



Designation: D 5831 – 02

Standard Test Method for Screening Fuels in Soils¹

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1. Scope

1.1 This test method is a screening procedure for determining the presence of fuels containing aromatic compounds in soils. If the contaminant fuel is available for calibration, the approximate concentration of the fuel in the soil can be calculated. If the contaminant fuel type is known, but the contaminant fuel is not available for calibration, an estimate of the concentration of the fuel in the soil can be determined using average response factors. If the nature of the contaminant fuel is unknown, this screening test method can be used to identify the possible presence of contamination.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 2777 Practice for the Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water²
- E 131 Terminology Relating to Molecular Spectroscopy³
- E 169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis³
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁴
- E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers³
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁴
- E 925 Practice for the Periodic Calibration of Narrow Band-Pass Spectrophotometers³

¹ This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01 on Sampling and Monitoring.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 03.06.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

3. Terminology

3.1 *Definitions*—For definitions of terms used in this screening test method, refer to Terminology E 131.

4. Summary of Test Method

4.1 A sample of soil is extracted with isopropyl alcohol, and the extract is filtered. The ultraviolet absorbance of the extract is measured at 254 nm. If the contaminant fuel is available for calibration, the approximate concentration of contamination is calculated. If the contaminant fuel type is known, but the contaminant fuel is not available for calibration, an estimate of the contaminant concentration is determined using average response factors. If the nature of the contaminant fuel is not known, the absorbance value is used to indicate the presence or absence of fuel contamination. Calcium oxide is added to the soil as a conditioning agent to minimize interferences from humic materials and moisture present in the soil. Particulate interferences are removed by passing the extract through a filter.

5. Significance and Use

5.1 This screening test method is intended primarily for field use to define the boundaries of soil contamination. If the contaminant fuel is available for calibration, the approximate concentration of the fuel in the soil can be calculated. If the fuel type is known, but the contaminant fuel is not available for calibration, an estimate of the contaminant fuel concentration can be calculated using average response factors. If the nature of the contaminant fuel is unknown, a contaminant concentration cannot be calculated, and the test method can only be used only to indicate the presence or absence of fuel contamination.

5.2 Fuels containing aromatic compounds, such as diesel fuel and gasoline, as well as other aromatic-containing hydrocarbon materials, such as crude oil, coal oil, and motor oil, can be determined by this test method. The quantitation limit for diesel fuel is about 75 mg/kg. Approximate quantitation limits for other aromatic-containing hydrocarbon materials that can be determined by this screening test method are given in Table 1. Quantitation limits for highly aliphatic materials, such as aviation gasoline and synthetic motor oil, are much higher than those for more aromatic materials, such as coal oil and diesel fuel.

TABLE 1 Approximate Quantitation Limits for Various Fuel Types in Soils Based on 0.036 AU

Material	Limit of Quantitation (LOQ), mg/kg
Coal Oil	21
Crude Oil	61
Diesel Fuel	75
Weathered Diesel Fuel	21
Used Motor Oil	162
Weathered Gasoline	170
Unleaded Gasoline	316
Jet Fuel JP-2	378
Motor Oil	533
Aviation Gasoline	1066
Synthetic Motor Oil	1382

NOTE 1—The quantitation limits listed in Table 1 are approximate values because in this test method, the quantitation limit can be influenced by the particular fuel type and soil background levels. For information on how the values given in Table 1 were determined, see Appendix X1. Data generated during the development of this screening test method and other information pertaining to this test method can be found in the research reports. (1,2)⁵

5.3 Extractable material, which scatters or absorbs light at 254 nm, is a potential interference for this screening test method.

5.4 This test method may not be applicable to soil located under coniferous trees, because pine tar and turpentine respond as fuel when tested by this test method, giving high blank absorbance values. See 8.1.2.1 and Note 3 for information on determining if this test method can be applied to a specific soil located under coniferous trees.

6. Apparatus

6.1 *Glass Bottles*, wide-mouth, 125-mL (4-oz) with polytetrafluoroethylene-lined lids.

6.2 *Portable Scale*, (for field testing) or laboratory balance, capable of weighing to 0.1 g.

