

TURBIDIMETRIC SCREENING METHOD FOR TOTAL RECOVERABLE  
PETROLEUM HYDROCARBONS IN SOIL

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

## 1.0 SCOPE AND APPLICATION

1.1 This method may be used to screen soil samples to determine the total amount of recoverable petroleum hydrocarbon contamination in soil including a wide range of fuels, oils, and greases. The turbidimetric approach in this method is designed to quickly screen soil samples using a system calibrated with a blank and a single calibration standard.

1.2 The definition of total recoverable petroleum hydrocarbons for this method can be found in Sec. 3.0.

1.3 This screening technique is specifically designed to be used in the field but may also have some screening applications in the laboratory. The system analysis range is 10 - 2000 ppm for most hydrocarbons.

1.4 This method is considered a screening technique because of the broad spectrum of hydrocarbons it detects. The method may be especially useful in quickly determining that a site does not contain hydrocarbon contamination. However, it cannot be used to determine specific hydrocarbon compounds or groups of compounds that may be part of a larger hydrocarbon mixture. As with other screening techniques, it is advisable to confirm a certain percentage of both positive and negative test results, especially when near or above a regulatory action limit or when the presence of background or interfering hydrocarbons is suspected. The limitations of this procedure are described in more detail in Sec. 4.0 (Interferences).

1.5 This method does not address the evaporation of volatile petroleum hydrocarbon mixtures (i.e. gasoline) during sample collection, preparation, and analysis. Although the screening kit can be used to qualitatively detect volatile hydrocarbons, it is NOT recommended that the system be used to quantitatively determine volatile petroleum hydrocarbons unless evaporation during sample handling is addressed, appropriate response factor corrections are made, and method performance is demonstrated on real world samples.

1.6 Prior to employing this method, analysts are advised to consult the manufacturer's instructions for additional information on quality control procedures, development of QC

acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.7 Use of this method is restricted to use by, or under supervision of, appropriately experienced and trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

## 2.0 SUMMARY OF METHOD

2.1 A sample of soil is extracted with a solvent mixture composed primarily of methanol. The resulting mixture is allowed to settle and the free liquid is decanted into the barrel of a filter-syringe assembly. The liquid is filtered through a filter into a vial containing an aqueous emulsifier development solution. The filtered sample is allowed to develop for 10 min. During the development, any hydrocarbons present precipitate out and become suspended in solution.

2.2 The developed sample is placed in a turbidimeter that has been calibrated using a blank and a single calibration standard. A beam of yellow light at 585 nm is passed through the sample and the scattering of light through the suspension at 90° is measured. The concentration of total recoverable petroleum hydrocarbons present is calculated relative to the standard curve.

## 3.0 DEFINITIONS

3.1 See Chapter One and the manufacturer's instructions for definitions that may be relevant to this procedure.

3.2 For the purpose of this method, the phrase "total recoverable petroleum hydrocarbons" is defined as those hydrocarbons that are recovered using the solvent-specific extraction procedure provided with this kit. Since there is no cleanup step to separate any co-extracted naturally occurring hydrocarbons from the petroleum hydrocarbons, elevated turbidimetric readings are likely without performing background correction. See Sec. 4.0 (Interferences) for additional details.

## 4.0 INTERFERENCES

4.1 This method is considered a screening technique because of the broad spectrum of hydrocarbons it detects. It cannot distinguish between co-extracted naturally occurring hydrocarbons and petroleum hydrocarbons. Using background correction and/or a selected response factor discussed in the manufacturer's instructions, an analyst may be able to eliminate some of the interferences caused by co-extracted naturally occurring hydrocarbons. However, it is very difficult to find a truly clean, representative sample for use as a background.

4.2 This method was shown to be susceptible to interference from vegetable oils (positive interference). It is anticipated that co-extracted naturally occurring oils from vegetative materials would be one of the most probable positive interferants found in the field. To demonstrate this interference, standard soil samples were spiked with corn oil at levels of 50 to 1000 ppm and tested with the PetroFLAG™ system. Soil samples spiked with mineral oil were also analyzed for comparison. These data indicate that, over the range tested, the slope of the PetroFLAG™ vegetable oil response is approximately 18% of the response of the mineral oil standard. Supporting data are presented in Table 2. These data are provided for guidance purposes only.

