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# Standard Practice for Screening Fuels in Soils<sup>1</sup>

This standard is issued under the fixed designation D5831; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 1. Scope

1.1 This practice is a screening procedure for assessing the presence of fuels containing aromatic compounds in soils. If a sample of the contaminant fuel is available, the concentration of the fuel in the soil can be determined. If the contaminant fuel type is known but a sample of the contaminant fuel is not available, an estimate of the concentration of the fuel in the soil can be made using average response factors based on composition of the fuel in the soil. If the kind of contaminant fuel is unknown, this screening method can be used to identify the presence of contamination.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

- [D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water](#)
- [D5681 Terminology for Waste and Waste Management](#)
- [E131 Terminology Relating to Molecular Spectroscopy](#)
- [E169 Practices for General Techniques of Ultraviolet-Visible](#)

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.05 on Screening Methods.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

## Quantitative Analysis

- [E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods](#)
- [E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers](#)
- [E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)
- [E925 Practice for Monitoring the Calibration of Ultraviolet-Visible Spectrophotometers whose Spectral Bandwidth does not Exceed 2 nm](#)

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this screening practice, refer to Terminologies [D5681](#) and [E131](#).

## 4. Summary of Practice

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 a sample of the contaminant fuel is available, the approximate concentration of contamination can be calculated. If the contaminant fuel type is known but a sample of the contaminant fuel is not available, an estimate of the contaminant concentration is determined using average response factors based on composition of the fuel in the soil. If the composition 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 practice is a screening procedure for determining the presence of fuels containing aromatic compounds in soils. If a sample of the contaminant fuel is available for use in calibration, the approximate concentration of the fuel in the soil can be calculated. If the fuel type is known but a sample of the contaminant fuel is not available for calibration, an estimate of the contaminant fuel concentration can be calculated using average response factors based on composition of the fuel in the soil. If the composition of the contaminant fuel is unknown, a contaminant concentration cannot be calculated, and this practice can only be used only to indicate the presence or absence of fuel contamination.

**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

**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

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 practice. 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 practice 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.

NOTE 1—The quantitation limits listed in **Table 1** are estimated values because in this practice, the quantitation limit can be influenced by the particular fuel type and soil background. For information on how the values given in **Table 1** were determined, see **Appendix X1**. Data generated during the development of this screening practice and other information pertaining to this practice can be found in the referenced research reports **(1, 2)**.<sup>3</sup>

5.3 When applying this practice to sites contaminated by diesel fuel, care should be taken in selecting the appropriate response factor from the list given in **Table 2**, with consideration given to whether or not the fuel contamination is fresh or has undergone weathering or biodegradation processes. See **Appendix X2**.

5.4 A consideration in using this practice is whether the contamination is a mixture of one or more fuel types. If this is the case, and a site-specific response factor (see **X2.3**) cannot be determined, the response factors for the individual fuel types in the mixture should be used to estimate contaminant concentrations.

5.5 Certain materials, such as asphalts and asphalt residuals and oils and pitch from trees and other vegetation, which respond as fuel when tested by the practice, give high blank absorbance values which may interfere with use of this practice. See **8.1.2.1** and **Note 3** for information on determining if this practice can be applied to a specific soil containing one or more of these types of materials.

5.6 Extractable material, which scatters or absorbs light at 254 nm, is a potential interference for this screening practice.

<sup>3</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.

## 6. Apparatus

6.1 *Glass Bottles*, wide-mouth, 125 mL 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 μm 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 Chemical Society where such specifications are available.<sup>4</sup> Other grades may be used, provided that the reagent is demonstrated to be 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.

<sup>4</sup> 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.

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 is not 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 do not contain substances that contribute to the absorbance reading, it is recommended that the procedure be performed as specified in 8.3 and 8.4, in the absence of soil and using 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 for contamination by contacting them with the extraction solvent. Contaminated reagents are replaced prior to performing sample analysis.

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 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 soil blank extract is less than 0.05, extraction of soil samples at the site can be performed using 5 g of calcium oxide. If the absorbance value of the soil blank extract is greater than 0.05, a second blank sample should be extracted using additional calcium oxide. If a second blank analysis is required, approximately 10 g of calcium oxide should be added to the sample. If the absorbance value of the second blank extract is lower than for 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  $\pm 0.02$  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 alternative solvent.

8.1.2.2 Record 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 is *n*-heptane. Information on use of this solvent can be found in the research report referenced at the end of this standard (2).