6.3 *Portable Stirring Device*, (for field testing) or magnetic stir bar and stirrer, which result in motion of the solids during stirring.

6.4 *Syringes*, disposable, polyethylene or polypropylene, 10-mL capacity.

6.5 *Syringe Filters*, disposable, polytetrafluoroethylene, 0.45- μ m pore size, 25-mm diameter.

6.6 *Spectrometer*, set at 254 nm with a 1-cm path length, quartz cell (cuvette).

6.7 *Volumetric Flasks and Pipets*, for preparing standard solutions.

6.8 *Laboratory Balance*, capable of weighing to 0.0001 g.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all screening tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemi-

cal Society where such specifications are available.⁶ Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Calcium Oxide Powder, Reagent Grade*—Use calcium oxide powder, reagent grade dried at 900°C for 12 h and stored in a desiccator or tightly sealed glass container prior to use. This is a conditioning agent for removal of interferences caused by the presence of humic material or moisture, or both, in the sample.

7.3 *Isopropyl Alcohol, Reagent Grade*—The extraction solvent should have an absorbance value versus air that is less than 0.1. To maintain purity, the solvent should not be stored for longer than one week in a container having a composition that may leach UV-absorbing materials.

7.3.1 Transportation of isopropyl alcohol for field testing must comply with current Department of Transportation (DOT) regulations.

8. Procedure

8.1 Running Blank Analyses:

8.1.1 To ensure that the batch of conditioning agent, syringe, filter cartridge, and so forth, are not contributing to the absorbance reading, it is recommended that the procedure be performed as specified in 8.3 and 8.4, except using no soil and approximately 5 g of calcium oxide. If the resulting extract has an absorbance value greater than 0.03, the various components should be tested individually by contacting them with the extraction solvent, and the problem component(s) should be replaced.

8.1.2 In this procedure, the conditioning agent inhibits the extraction of most humic materials, and there is very little, if any, background from inorganic materials. It is recommended, however, that a blank soil sample be tested as specified in 8.3 and 8.4 by extracting contaminant-free soil of the same type and from the same general area as the site being studied. Approximately 5 g of calcium oxide should be used for this blank extraction. Results from the blank soil analysis can be used to provide information on the blank soil absorbance value, the amount of calcium oxide required to dry the soil and inhibit extraction of humic materials, and the time it takes the soil and calcium oxide to settle after stirring.

8.1.2.1 If the absorbance value of the first soil blank extract is less than 0.05, extraction of the soil samples at the site should be performed using 5 g of calcium oxide. If the absorbance value of the first soil blank extract is greater than 0.05, a second blank sample should be extracted using additional calcium oxide. As stated in 8.1.2, for the first blank sample, approximately 5 g of calcium oxide should be used. If a second blank analysis is required, approximately 10 g of calcium oxide should be added to the soil sample. If the absorbance value of the second blank extract is lower than for

⁵ The boldface numbers given in parentheses refer to a list of references at the end of this test method.

⁶ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

the first blank extract, but is still greater than 0.05, a third blank sample should be tested using approximately 15 g of calcium oxide. These steps can be repeated, increasing the amount of calcium oxide by approximately 5 g each time, until the blank absorbance value is less than 0.05. In this way, the amount of calcium oxide required to inhibit interferences from humic material and moisture in the soil can be determined. Excess calcium oxide will not affect the analysis results. If the absorbance of the value of the second blank extract is not decreased by the addition of 10 g of calcium oxide to the blank sample or if the addition of calcium oxide does not lower the absorbance of the blank extract to less than 0.05, even with the addition of a large quantity of conditioning agent, and the absorbance of the blank extract is less than 0.1, the blank absorbance value can be subtracted from the sample absorbance values. If this is done, blank samples from around the site should be tested to ensure that the blank soil absorbance is constant by ± 0.02 absorbance units. If the blank absorbance for the second blank is not decreased by the addition of 10 g of calcium oxide and the absorbance of the blank extract is greater than 0.1, or if blank, correction is not desired, use of an alternative non-UV-absorbing extraction solvent should be considered. If an alternative solvent is used, the steps described in 8.1.1 and 8.1.2 should be repeated using the different solvent.