4.3 This method was shown to be susceptible to interference from water (negative interference). To demonstrate this interference, soils were spiked with diesel fuel at 100 ppm. The samples were then spiked with varying amounts of water, up to saturation. The samples were analyzed using the PetroFLAG™ system and the results were below that expected for the spike added. The low bias may be due to a decrease in extraction efficiency in samples containing large amounts of water, as a result of dilution of the extraction solvent. Supporting data are presented in Table 3. These data are provided for guidance purposes only.

4.4 This method was shown to NOT be significantly affected by up to 5% sodium chloride contamination. Supporting data are presented in Table 6. These data are provided for guidance purposes only.

4.5 This method was shown to NOT be significantly affected by up to 1000 ppm of common surfactants such as trisodium phosphate (TSP), soap, and sodium dodecyl sulfate (SDS). Supporting data are presented in Tables 7, 8, and 9. These data are provided for guidance purposes only.

4.6 Polycyclic aromatic hydrocarbons (PAHs) are a class of compounds present in many hydrocarbon mixtures that are detected by the PetroFLAG™ system. These compounds are often targeted because of their toxic characteristics and may be present individually as soil contaminants. However, the response of the individual PAHs varies greatly from compound to compound. Therefore, use of the PetroFLAG™ system to quantitate individual PAHs is not recommended without good knowledge of the site and after adjusting the analytical approach. Quantitation of PAHs as part of a larger hydrocarbon fraction, such as diesel fuel, is recommended. Supporting data are presented in Table 12. These data are provided for guidance purposes only.

4.7 The PetroFLAG™ analyzer can be used at temperatures from 4 °C to 45 °C. The analyzer is equipped with an on-board temperature sensor to measure the ambient temperature at which measurements are being made. The software uses this temperature reading to correct the optical drift caused by temperature fluctuations.

4.8 Temperature at which the calibration is run should be recorded because of the effect temperature has on the suspension. This can be done by taking a reading without inserting a vial. If, during sample analysis, the temperature fluctuates more than  $\pm 10$  °C from the temperature at the calibration, the calibration should be rerun at the new temperature.

## 5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A

reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

5.2 Safety practices appropriate for handling potentially contaminated hazardous or toxic samples and extraction solvents should be employed.

## 6.0 EQUIPMENT AND SUPPLIES

PetroFLAG™ Hydrocarbon Analysis System, (Dexsil Corporation, One Hamden Park Drive, Hamden, CT), or equivalent. Each commercially-available test kit will supply or specify the apparatus and materials necessary for successful completion of the test.

## 7.0 REAGENTS AND STANDARDS

Each commercially-available test kit will supply or specify the reagents necessary for successful completion of the test. Reagents should be labeled with appropriate expiration dates, and reagents should not be employed beyond such dates.

## 8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 See the introductory material to Chapter Four, "Organic Analytes."

8.2 Soil samples may be contaminated, and should therefore be considered hazardous and handled accordingly.

8.3 To achieve accurate analyses, soil samples should be well homogenized prior to testing. The hydrocarbons may not be evenly distributed in a soil sample and extensive mixing is necessary to assure homogeneity.

**CAUTION:** It is strongly recommended that any free aqueous liquid be decanted from samples prior to analysis with the PetroFLAG™ system. Free aqueous liquid will dilute the extraction solvent and produce a negative interference.

**NOTE:** When users of the PetroFLAG™ system wish to report their results on a dry weight basis, additional representative samples should be collected for percent moisture determination. See the extraction Methods 3540 or 3550 for the procedure for determining percent moisture.

## 9.0 QUALITY CONTROL

9.1 Follow the manufacturer's instructions for quality control procedures specific to the test kit used. Also, refer to Chapter One for additional guidance on quality assurance (QA) and quality control (QC) protocols that may be applicable. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results.

9.2 Use of replicate analyses, particularly when results indicate concentrations near the action level, is recommended to refine information gathered with the kit.

9.3 This method is intended for use as a screening procedure in either the field or a fixed laboratory. Wherever it is employed, a quality assurance program appropriate for a screening procedure should be employed as a means of documenting the quality of the resulting data.

