
L4-500STD RESTEK: Unleaded Gas Composite Standard	50,000	100	10	500

Appendix C

Summary of Oklahoma method (ODEQ):

A flame ionization detector (FID) that is in series with a photo ionization detector (PID), in conjuction with purge and trap methods, is used for detection and quantitation of certain volatile petroleum fractions. This method can be used for determination and quantitation of individual compounds as shown in Table c.2. This method requests soil sample results be reported on a "wet-weight" basis. Since the majority of our clients request reports using a "dry-weight" basis, the laboratory uses client discretion for this reporting option.

Specific information relating to method:

C.1 STOCK STANDARDS:

c.1.1 Individual mix-Revised WISC PVOC/GRO mix.

c.1.2 Surrogate- Triflourotoluene.

C.2 CALIBRATION INFORMATION:

c.2.1 A correlation coefficient of 0.99 or higher should be obtained. Alternatively, the average response factor can be used if a 20% or less percent relative standard deviation is achieved.

c.2.2 A daily continuing calibration standard response factor must be within +/- 20% of the initial calibration average response factor or within +/-20 % drift for linear regression curves. Otherwise, another initial calibration will need to run.

C.3 QUALITY CONTROL:

Note: The QC acceptance criteria for spikes and surrogates are derived statistically per SW846 and are used to determine if analytical batches are in control. The recoveries are monitored by the use of Control Charts and may be found in the SWL Blue Book.

c.3.1 Calculated control limits

Table c.1
TPH Control limits

Matrix	Surrogate TFT	Laboratory Control Spike	Relative Percent Difference	Matrix Spike	Relative Percent Difference
Water	49-133	73-137	18%	73-137	18%
Soil	21-153	64-110	19%	41-117	22%

C.4 COMPOUND AND METHOD RECOVERY LIMIT:

Table c.2
Recommended Practical Quantitation Limits

Compounds	Water (ug/l)	Soil(ug/kg)
Total Gasoline Range Organics	20	20
Methyl-tert-Butyl Ether	2	2
Benzene	2	2
Toluene	2	2
Ethylbenzene	2	2
m-Xylene	2	2
p-Xylene	2	2
o-Xylene	2	2
1,3,5-Trimethylbenzene	2	2
1,2,4-Trimethylbenzene	2	2
Naphthalene	2	2

C.5 STANDARD PREPARATION:

Table c.3

Standards Preparation

Compound or Standard Mix	Begin Conc. (µg/mL)	Aliquot (µL)	Total Vol. (mL)	Final Conc. (µg/mL)
Surrogate: RESTEK: a,a,a-TFT	2,000	1,250	25	100
L4-500STD RESTEK: WISC/PVOC/GRO	10,000	500	10	500

Appendix D

Summary of Arizona method (8015AZ):

A flame ionization detector (FID) in conjuction with purge and trap methods, is used for detection and quantitation of gasoline range organics (GRO). The range will be defined as the sum of hydrocarbons falling between benzene and naphthalene.

Specific information relating to method:

D.1 STOCK STANDARDS:

d.1.1 Unleaded Gasoline Composite Standard

d.1.2 Individual mix-Revised WISC PVOC/GRO mix. (Retention Time Verification Standard)

d.1.3 Surrogate- Triflourotoluene.

D.2 CALIBRATION INFORMATION:

d.2.1 A correlation coefficient of 0.995 or higher should be obtained. Alternatively, the average response factor can be used if a 20% or less percent relative standard deviation is achieved.

d.2.2 A daily continuing calibration standard must be within +/-30 % drift of initial calibration. Otherwise, another initial calibration will need to run.

d.2.3 A secondary source standard is injected after the initial calibration and must be within +/-20 % drift of actual concentration. Otherwise, another initial calibration will need to run.

d.2.4 A retention time verification standard is also run with the initial calibration and as needed.

D.3 EXTRACTION AND ANALYSIS OF SOIL SAMPLES:

d.3.1 All soil extractions must be completed within 72 hours of sampling and the subsequent analysis must be completed within 14 days of sampling.

d.3.2 The following step is optional.: Add 1-3 grams of anhydrous sodium sulfate and silica gel to an empty 40 mL voa vial, if the sample contains moisture or non-petroleum hydrocarbon interferences that inhibit analysis.

d.3.3 Remove samples from refrigerator and weigh out 10 grams of soil into previously prepared 40 mL voa vial.

d.3.4 Spike all samples, method blanks, and qc samples with surrogate and matrix spike solutions according to individual sample requirements.

d.3.5 Add 10 mL of methanol to voa vials. Cap samples and vortex for 2 minutes.

d.3.6 Allow samples to settle.

d.3.7 Transfer samples to a labled vial for storage and seal.

d.3.8 Take appropriate aliquot from samples; spike it into 5 mL of water and analyze using purge and trap methods.