8.1.2.2 Note the time required for the soil and calcium oxide to settle after stirring as determined in 8.1.2 or 8.1.2.1 by performing the blank soil analysis(es).

NOTE 2—An example of a non-UV-absorbing solvent that has been used in place of isopropyl alcohol in this method is *n*-heptane. Information on use of this solvent can be found in the research report. (2)

NOTE 3—In testing soil located under coniferous trees, it is recommended that if the blank absorbance value cannot be lowered to less than 0.05 by the addition of calcium oxide, the blank absorbance value should be subtracted from the sample absorbance values. However, as stated in 8.1.2.1, this should only be done if the blank absorbance is less than 0.1. If the blank absorbance is greater than 0.1, the method should not be used to test the soil.

8.1.3 Also, it is recommended that one spike should be run for every batch of samples or for every 20 samples, whichever is most frequent. A soil sample is spiked by adding 5 μL of diesel fuel or 25 μL of gasoline and shaking the bottle for 3 min. The extraction and analysis then are performed as outlined in 8.3.3-8.4.5. Recovery is calculated by comparing the absorbance of the extract from the spiked soil at 254 nm with the absorbance of a solution of 5 μL of diesel fuel or 25 μL of gasoline in 50 mL of isopropyl alcohol. After correction for any material appearing in the unspiked soil, the recovery should be within 20 % of the true value.

8.2 Preparation of Standard Solutions:

8.2.1 Weigh out 200 mg (weighed to ± 0.1 mg) of the fuel type of interest into a 100-mL volumetric flask and dilute to volume using isopropyl alcohol. This gives a 2000-mg/L standard stock solution. Other standard solutions can be prepared as needed by appropriate dilution of this stock solution. For example, to prepare a 200-mg/L solution of the fuel type of interest, pipet 5 mL of the stock solution into a 50-mL volumetric flask and dilute to volume using isopropyl alcohol. For work in the field, a standard stock solution can be

prepared by diluting 25 μL of a fuel standard (density can vary from ~ 0.75 – 0.90 g/mL) to 100 mL with isopropyl alcohol.

8.3 Sample Preparation:

8.3.1 Preweigh a 125-mL (4-oz), wide-mouth, glass sample collection bottle having a polytetrafluoroethylene-lined lid. Record the mass of the empty sample collection bottle to ± 0.1 g.

8.3.2 Add 5 g (weighed to ± 0.1 g) of soil directly to the preweighed sample collection bottle. Weigh the sample bottle-plus-sample, and record the mass of the soil sample added to the bottle to ± 0.1 g.

8.3.3 Add the appropriate amount of calcium oxide as determined in 8.1.2.1 to the soil. The calcium oxide should be prepared as specified in 7.2. Stir the soil and calcium oxide with a spatula until a uniform dry mixture is obtained.

8.4 Sample Extraction and Analysis:

8.4.1 Pour 50 mL of isopropyl alcohol into the sample bottle.

8.4.2 Stir the slurry for 3 min using a portable stirring device or magnetic stir bar and stirrer so that the solids are in motion during stirring. A shorter stirring time or hand shaking may decrease the extraction efficiency. Close attention should be paid to the extraction step to make sure that the solids are in motion.

8.4.3 Allow the soil slurry to settle for the length of time determined in 8.1.2 or 8.1.2.1, then remove the lid and draw the supernatant solution into a 10-mL disposable syringe. Attach a filter cartridge to the end of the syringe. Rinse the sample cuvette with filtered extract. Then fill the cuvette with filtered extract for analysis.

NOTE 4—If the soil slurry is not allowed to settle after extraction, the filter will clog, and use of multiple filters will be required.

8.4.4 Calibration procedures specific to the spectrometer being used to perform the absorbance measurements must be followed. Instrument instructions for spanning from 0 to 1 absorbance unit must be followed. Calibration is to be performed using isopropyl alcohol to zero the instrument, and if a calibration line is to be established, calibration standards prepared from the standard stock solution should be used (see 8.2.1). Calibration using three standards is recommended. Calibration curves are nonlinear above 1 AU (>90 % of the light absorbed). As a result, readings must be made below this level. In addition, the extract absorbance reading must fall between the absorbance readings of two calibration standards.

