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Standard Test Method for Naphthalene Hydrocarbons in Aviation Turbine Fuels by Ultraviolet Spectrophotometry[1]

This standard is issued under the fixed designation D1840; 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 (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination, by ultraviolet spectrophotometry, of the total concentration of naphthalene, acenaphthene, and alkylated derivatives of these hydrocarbons in jet fuels. This test method is designed to analyze fuels containing not more than 5 % of such components and having end points below 315°C (600°F); however, the range of concentrations used in the interlaboratory test programs which established the precision statements for this test method were 0.03 to 4.25 volume % for Procedure A, and 0.08 to 5.6 volume % for Procedure B. This test method determines the maximum amount of naphthalenes that could be present.

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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific warning statements, see 8.1 and 8.2.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E131 Terminology Relating to Molecular Spectroscopy

E169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Terminology

3.1 *Definitions:*

3.1.1 Definitions of terms and symbols relating to absorption spectroscopy in this test method shall conform to Terminology E131. Terms of particular significance are the following:

3.1.2 *radiant energy, n*—energy transmitted as electromagnetic waves.

3.1.3 *radiant power, P, n*—rate at which energy is transported in a beam of radiant energy.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *absorbance, A, n*—the molecular property of a substance that determines its ability to take up radiant power, expressed by

$$A = \log_{10}(1/T) = -\log_{10}T \quad (1)$$

where:

T = transmittance as defined in 3.2.5.

3.2.1.1 *Discussion*—It may be necessary to correct the observed transmittance (indicated by the spectrophotometer) by compensating for reflectance losses, solvent absorption losses, or refraction effects.

3.2.2 *absorptivity, a, n*—the specific property of a substance to absorb radiant power per unit sample concentration and path length, expressed by

$$a = A/bc \quad (2)$$

where:

A = absorbance defined in 3.2.1,
b = sample cell path length, and
c = quantity of absorbing substance contained in a unit volume of solvent.

3.2.2.1 *Discussion*—Quantitative ultraviolet analyses are based upon the absorption law, known as Beer's law. The law states that the absorbance of a homogeneous sample containing an absorbing substance is directly proportional to the concentration of the absorbing substance at a single wavelength, expressed by

$$A = abc \quad (3)$$

where:

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.04.0F on Absorption Spectroscopic Methods.

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

A = absorbance as defined in 3.2.1,
a = absorptivity as defined in 3.2.2,
b = sample cell path length, and
c = quantity of absorbing substance contained in a unit volume of solvent.

3.2.3 *concentration, c, n*—the quantity of naphthalene hydrocarbons in grams per litre of *iso*octane.

3.2.4 *sample cell path length, b, n*—the distance, in centimetres, measured in the direction of propagation of the beam of radiant energy, between the surfaces of the specimen on which the radiant energy is incident and the surface of the specimen from which it is emergent.

3.2.4.1 *Discussion*—This distance does not include the thickness of the cell in which the specimen is contained.

3.2.5 *transmittance, T, n*—the molecular property of a substance that determines its transportability of radiant power expressed by

$$T = P/P_o \tag{4}$$

where:

P = radiant power passing through the sample, and
P_o = radiant power incident upon the sample.

4. Summary of Test Method

4.1 The total concentration of naphthalenes in jet fuels is determined by measurement of the absorbance at 285 nm of a solution of the fuel at known concentration.

5. Significance and Use

5.1 This test method for naphthalene hydrocarbons is one of a group of tests used to assess the combustion characteristics of aviation turbine fuels of the kerosene boiling range. The naphthalene hydrocarbon content is determined because naphthalenes, when burned, tend to have a relatively larger contribution to a sooty flame, smoke, and thermal radiation than single ring aromatics.

6. Interferences

6.1 Interferences add to the apparent naphthalene content. Phenanthrenes, dibenzothiophenes, biphenyls, benzothiophenes, and anthracenes interfere if present. The end point limitation of 315°C will minimize this interference except for benzothiophenes and biphenyls. The contribution to measured naphthalene content by the presence of 1 % of such interfering compounds can be estimated from Table 1.