## 10.0 CALIBRATION AND STANDARDIZATION

See the PetroFLAG™ Hydrocarbon Analyzer User's Manual for instruction on generating an initial calibration curve using the PetroFLAG™ analyzer. Contact the manufacturer for specific details on the calibration calculations programmed into the PetroFLAG™ analyzer.

## 11.0 PROCEDURE

Follow the manufacturer's instructions in the PetroFLAG™ Hydrocarbon Analyzer User's Manual to extract, develop, and analyze soil samples. Test kits used must meet the performance specifications for the intended application.

## 12.0 DATA ANALYSIS AND CALCULATIONS

Consult the PetroFLAG™ Hydrocarbon Analyzer User's Manual for the procedure used to generate concentration readings from samples using the PetroFLAG™ analyzer. Contact the manufacturer for specific details on the concentration calculations programmed into the PetroFLAG™ analyzer.

## 13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 In the case of this method (which may be used in either the field or the laboratory), any test kits used must be able to meet the performance specifications for the intended application. However, required performance criteria for a particular testing product may be included in the manufacturer's instructions.

13.3 Table 1 shows example precision data determined using the procedures in Chapter One. The procedure was modified slightly because the instrument automatically subtracts an average blank value for each analysis (blank analysis is part of the calibration procedure of the PetroFLAG™ test system). Two sets of seven samples each were prepared, one set spiked with 30 ppm of diesel fuel, and one set spiked with 30 ppm of used motor oil. The standard deviation (SD) of the results for each oil type were calculated. These data are provided for guidance purposes only.

13.4 Samples of a standard soil were prepared by spiking with either diesel fuel or used motor oil at 100 ppm intervals from 100 ppm to 1000 ppm. Each sample was analyzed in duplicate by the PetroFLAG™ system and by Methods 3550 and 8015. These data were analyzed using regression analysis. The results of the sample analysis and regression analysis are provided in Table 4. In addition, an analysis of variance (ANOVA) analysis was performed. The F-statistic from the ANOVA revealed a significant bias between the two methods, with the PetroFLAG™ providing consistently higher values for both types of contamination. The results confirm that the kit design is intentionally conservative, in that it favors a high bias in order to avoid reporting false negative results (Ref. 1). These data are provided for guidance purposes only.

13.5 Precision and bias were determined by analysis of variance (ANOVA) of the results obtained from spiked soil samples. Four sets of spiked samples were prepared, containing either diesel fuel or used motor oil at two different concentrations (200 and 1000 ppm). Each analyte at each concentration was analyzed in duplicate 10 times (e.g., 20 replicates of each). The results were transformed into recovery data. The ANOVA used these transformed data. The results are presented in Table 5. The F-statistic for the diesel fuel analysis indicate a slight day effect for these samples. The F-statistic seems to be driven more by the very low value of the mean square error within days rather than by any large value for the mean square error between days (Ref. 1). These data are provided for guidance purposes only.

13.6 The response of the PetroFLAG™ system to a soil spiked with 500 ppm of diesel fuel and 0 to 5% of dry sodium chloride is provided in Table 6 (Ref. 2). These data are provided for guidance purposes only.

13.7 The responses of the PetroFLAG™ system to a soil spiked with 500 ppm of diesel fuel and up to 1000 ppm of common surfactants such as trisodium phosphate (TSP), soap, and sodium dodecyl sulfate (SDS), are presented in Tables 7, 8, and 9 (Ref. 2). These data are provided for guidance purposes only.

13.8 Performance of the PetroFLAG™ system on anthracene from 100 to 2000 ppm and on creosote from 100 to 1000 ppm are presented in Tables 10 and 11, respectively. An explanation of the erratic performance of anthracene is provided in the Table 10 narrative (Ref. 2). These data are provided for guidance purposes only.

13.9 The performance of the PetroFLAG™ system for several PAHs relative to the mineral oil calibrator on soil is presented in Table 12 (Ref. 4). These data are provided for guidance purposes only.