NOTE 5—For general information on the techniques most often used in ultraviolet analysis, see Practice E 169. For additional information on the performance of ultraviolet spectrophotometers, see Practice E 275. For information on evaluating the performance of an ultraviolet spectrophotometer to verify its suitability for continued routine use, see Practice E 925.

8.4.5 Read and record the absorbance of the extract at 254 nm.

8.4.6 Determine an approximate or estimated concentration of a known fuel type in the filtered extract.

8.4.6.1 If the contaminant fuel was used for calibration, an approximate concentration of the fuel in the extract can be calculated using a calibration line. Record this approximate concentration of the fuel in the extract in milligrams/litre.

8.4.6.2 If the contaminant fuel type is known, but the contaminant fuel was not used for calibration, an estimated concentration of the fuel type in the extract can be calculated by multiplying the absorbance of the extract by the reciprocal absorptivity for that fuel type (see Table 2 and Eq 1). Record this estimated concentration of the fuel in the extract in milligrams/litre.

$$\text{(Absorbance)} \times (1/\text{Absorptivity}) = \text{Estimated concentration of the fuel in the filtered extract (mg/L)} \quad (1)$$

NOTE 6—For information pertaining to the reciprocal absorptivity values (response factors), see Appendix X2 and Tables X1.1 and X2.1 of the appendix.

8.4.7 Convert the approximate or estimated concentration of fuel in the extract (see 8.4.6.1 or 8.4.6.2) to an approximate or estimated concentration of the fuel in the original soil sample in milligrams/kilograms by multiplying the concentration of the fuel in the extract in milligrams/litre by a factor representing the solvent volume in millilitres-to-sample mass in grams ratio used in the extraction, that is, a factor of ten is used for a solvent volume-to-soil mass ratio of 50 mL of isopropyl alcohol: 5 g of soil. If the extract is diluted, the appropriate correction must be made. Record the approximate/estimated concentration of the fuel in the soil sample in milligrams/kilograms.

8.4.8 If the nature of the fuel-type contaminant is unknown, the concentration of the contaminant can not be calculated. In this case, the absorbance of the extract at 254 nm (see 8.4.5) can be used to indicate the presence of fuel contamination in the soil.

9. Record

9.1 Record the following information:

- 9.1.1 Type of the fuel contaminant,
- 9.1.2 Mass of the empty sample collection bottle, g,
- 9.1.3 Mass of the sample bottle-plus-soil sample, g,
- 9.1.4 Mass of the soil sample, g,
- 9.1.5 Volume of isopropyl alcohol (solvent) used in the extraction, mL,
- 9.1.6 Solvent for zeroing spectrometer,
- 9.1.7 Calibration standard solutions and absorbance values at 254 nm,
- 9.1.8 One/absorptivity for the fuel type of interest, if the contaminant fuel is not used for calibration,
- 9.1.9 Absorbance of the soil sample extract at 254 nm,

TABLE 2 Reciprocal Absorptivities at 254 nm for a 1-cm Path Length Cell

Material	1/Absorptivity, mg/L/AU
Coal Oil	59
Crude Oil	169
Diesel Fuel	209
Weathered Diesel Fuel	58
Used Motor Oil	450
Weathered Gasoline	473
Unleaded Gasoline	877
Jet Fuel JP-2	1050
Motor Oil	1480
Aviation Gasoline	2960
Synthetic Motor Oil	3840

9.1.10 Approximate/estimated concentration of the fuel in the filtered extract, mg/L, and

9.1.11 Approximate/estimated concentration of the fuel in the soil sample, mg/kg,

9.1.12 Suggested data recording form for performing this screening procedure is given in Fig. 1.

10. Report

10.1 Report the indicated presence or absence of fuel contamination or approximate or estimated concentration of contaminant fuel in the sample. Contaminant concentration should be reported to two or three significant figures, depending on the number of significant figures of the soil mass and response factor.