D.4 QUALITY CONTROL:

Note: The QC acceptance criteria for spikes and surrogates are derived statistically per SW846 and are used to determine if analytical batches are in control. The recoveries are monitored by the use of Control Charts and may be found in the SWL Blue Book.

d. 4.1 Calculated control limits

Table d.1
TPH Control limits

Matrix	Surrogate TFT	Laboratory Control Spike	Relative Percent Difference	Matrix Spike	Relative Percent Difference
Water	49-133	64-110	19%	55-111	21%
Soil	21-153	64-110	19%	41-117	22%

D.5 COMPOUND AND METHOD RECOVERY LIMIT:

Table d.2
Recommended Practical Quantitation Limits

Compounds	Water (ug/l)	Soil(ug/kg)
Total Gasoline Range Organics	20	20

D.6 STANDARD PREPARATION:

Table d3

Standards Preparation

Compound or Standard Mix	Begin Conc. (µg/mL)	Aliquot (µL)	Total Vol. (mL)	Final Conc. (µg/mL)
Surrogate: RESTEK: a,a,a-TFT	2,000	1,250	25	100
L4-500STD RESTEK: Unleaded Gas Composite Standard	50,000	100	10	500

Appendix E

Summary of Tennessee method:

A flame ionization detector (FID) in conjuction with purge and trap methods, is used for detection and quantitation of gasoline range organics (GRO). The range will be defined as the sum of hydrocarbons falling between 2-methyl pentane and 1,2,4-trimethylbenzene.

Specific information relating to method:

E.1 STOCK STANDARDS:

e.1.1 Gasoline Component Standard. (see table e.3)

e.1.2 Unleaded Gasoline Composite Standard.

e.1.3 Surrogate- Triflourotoluene.

E.2 CALIBRATION INFORMATION:

e.2.1 A correlation coefficient of 0.990 or higher should be obtained. Alternatively, the average response factor can be used if a 20% or less percent relative standard deviation is achieved.

e.2.2 A daily continuing calibration standard must be within +/-25 % drift of initial calibration. Otherwise, another initial calibration will need to run.

E.3 QUALITY CONTROL:

Note: The QC acceptance criteria for spikes and surrogates are derived statistically per SW846 and are used to determine if analytical batches are in control. The recoveries are monitored by_the use of Control Charts and may be found in the SWL Blue Book.

e. 3.1 Calculated control limits

Table e.1
TPH Control limits

Matrix	Surrogate TFT	Laboratory Control Spike	Relative Percent Difference	Matrix Spike	Relative Percent Difference
Water	49-133	64-110	19%	55-111	21%
Soil	21-153	64-110	19%	41-117	22%

e. 3.2 Quantification standards will be made up using a gasoline component standard made up of 10 components (see table e.3) which will also serve as a retention time window-defining mix. Quantification will be based on a comparison using the total area of the component standard range. QC samples are spiked with a gasoline control standard (unleaded gasoline composite).

E.4 COMPOUND AND METHOD RECOVERY LIMIT:

Table e.2
Recommended Practical Quantitation Limits

Compounds	Water (ug/l)	Soil(ug/kg)
Total Gasoline Range Organics	20	20

E.5 STANDARD PREPARATION:

Table e3

Standards Preparation

Compound or Standard Mix	Begin Conc. (µg/mL)	Aliquot (µL)	Total Vol. (mL)	Final Conc. (µg/mL)
Surrogate: RESTEK: a,a,a-TFT	2,000	1,250	25	100
L4-500STD AccuStandard: Gasoline Component Standard (GRH-002S)	10,000	500	10	500
2-Methylpentane	1,500			75
2,2,4-Trimethylpentane	1,500			75
1,2,4-Trimethylbenzene	1,000			50
Heptane	500			25
Benzene	500			25
Toluene	1,500			75
Ethylbenzene	500			25
m-Xylene	1,000			50
p-Xylene	1,000			50
o-Xylene	1,000			50