13.10 Performance of the PetroFLAG™ system on Jet-A from 40 to 2810 ppm (Ref. 4) and on gasoline from 1000 to 4070 ppm (Ref. 2) are provided in Tables 13 and 14, respectively. An explanation of the performance of Jet-A and gasoline are provided in the narrative in Tables 13 and 14. These data are provided for guidance purposes only.

## 14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention

techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, <http://www.acs.org>.

14.3 This method does not use any halogenated solvents and may be used to help reduce the number of samples sent to the laboratory under certain project scenarios.

## 15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

## 16.0 REFERENCES

1. Data Validation Package, *Testing for Petroleum Hydrocarbons in Soil by Turbimetric Analysis*, PetroFLAG™ Test System, DEXSIL Corp., Hamden, CT.
2. Supplementary Validation Data, *Additional Analyte and Contaminant Testing Data for the PetroFLAG™ Hydrocarbon Analysis System*, DEXSIL Corp., Hamden, CT, August 24, 1995.
3. PetroFLAG™ Hydrocarbon Analyzer User's Manual, DEXSIL Corp., Hamden, CT.
4. Supplementary Data Validation Package III, *Additional Analyte Testing Data for Petroleum Hydrocarbons in Soil by Turbimetric Analysis - PetroFLAG™ Test System*, DEXSIL Corp., Hamden, CT, June 20, 1997.
5. Supplementary Data Validation Package IV, *Polycyclic Aromatic Hydrocarbon Response data for Method 9074 Petroleum Hydrocarbons in Soil by Turbimetric Analysis - PetroFLAG™ Test System*, DEXSIL Corp., Hamden, CT, August 22, 1997.

## 17. TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method.

TABLE 1

## EXAMPLE PRECISION FOR PetroFLAG™ TEST SYSTEM

| Trial #       | 30 ppm diesel fuel | 30 ppm motor oil |
|---------------|--------------------|------------------|
| 1             | 34                 | 35               |
| 2             | 24                 | 41               |
| 3             | 28                 | 40               |
| 4             | 34                 | 53               |
| 5             | 36                 | 46               |
| 6             | 32                 | 48               |
| 7             | 30                 | 42               |
| Average (ppm) | 31                 | 44               |
| SD (ppm)      | 4.1                | 5.9              |

Data taken from Reference 1.

These data are provided for guidance purposes only.



TABLE 2

## EXAMPLE RELATIVE RESPONSE OF VEGETABLE OILS AS AN INTERFERANT

| Analyte Spike Concentration (ppm) | Mineral Oil Response (ppm) | Vegetable Oil Response <sup>a</sup> (ppm) |
|-----------------------------------|----------------------------|---|
| 50                                | 55                         | 30  |
| 100                               | 100                        | 45  |
| 200                               | 189                        | 94  |
| 500                               | 504                        | 111                                       |
| 1000                              | 947                        | 208                                       |

- <sup>a</sup> The vegetable oil samples were analyzed using the PetroFLAG™ system set to response factor 10. The slope of the PetroFLAG™ vegetable oil response is approximately 18% of the response of the mineral oil standard. This means that a sample containing 5,560 ppm vegetable oil would provoke a response equivalent to that given by 1,000 ppm mineral oil.

Data taken from Reference 1. These data are provided for guidance purposes only.

TABLE 3

## EXAMPLE OF THE EFFECT OF WATER ON PetroFLAG™ RESULTS

| % Water Saturation (% Water) | % Recovery of Mineral Oil <sup>a</sup> |
|------------------------------|--|
| 0 (0)                        | 100                                    |
| 5 (1)                        | 94                                     |
| 25 (5)                       | 98                                     |
| 50 (10)                      | 95                                     |
| 100 (20)                     | 85                                     |

- <sup>a</sup> Soil sample spiked with 100 ppm of mineral oil. (Ref. 1)  
These data are provided for guidance purposes only.