11. Precision and Bias ⁷

11.1 Precision:

11.1.1 A collaborative study of this screening test method involving eight participants was conducted. Each participant tested seven materials in triplicate. The test materials were a sand spiked with three different concentrations of diesel fuel (Test Materials A, B, and C), an unspiked sand (Test Material D), an organic soil spiked with two different concentrations of diesel fuel (Test Materials E and F), and an unspiked organic soil (Test Material G). The absorbance values of three calibration standards, which were prepared by the participants, were also determined for generation of a calibration line by each participant. The collaborative study materials were tested for and met a specified criterion for homogeneity prior to being sent to the collaborative study participants.

11.1.2 The collaborative study participants used the absorbance values they recorded to calculate the approximate and estimated concentrations of diesel fuel in the test materials. The approximate concentrations were determined using a calibrated line, which was generated by each participant from analysis of their calibration standards. The estimated concentrations of diesel fuel in the test materials were calculated using a response factor of 209 mg/L/AU (see Table 2).

11.1.3 In the collaborative study, to keep the identity of the samples unknown, the participants were not given any information on sample type or if any of the samples were unspiked. As a result, the participants did not know they had blank data, which could be used to correct sample values for background (see 8.1.2.1). Calculations to correct the approximate and estimated spiked sample concentrations for concentrations reported in the blank materials were performed by the collaborative study coordinator using the data provided by the participants. The blank-corrected approximate and estimated concentration values calculated for the test materials are listed in Table 3 and Table 4.

11.1.4 Practices D 2777, E 177, and E 691 were used as guidance in performing statistical evaluation of the data listed in Table 3 and Table 4. The index used for expressing reproducibility and repeatability of this test method is the 95 % limit on the difference between two test results. The 95 % limit

⁷ A copy of the research report used to develop the precision statement is available from ASTM Headquarters. Request RR: D34-1011.

TABLE 3 Tabulation of Collaborative Study Data for the Fuels in Soils Screening Method: Blank-Corrected Approximate Concentrations of Diesel Fuel in the Test Materials, mg/Kg

Participant	Material				
	A	B	C	E	F
1	153	364	761	220	714
	167	407	881	200	673
	178	371	847	220	819
2	172	340	763	101	577
	156	366	770	85	598
	158	386	762	86	574
3	157	403	830	132	587
	159	403	841	120	641
	159	405	848	122	634
4	180	405	851	101	690
	185	414	874	113	687
	167	404	793	117	685
5	168	389	751	87	593
	152	358	768	87	609
	156	375	792	101	576
6	137	341	662	95	471
	170	378	763	116	597
	153	369	768	97	555
7	101	314	801	84	472
	107	322	721	112	500
	104	301	781	76	505
8	132	380	793	97	561
	107	395	957	98	540
	114	375	764	93	607

TABLE 4 Tabulation of Collaborative Study Data for the Fuels in Soils Screening Method: Blank-Corrected Estimated Concentrations of Diesel Fuel in the Test Materials, mg/Kg

Participant	Material				
	A	B	C	E	F
1	182	435	906	262	853
	199	460	1048	234	828
	212	442	1008	264	1085
2	216	424	949	127	727
	195	457	957	106	754
	197	481	948	108	724
3	177	455	937	150	663
	180	455	949	136	724
	179	457	958	138	716
4	212	498	1029	122	833
	218	508	1056	136	829
	197	495	960	142	827
5	204	473	914	107	723
	185	436	935	107	742
	189	457	965	123	702
6	165	409	794	114	566
	204	453	915	139	716
	183	443	921	116	666
7	128	394	1006	108	595
	135	401	914	141	637
	131	385	981	98	642
8	151	528	1115	137	783
	117	552	1335	138	750
	127	521	1065	131	851

the diesel-spiked organic soil is constant over the diesel concentration range tested (from 103 to 737 mg/Kg), and does not vary between approximate and estimated concentration determinations. It appears that the characteristics of the diesel-spiked organic soil mask any variations in the reproducibility precision of the test method related to concentration or approximate/estimated concentration determinations. If the 95 % reproducibility limits listed for the diesel-spiked sand in Table 7 are averaged, the result is an overall 95 % reproducibility limit of 32 %, which corresponds very closely to 35 %, the 95 % reproducibility limit for the diesel-spiked organic soil.

