



## 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:

- D 2777 Practice for the Determination of Precision and Bias of Applicable Test Methods of Committee D-19 on Water<sup>2</sup>
- E 131 Terminology Relating to Molecular Spectroscopy<sup>3</sup>
- E 169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis<sup>3</sup>
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods<sup>4</sup>
- E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers<sup>3</sup>
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>4</sup>
- E 925 Practice for the Periodic Calibration of Narrow Band-Pass Spectrophotometers<sup>3</sup>

### 3. Terminology

3.1 *Definitions*—For definitions of terms used in this

<sup>1</sup> This test method 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> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.06.

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

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.

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

**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
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

generated during the development of this screening test method and other information pertaining to this test method can be found in the research report.<sup>5</sup>

5.3 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 all solids during stirring.

6.4 *Syringe*, disposable, plastic, 10-mL capacity.

6.5 *Syringe Filters*, disposable, 0.45- $\mu$ m, in 25-mm polytetrafluoroethylene cartridges.

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>6</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.

<sup>5</sup> Schabron, J. F., Niss, N. D., Hart, B. K., and Sorini, S. S., "Remote Chemical Sensor Development: A New Field Screening Method for Soil Fuel Contamination," Laramie, WY, WRI-95-R016, 1995.

<sup>6</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.

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

## 8. Procedure

### 8.1 Preparation of Standard Solutions:

8.1.1 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  $\sim 0.75$ – $0.90$  g/mL) to 100 mL with isopropyl alcohol.

### 8.2 Sample Preparation:

8.2.1 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.

8.2.2 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.

8.2.3 Add approximately 5 g of calcium oxide, prepared as specified in 7.3, to the soil, and stir with a spatula until a uniform dry mixture is obtained. If the soil contains an excessive amount of water ( $>50\%$ ), add approximately 5 g more of calcium oxide.

### 8.3 Sample Extraction and Analysis:

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

8.3.2 Stir the slurry for 3 min using a portable stirring device or magnetic stir bar and stirrer so that all solids are in motion during stirring. A shorter stirring time or hand shaking may decrease the extraction efficiency.

8.3.3 Allow the soil slurry to settle briefly, 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.

8.3.4 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 8.1.1). 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 2—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.

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

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

8.3.6.1 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.

8.3.6.2 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 3—Information pertaining to the reciprocal absorptivity values are listed in Table X1.1 of the appendix.

8.3.7 Convert the approximate or estimated concentration of fuel in the extract (see 8.3.6.1 or 8.3.6.2) 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.

8.3.8 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 8.3.5) can be used to indicate the presence of fuel contamination in the soil.

8.3.9 To ensure that the batch of conditioning agent, plastic syringe, filter cartridge, and so forth, are not contributing to the absorbance reading, it is recommended that the procedure also

should be performed as specified, except with no soil. If significant absorbance is noted, the various components should be tested individually by contacting them with isopropyl alcohol, and the problem component(s) should be replaced.

8.3.10 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 should be tested by extracting contaminant-free soil of the same type and from the same general area as the site being studied. If any absorbance is measured for the blank, the sample values should be corrected for this procedure.

8.3.11 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 8.2.3-8.3.5. 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.

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,
  - 9.1.11 Approximate/estimated concentration of the fuel in the soil sample, mg/kg, and,
  - 9.1.12 Suggested data recording form for performing this screening procedure as 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.

11. Precision and Bias <sup>7</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

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

Material	1/Absorptivity, mg/L/AU
Coal Oil	58.7
Crude Oil	169
Diesel Fuel	209
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

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

