



Designation: ~~D5831-02~~ Designation: **D 5831 – 03 (Reapproved 2008)**

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

This standard is issued under the fixed designation D 5831; 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 reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 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:<sup>2</sup>

- D 2777 Practice for the Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
- E 131 Terminology Relating to Molecular Spectroscopy
- E 169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near-Infrared Spectrophotometers
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- E 925 Practice for the Periodic Calibration of Narrow Band-Pass Spectrophotometers<sup>3</sup> Practice for Monitoring the Calibration of Ultraviolet-Visible Spectrophotometers whose Spectral Slit Width does not Exceed 2 nm

### 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 a screening procedure for field use to define the boundaries of soil contamination.~~ 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 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

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<sup>3</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.

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.

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)

~~5.3 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.<sup>3</sup>~~

~~5.3 When applying this test method 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 factor to consider in using this test method 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 Appendix X2, Section 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 method giving high blank absorbance values, may interfere with use of this test method. See 8.1.2.1 and Note 3 for information on determining if the test method 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 test method.~~

## 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- $\mu$ 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 Chemical Society

<sup>2</sup> Annual Book of ASTM Standards, Vol 03.06.

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

**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

where such specifications are available.<sup>4</sup> 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 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 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).

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02-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.

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~~, suspected of containing asphalt 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, 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  $\mu\text{L}$  of diesel fuel or 25  $\mu\text{L}$  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  $\mu\text{L}$  of diesel fuel or 25  $\mu\text{L}$  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 true 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  $\mu\text{L}$  of a fuel standard (density can vary from  $\sim 0.75$ – $0.90$  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  $\pm 0.1$  g.

8.3.2 Add 5 g (weighed to  $\pm 0.1$  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 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,

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 <sup>5</sup>

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 means that approximately 95 % of all pairs of test results from users similar to the participants in the collaborative study can be expected to differ in absolute value by less than  $1.960 (2)^{1/2}s$ , which corresponds to 2.8 s or 2.8 CV % (percent coefficient of variation) (Practice E 177). The steps involved in the data analysis were (1) eliminating “outlier” participants (participants that are so consistently high or low that their results are unreasonable), (2) eliminating individual “outlier” data points, (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  $2.8 CV \%_R$ ), and (6) determining the 95 % repeatability limit ( $2.8 S_r$  or  $2.8 CV \%_r$ ).

<sup>6</sup> The boldface numbers given in parentheses refer to a list of references at the end of this test method.

<sup>5</sup> A copy of the research report used to develop the precision statement is available from ASTM Headquarters. Request RR: D34-1011.