TABLE 4

## EXAMPLE COMPARISON OF PetroFLAG™ AND GC TEST RESULTS

| Spike Conc.<br>(µg/g) | PetroFLAG™<br>(µg/g) |         | Methods 3550/8015<br>(µg/g) |         |         |
|-----------------------|----------------------|---------|-----------------------------|---------|---------|
|                       | Diesel Fuel          | Trial 1 | Trial 2                     | Trial 1 | Trial 2 |
| 100                   |                      | 112     | 116                         | 73      | 82      |
| 200                   |                      | 230     | 248                         | 158     | 156     |
| 300                   |                      | 312     | 370                         | 242     | 218     |
| 400                   |                      | 420     | 455                         | 299     | 275     |
| 500                   |                      | 538     | 564                         | 342     | 344     |
| 600                   |                      | 626     | 654                         | 460     | 439     |
| 700                   |                      | 774     | 790                         | 509     | 494     |
| 800                   |                      | 910     | 900                         | 612     | 607     |
| 900                   |                      | 1090    | 977                         | 678     | 614     |
| 1000                  |                      | 1180    | 1060                        | 646     | 649     |
| Corr Coef             |                      | 0.999   |                             | 0.992   |         |
| Slope                 |                      | 1.13    |                             | 0.679   |         |
| Intercept             |                      | -2.8    |                             | 30.5    |         |
| Motor Oil             | Trial 1              | Trial 2 | Trial 1                     | Trial 2 |         |
| 100                   | 121                  | 128     | 123                         | 82      |         |
| 200                   | 243                  | 292     | 200                         | 200     |         |
| 300                   | 381                  | 408     | 301                         | 275     |         |
| 400                   | 428                  | 497     | 341                         | 343     |         |
| 500                   | 531                  | 554     | 441                         | 452     |         |
| 600                   | 654                  | 668     | 534                         | 528     |         |
| 700                   | 717                  | 771     | 609                         | 652     |         |
| 800                   | 880                  | 883     | 711                         | 746     |         |
| 900                   | 931                  | 1050    | 835                         | 881     |         |
| 1000                  | 1010                 | 1100    | 887                         | 846     |         |
| Corr Coef             |                      | 0.998   |                             | 0.997   |         |
| Slope                 |                      | 1.02    |                             | 0.887   |         |
| Intercept             |                      | 50.9    |                             | 20.5    |         |

Data taken from Reference 1. These data are provided for guidance purposes only.

TABLE 5

## EXAMPLE ANOVA RESULTS FOR SPIKED PETROLEUM HYDROCARBON SAMPLES

| Analyte/Concentration | n  | Mean<br>( $\bar{x}$ ) | Variance<br>( $\sigma_{n-1}^2$ ) | Standard<br>Deviation ( $\sigma_{n-1}$ ) | Standard<br>Error ( $\sigma_{\bar{x}}$ ) |
|-----------------------|----|-----------------------|----------------------------------|--|--|
| Diesel, 200 ppm       | 20 | 1.09                  | 0.0059                           | 0.0768                                   | 0.0172                                   |
| Diesel, 1000 ppm      | 20 | 1.00                  | 0.00430                          | 0.0656                                   | 0.0147                                   |
| Motor Oil, 200 ppm    | 20 | 1.12                  | 0.00266                          | 0.0515                                   | 0.0115                                   |
| Motor Oil, 1000 ppm   | 20 | 0.937                 | 0.000919                         | 0.0303                                   | 0.00678                                  |

Data taken from Reference 1. These data are provided for guidance purposes only.

TABLE 6

EXAMPLE RESPONSE OF PetroFLAG™ SYSTEM WITH VARIOUS LEVELS OF SODIUM CHLORIDE <sup>a</sup>

|                           | % Sodium Chloride |     |     |     |     |
|---------------------------|-------------------|-----|-----|-----|-----|
|                           | 0                 | 0.5 | 1.0 | 2.0 | 5.0 |
| PetroFLAG™ Response (ppm) | 518               | 539 | 529 | 516 | 524 |

<sup>a</sup> A series of soil samples consisting of sand, clay, and topsoil was spiked with 500 ppm of diesel fuel and varying levels of dry sodium chloride (NaCl) from 0 to 5 percent. The samples were analyzed using the PetroFLAG™ system set to response factor 5 (Ref. 2).

These data are provided for guidance purposes only.