11.1.8 The repeatability precision (95 % repeatability limit) of the screening test method does not vary between the two materials (Table 7). It is slightly less at the lower approximate and estimated diesel concentrations of 103 and 179 mg/Kg, respectively. The repeatability precision of the test method does not vary between approximate and estimated concentration determinations at test levels between approximately 100 to 450 mg/Kg. However, at the higher concentrations, approximately 600 to 970 mg/Kg diesel fuel, there is a slight difference between the repeatability precision of the test method for approximate and estimated concentration determinations. Overall, the repeatability precision of the screening test method does not vary significantly with concentration nor between approximate and estimated concentration determinations for the diesel-spiked sand and organic soil.

11.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test

TABLE 5 Reproducibility Precision Statistics for the Screening Method for Fuels in Soils^A

Approximate Concentration Statistics for Testing the Sand			
\bar{x}	s_{Ra}^B	2.8 s_{Ra}	2.8 CV % $_{Ra}$
156	20	56	36 %
382	22	62	16 %
802	63	176	22 %
Estimated Concentration Statistics for Testing the Sand			
\bar{x}	s_{Re}^C	2.8 s_{Re}	2.8 CV % $_{Re}$
179	32	90	50 %
459	44	123	27 %
972	73	204	21 %
Approximate Concentration Statistics for Testing the Organic Soil			
\bar{x}	s_{Ra}	2.8 s_{Ra}	2.8 CV % $_{Ra}$
103	14	39	38 %
618	77	216	35 %
Estimated Concentration Statistics for Testing the Organic Soil			
\bar{x}	s_{Re}	2.8 s_{Re}	2.8 CV % $_{Re}$
125	15	42	34 %
737	85	238	32 %

^A Units are mg/Kg unless otherwise specified.

^B Reproducibility (between participants) standard deviation for determining approximate concentration.

^C Reproducibility (between participants) standard deviation for determining estimated concentration.

method for screening fuels in soils, no statement on bias is being made. Available reference materials vary in their aromaticity, and, as a result, may not give a value when tested by this



TABLE 6 Repeatability Precision Statistics for the Screening Method for Fuels in Soils^A

Approximate Concentration Statistics for Testing the Sand			
\bar{x}	s_{ra}^B	2.8 s_{ra}	2.8 CV % _{ra}
156	11	31	20 %
382	16	45	12 %
802	54	151	19 %
Estimated Concentration Statistics for Testing the Sand			
\bar{x}	s_{re}^C	2.8 s_{re}	2.8 CV % _{re}
179	13	36	20 %
459	17	48	10 %
972	46	129	13 %
Approximate Concentration Statistics for Testing the Organic Soil			
\bar{x}	s_{ra}	2.8 s_{ra}	2.8 CV % _{ra}
103	8	22	21 %
618	42	118	19 %
Estimated Concentration Statistics for Testing the Organic Soil			
\bar{x}	s_{re}	2.8 s_{re}	2.8 CV % _{re}
125	12	34	27 %
737	38	106	14 %

^A Units are mg/Kg unless otherwise specified.

^B Repeatability (within participant) standard deviation for determining approximate concentration.

^C Repeatability (within participant) standard deviation for determining estimated concentration.

TABLE 7 95 % Reproducibility^A and Repeatability^B Limits for Testing Diesel-Spiked Sand and Organic Soil Using the Screening Method for Fuels in Soils

Material: Diesel-Spiked Sand

Test Range, mg/Kg	95 % Reproducibility Limit (% of the test result)
156 (approximate)	36 %
382 to 802 (approximate)	19 % (16 %, 22 %)
179 (estimated)	50 %
459 to 972 (estimated)	24 % (24 %, 27 %)

Material: Diesel-Spiked Organic Soil

Test Range, mg/Kg	95 % Reproducibility Limit (% of the test result)
103 to 737 (approximate or estimated)	35 % (32 to 38 %)

Materials: Diesel-Spiked Sand and Organic Soil

Test Range, mg/Kg	95 % Repeatability Limit (% of the test result)
103 to 179 (approximate or estimated)	22 % (20 to 27 %)
382 to 459 (approximate or estimated)	11 % (10 %, 12 %)
618 to 802 (approximate)	19 % (19 %, 19 %)
737 to 972 (estimated)	14 % (13 %, 14 %)

^A Between participants.