NOTE 3—In testing soil suspected of containing asphaltic materials or oils or pitch from trees or other vegetation, 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, this method cannot be used to test the soil.

8.1.3 It is recommended that one spike is run for every batch of samples or for every 20 samples, whichever is most frequent. A soil sample is spiked by adding 5  $\mu$ L of diesel fuel or 25  $\mu$ L 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  $\mu$ L of diesel fuel or 25  $\mu$ L 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 theoretical value.

### 8.2 *Preparation of Standard Solutions:*

8.2.1 Weigh out 200 mg (weighed to  $\pm 0.1$  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  $\mu$ L of a fuel standard (density can vary from  $\sim 0.75$  to 0.90 g/mL) to 100 mL with isopropyl alcohol.

### 8.3 *Sample Preparation:*

8.3.1 Preweigh a 125 mL, wide-mouth glass sample collection bottle having a polytetrafluoroethylene-lined lid. Record the mass of the empty sample collection bottle to  $\pm 0.1$  g.

8.3.2 Add  $5 \pm 0.1$  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  $\pm 0.1$  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 ensure 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 curve is to be established, calibration standards prepared from the standard stock solution are used (see 8.2.1). Calibration using a minimum of three standard concentrations 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 maximum and minimum absorbance readings of the calibration curve.

NOTE 5—For general information on the techniques commonly used in ultraviolet analysis, see Practice E169. For additional information on the performance of ultraviolet spectrophotometers, see Practice E275. For information on evaluating the performance of an ultraviolet spectrophotometer to verify its suitability for continued routine use, see Practice E925.

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 curve. Record this approximate concentration of the fuel in the extract in mg/L.

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 mg/L.

$$(\text{Absorbance}) \times (1/\text{Absorptivity}) = \text{Estimated concentration of the}$$

fuel in the filtered extract (mg/L)

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

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 mg/kg by multiplying the concentration of the fuel in the extract in mg/L 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 to 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 mg/kg.

8.4.8 If the type of fuel 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 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,

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 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<sup>5</sup>

11.1 Precision:

11.1.1 A collaborative study of this screening practice 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 curve by each

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D34-1011. Contact ASTM Customer Service at service@astm.org.



**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

**TABLE 5** Reproducibility Precision Statistics for the Screening Method for Fuels in Soils<sup>A</sup>

Approximate Concentration Statistics for Testing the Sand			
$\bar{x}$	$s_{Ra}^B$	2.8 $s_{Ra}$	2.8 CV % <sub>Ra</sub>
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 % <sub>Re</sub>
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 % <sub>Ra</sub>
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 % <sub>Re</sub>
125	15	42	34 %
737	85	238	32 %

<sup>A</sup> Units are mg/kg unless otherwise specified.

<sup>B</sup> Reproducibility (between participants) standard deviation for determining approximate concentration.

<sup>C</sup> Reproducibility (between participants) standard deviation for determining estimated concentration.

(3) calculating reproducibility (between participants) standard deviation ( $s_R$ ), (4) calculating repeatability (within participant) standard deviation ( $s_r$ ), (5) determining the 95 % reproducibility limit (2.8  $S_R$  or 2.8 CV %<sub>R</sub>, and (6) determining the 95 % repeatability limit (2.8  $S_r$  or 2.8 CV %<sub>r</sub>).

11.1.5 The reproducibility and repeatability precision statistics calculated for this practice using the collaborative study data are listed in **Tables 5 and 6**. Based on these data, the 95 % reproducibility and repeatability limits for testing the diesel-spiked sand and diesel-spiked organic soil using this practice were determined. These limits are listed in **Table 7**.

11.1.6 The data listed in **Table 7** give information on the reproducibility and repeatability of this screening practice when it is applied to a sand and organic soil contaminated with various concentrations of diesel fuel. The data are specific to the test materials used in the study. For other soil types and fuel contaminants, these data may not apply.

11.1.7 The information given in **Table 7** shows that the reproducibility precision (95 % reproducibility limit) of this practice varies between the two test materials. The reproducibility of the practice when applied to the diesel-spiked sand varies with diesel concentration and also between approximate and estimated concentration determinations. As expected, the reproducibility precision of this practice at a lower diesel concentration in the sand (~160 mg/kg) is less than at higher concentrations (~400 to 970 mg/kg). The reproducibility precision of the method for testing the diesel-spiked sand is approximately two times higher at the higher diesel concentrations. The data in **Table 7** show that the reproducibility precision of the screening practice for testing 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