TABLE 7

EXAMPLE RESPONSE OF PetroFLAG™ SYSTEM WITH VARIOUS TSP CONCENTRATIONS <sup>a</sup>

|                           | TSP Concentration (ppm) |     |     |     |      |
|---------------------------|-------------------------|-----|-----|-----|------|
|                           | 0                       | 100 | 200 | 500 | 1000 |
| PetroFLAG™ Response (ppm) | 522                     | 511 | 512 | 500 | 492  |

<sup>a</sup> Response of the PetroFLAG™ system for soil containing 500 ppm of diesel fuel and various levels of trisodium phosphate (TSP), a common surfactant. The samples were analyzed using the PetroFLAG™ system set to response factor 5 (Ref. 2).

These data are provided for guidance purposes only.

TABLE 8

EXAMPLE RESPONSE OF PetroFLAG™ SYSTEM WITH VARIOUS  
SOAP CONCENTRATIONS <sup>a</sup>

|                           | Soap Concentration (ppm) |     |     |     |      |
|---------------------------|--------------------------|-----|-----|-----|------|
|                           | 0                        | 100 | 200 | 500 | 1000 |
| PetroFLAG™ Response (ppm) | 500                      | 494 | 488 | 502 | 528  |

<sup>a</sup> Response of the PetroFLAG™ system for soil containing 500 ppm of diesel fuel and various levels of soap (non-ionic and anionic surfactants). The samples were analyzed using the PetroFLAG™ system set to response factor 5 (Ref. 2).

These data are provided for guidance purposes only.

TABLE 9

EXAMPLE RESPONSE OF PetroFLAG™ SYSTEM WITH VARIOUS SDS CONCENTRATIONS  
<sup>a</sup>

|                           | SDS Concentration (ppm) |     |     |     |      |
|---------------------------|-------------------------|-----|-----|-----|------|
|                           | 0                       | 100 | 200 | 500 | 1000 |
| PetroFLAG™ Response (ppm) | 472                     | 474 | 488 | 486 | 496  |

<sup>a</sup> Response of the PetroFLAG™ system for soil containing 500 ppm of diesel fuel and various levels of sodium dodecyl sulfate, a surfactant. The samples were analyzed using the PetroFLAG™ system set to response factor 5 (Ref. 2).

These data are provided for guidance purposes only.

TABLE 10

EXAMPLE RESPONSE OF PetroFLAG™ SYSTEM WITH VARIOUS AMOUNTS  
OF ANTHRACENE<sup>a</sup>

|                           | Anthracene Conc. (ppm) |      |      |      |      |
|---------------------------|------------------------|------|------|------|------|
|                           | 100                    | 200  | 500  | 1000 | 2000 |
| PetroFLAG™ Response (ppm) | 798                    | 1380 | 1640 | 1380 | 1740 |

<sup>a</sup> Response of the PetroFLAG™ system for soil containing various levels of anthracene. The results show that the PetroFLAG™ system returns a strong response to anthracene. The response to anthracene is higher than the response to the calibrator, therefore, the meter displays a reading that over-estimates the concentration. For concentrations greater than 200 ppm, the turbidity developed exceeded the recommended level (i.e., a reading greater than 1000 on response factor 10). To obtain accurate results the user should rerun the sample using a smaller sample size. This will bring the results into linear range. The samples were analyzed using the PetroFLAG™ system set to response factor 10 (Ref. 2).

These data are provided for guidance purposes only.

TABLE 11

EXAMPLE RESPONSE OF PetroFLAG™ SYSTEM WITH VARIOUS AMOUNTS  
OF CREOSOTE<sup>a</sup>

|                           | Creosote Conc. (ppm) |     |     |      |
|---------------------------|----------------------|-----|-----|------|
|                           | 100                  | 200 | 500 | 1000 |
| PetroFLAG™ Response (ppm) | 103                  | 210 | 538 | 1040 |

<sup>a</sup> Response of the PetroFLAG™ system for soil containing various levels of creosote. The samples were analyzed using the PetroFLAG™ system set to response factor 8 (Ref. 2).

These data are provided for guidance purposes only.