^B Within participant.

test method that corresponds to a value determined by a test method using a different measurement principle.

12. Keywords

12.1 absorbance; contamination; extraction; field screening; fuels; soils



APPENDIXES

(Nonmandatory Information)

XI. APPROXIMATE QUANTITATION LIMITS

X1.1 In the research to develop this screening test method, a series of approaches to determine the LOQ for various fuel types were taken. For example, one approach to estimate the LOQ for diesel fuel was to multiply the standard deviation of several absorbance measurements of a diesel standard in isopropyl alcohol ($s = 0.00315$) by a factor of 10 (3,4) and convert the resulting absorbance value (0.0315) to concentration of diesel in soil using the equation:

$$\begin{aligned} & (\text{absorbance}) \times (1/\text{absorptivity}) \\ & \times (\text{solvent volume to sample mass ratio}) \\ & = \text{LOQ} \qquad \qquad \qquad \text{(X1.1)} \\ & (0.0315) \times (209 \text{ mg/L}) \times (10 \text{ mL/g}) \\ & = 66 \text{ mg/kg diesel in soil as the LOQ} \end{aligned}$$

X1.2 The 1/absorptivity value is given in Table X1.1. It is the average response factor for diesel determined using a portable photometer. A second approach was to use the 99 % confidence level one-sided t statistic.(5) The standard deviation of 34 blank absorbance values determined using a variety of soil types was calculated to be 0.016 AU. This value multiplied by a t value of 2.442 gives an absorbance of 0.039 AU, which converts to an estimated LOQ of 67 mg/kg for the particular diesel used (red can diesel listed in Table X1.1, 1/Absorptivity = 171 mg/L) or 81 mg/kg based on the average diesel response factor (Table X1.1, 1/Absorptivity = 209 mg/L). This approach depends on the soils used and the blank values measured. Next, to estimate the LOQ for diesel using the conditioning agent, calcium oxide, a series of blank extracts were generated using a variety of wet and dry soils. The standard deviation of the absorbance values of these extracts was 0.0135. Using the 99 % one-sided t statistic, $ts = 0.035$ AU (2.602×0.0135). Using this value, the LOQ for diesel based on the average response factor given in Table X1.1 (209 mg/L) is 73-mg/kg diesel. This approach, which is based on typical soil blank background levels using calcium oxide, is considered to be a more realistic estimate of the LOQ. Based on additional testing in the laboratory, a typical soil blank background absorbance reading using calcium oxide was determined to be 0.036 AU. As a result, this value and the average reciprocal absorptivity values given in Table 2 of this test method were used to calculate the approximate LOQ values for the fuel types studied. These are the values given in Table 1 of this test method. For example, for weathered gasoline, an approximate LOQ of 170 mg/kg is listed in Table 1. This value was calculated using the following equation:

$$\begin{aligned} & (0.036) \times (473 \text{ mg/L}) \times (10 \text{ mL/g}) \\ & = 170\text{-mg/kg weathered gasoline in soil} \qquad \qquad \text{(X1.2)} \end{aligned}$$