TABLE 12

EXAMPLE RELATIVE RESPONSE OF PetroFLAG™ SYSTEM TO VARIOUS  
POLYCYCLIC AROMATIC HYDROCARBONS <sup>a</sup>

| Compound       | Spike Level in ppm<br>(Matrix Used) | PetroFLAG™ Reading<br>in ppm (Rf 10) | Response Relative to<br>Mineral Oil Calibrator |
|----------------|-------------------------------------|--------------------------------------|--|
| Anthracene     | 100 (Soil)                          | 798                                  | 8  |
| Benzo[a]pyrene | 50 (Soil)                           | 180                                  | 3.6  |
| Chrysene       | 16 (Solvent)                        | 172                                  | 11   |
| Fluoranthene   | 200 (Solvent)                       | 101                                  | 0.5  |
| Pyrene         | 200 (Solvent)                       | 216                                  | 1.1  |

<sup>a</sup> The data for anthracene and benzo(a)pyrene were generated by spiking each compound onto a composite sandy clay loam soil and homogenizing the sample for later analysis. The soil sample size was 10 g. The soil spiking procedure used for anthracene and benzo(a)pyrene produced inconsistent results for the other PAH compounds. These compounds (chrysene, fluoranthene, and pyrene), which are very soluble in the extraction solvent, were spiked directly into the extraction solvent and analyzed. All of the PAHs samples were analyzed on response factor 10 (the correct response factor for mineral oil). The data indicate that, for example, using a standard sample size analyzed on response factor 10 (the correct response factor for mineral oil), a 100 ppm anthracene sample read 798 ppm. The PetroFLAG™ response to the above analytes is equal to or greater than the calibrator in all cases except for fluoranthene which has a response equivalent to diesel fuel.

**NOTE:** When analyzing soils containing anthracene, benzo(a)pyrene, or chrysene the PetroFLAG™ meter will read over range for concentrations of 250, 550, and 180 ppm respectively. These soils can be analyzed using a 1 g sample size to increase the maximum quantifiable concentration.

These data are provided for guidance purposes only.

TABLE 13

EXAMPLE RESPONSE OF PetroFLAG™ SYSTEM WITH VARIOUS AMOUNTS OF JET-A<sup>a</sup>

|                           | Jet-A Conc. (ppm) |     |     |     |     |     |      |      |
|---------------------------|-------------------|-----|-----|-----|-----|-----|------|------|
|                           | 0                 | 40  | 79  | 198 | 397 | 793 | 1590 | 2780 |
| PetroFLAG™ Response (ppm) | 54                | 110 | 162 | 208 | 368 | 700 | 1590 | 2810 |

<sup>a</sup> Response of the PetroFLAG™ system for soil containing various levels of Jet-A. The composite soils were prepared from two types of clay-loam soil and sand. The component soils were air dried and sieved to remove particles larger than 850 µm and then mixed in the ratio 2:1:1, followed by tumbling for one hour. The soil was weighed out into 10 g aliquots. Each of the soil aliquots was spiked by direct injection of Jet-A fuel onto the soil using a microliter syringe, mixed, and analyzed by the PetroFLAG™ system with the instrument set to response factor 4. The coefficient of determination ( $r^2$ ) for the Jet-A data was 0.997, indicating that the PetroFLAG™ response was linear over the range 40 ppm to 2810 ppm (Ref. 4).

These data are provided for guidance purposes only.

TABLE 14

EXAMPLE RESPONSE OF PetroFLAG™ SYSTEM WITH VARIOUS AMOUNTS OF WEATHERED GASOLINE<sup>a</sup>

|                           | Weathered Gasoline Conc. (ppm) |      |      |      |
|---------------------------|--------------------------------|------|------|------|
|                           | 1000                           | 2040 | 3050 | 4070 |
| PetroFLAG™ Response (ppm) | 285                            | 1780 | 4340 | 6870 |

<sup>a</sup> Response of the PetroFLAG™ system for soil containing various levels of weathered gasoline (50% evaporated). The manufacturer recommends that PetroFLAG™ be used to qualitatively detect gasoline at these levels. It is not recommended that PetroFLAG™ be used quantitatively for gasoline unless significant response factor corrections are made and evaporation of the target hydrocarbons is addressed. The samples were analyzed using the PetroFLAG™ system set to response factor 2 (Ref. 2).

These data are provided for guidance purposes only.