TABLE X1.1 Reciprocal Absorptivities at 254 nm for a 1-cm Path Length Cell

Material	1/Absorptivity, mg/L per AU-Portable Photometer
Diesel Fuel	
Texaco #1	120
Texaco #2	194
Pilot	123
CG	308
4-C	371
Phillips	176
Red Can	171
Total	206
Average:	209
Coal Oil	
WY SRCII-A	55.2
WY SRCII-B	62.2
Average:	58.7
Crude Oil	
Recluse	210
S. Swan Hill	169
Wasson	129
Gach Saran	187
Gulf X-27, 683A	196
Gulf X-26, 483A	172
Wilmington	149
Prudhoe Bay	143
Swan Hill	165
Average:	169
Used Motor Oil	
Sample A	512
Sample B	389
Average:	450
Motor Oil	
Mobil 10W-40	972
Exxon 10W-40	1590
Amoco 10W-40	2140
Havoline 10W-30	445
Amoco 10W-30	1950
Castrol 5W-30	1440
Castrol SAE-30	1840
Average:	1480
Synthetic Motor Oil	
Castrol 5W-50	7040
Slick 50	1650
Penzoil 5W-50	2840
Average:	3840
Other Fuels	
Aviation Gas	2960
Jet Fuel JP-2	1050
Unleaded Gasoline	877
Weathered Gasoline	473
2-Cycle Oil	1890

Because of variability in absorptivity between fuels of the same type (see Appendix X2) and variations in soil background levels, the LOQ values given in Table 1 can only be approximate values.

X2. RELATIVE RESPONSE FACTORS

X2.1 Different aromatic-containing materials will have different absorptivities at 254 nm. In addition, because there are so many different sources of these materials, aromaticity can vary between materials of the same type, such as diesel fuels, and aromaticity can also vary between batches of the same material. Listed in Table X1.1 are reciprocal absorptivities for various aromatic-containing materials measured using a portable photometer. Similar values for these materials were also measured using a laboratory scanning spectrophotometer. Those values are given in the research report describing the development of this test method.(1) Because the fuels in the soils screening test method were developed for use in the field, the data generated using the portable photometer are given in Table 2 of this test method. The variability in absorptivity between materials of the same type is the reason the absorptivity calculation to determine the concentration of contamination (see 8.4.6.2) gives only an estimated value.

X2.2 A very common fuel contaminant in soil is weathered diesel fuel. As diesel fuel in soil is subjected to bacterial degradation and weathering processes, the remaining fuel is more aromatic than the original material and less volatile.(6) These weathered fuels can contain heavier hydrocarbon materials (>C₂₂). Components in fuel above C₂₂ are not detected well by laboratory gas chromatography methods. However, for this method to be useful for screening soils contaminated with weathered diesel fuel, the results must correlate with laboratory results. For this reason, laboratory data from analysis of weathered-diesel contaminated soils using EPA Method 8015B (7) were used to calculate a reciprocal absorptivity value for weathered diesel fuel in soil. This value, which is 58 mg/L/AU, was calculated using data from analysis of nine soil samples collected from various locations at four different field sites. The reciprocal absorptivity values that were used to calculate the value of 58 mg/L/AU are shown in Table X2.1.

TABLE X2.1 Determination of a Weathered Diesel Fuel Reciprocal Absorptivity Value at 254 nm for a 1-cm Path Length Cell

Weathered-Diesel Contaminated Soil	1/Absorptivity, mg/L/AU ^A
Rocky Alaskan soil A	39
Rocky Alaskan soil B	75
Wet, sandy soil A	49
Wet, sandy soil B	26
Wet, sandy soil C	88
Southern soil A	78
Southern soil B	51
Wet clay A	30
Wet clay B	90
Average:	58

^ADetermined using laboratory data from analysis of soil samples using EPA Method 8015B.

X2.3 If site data from laboratory analyses are available, and if there is time, field analyses can be performed prior to the start of field work to determine an appropriate response factor (reciprocal absorptivity value) for the fuel contaminant at the particular site. In this way, a response factor that is specific to the contaminant fuel, soil type, laboratory method, and so forth, can be determined for use in screening soil samples from the site. However, in many cases, there is not time for such preparatory work prior to starting field work. As a result, care should be taken in selecting the appropriate response factor from the list given in Table 2, with particular consideration given to whether or not the fuel contamination is fresh or has undergone weathering or biodegradation processes, or both. Another factor to consider is whether the contamination is a mixture of one or more fuel types. If this is the case, and a site-specific response factor cannot be determined, the response factors for the individual fuel types in the mixture should be used to estimate the contaminant concentrations, and a decision on how to interpret the test results should be made.

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