6.2 Saturated hydrocarbons, olefins, thiophenes, and alkyl or cycloalkyl derivatives of benzene will not interfere.

TABLE 1 Interfering Compounds

Type of Interfering Compound	Error in Percentage of Naphthalenes Caused by 1 % Interfering Compound
Phenanthrenes	2
Dibenzothiophenes	2
Biphenyls	1
Benzothiophenes	0.6
Anthracenes	0.1

7. Apparatus

7.1 *Spectrophotometer,* equipped to measure the absorbance of solutions in the spectral region 240 to 300 nm with a spectral slit width of 1 nm or less. Wavelength measurements shall be repeatable and known to be accurate within 0.1 nm or less as measured by mercury emission line at 253.65 nm or the absorption spectrum of either holmium oxide glass at 287.5 nm or holmium oxide solution at 287.1 nm. At the 0.4 absorbance level in the spectral region between 240 and 300 nm, absorbance measurements shall be repeatable within ±0.5 % or better. In the absorbance range encompassing 0.2 to 0.8, the photometric accuracy shall not differ by more than ±0.5 % of samples whose absorbance has been established by a standardizing laboratory.

7.1.1 *Discussion*—Many manufacturers provide secondary standards, traceable to NIST primary standards, for checking the wavelength accuracy and photometric accuracy of spectrophotometers. These materials may be used to verify spectrophotometer performance provided that they have been recalibrated periodically as recommended by the manufacturer.

7.2 It shall be initially and thereafter periodically demonstrated that an instrument can be operated in a manner to give test results equivalent to those described in 7.1.

NOTE 1—For recommended methods of testing spectrophotometers to be used in this test method, refer to Practice E275. Other preferred alternatives to those in 7.1 are potassium dichromate in perchloric acid (NIST SRM 935 series as described in Practice E275) for photometric accuracy and a 20 mg/L high (>99 %) purity naphthalene in spectroscopic grade *iso*octane for wavelength accuracy. The latter has a minor maximum at 285.7 nm. The naphthalene solution shall not be used for photometric accuracy.

7.3 *Vitreous Silica Cells,* two, having path lengths of 1.00 ± 0.005 cm.

7.4 *Pipets,* Class *A*.

7.5 *Lens Paper.*

7.6 *Balance,* capable of taring or weighing 100 g to the nearest 0.0001 g. The balance shall be accurate to ±0.0002 g at a 100-g load.

8. Solvents

8.1 *Spectroscopic 2,2,4 Trimethylpentane (Isooctane).* (**Warning**—*Iso*octane is extremely flammable, harmful if inhaled.)

NOTE 2—Spectroscopic-grade *iso*octane is available commercially. Technical-grade *iso*octane is a satisfactory base stock for the preparation of spectroscopic solvent. Allow about 4 or 5 L of this material to percolate through a column of activated silica gel (74 µm) 50.8 to 76.2 mm in diameter and 0.6 to 0.9 m in depth. Collect only the portion of the solvent that has a transmission compared to distilled water greater than 90 % over the entire spectral range from 240 to 300 nm. Store in scrupulously clean glass-stoppered bottles and always keep covered. In general it will be best to use a fresh portion of silica gel in preparing a new batch of solvent. However the gel can be reactivated by pouring 500 mL of acetone through the column, draining, drying by suction, and heating the gel in thin layers in an oven at 400°C until white color is restored. Activated silica gel is stored in closed containers.

8.2 *Solvents for Cleaning Cells*—Acetone or ethyl alcohol (**Warning**—Acetone and ethyl alcohol are extremely flammable and can be harmful if inhaled), with residue after evaporation no greater than 10 mg/kg.

NOTE 3—The 10 mg/kg is the American Chemical Society (ACS) reagent grade maximum specification. An ACS reagent grade solvent may be used without further testing.

9. Calibration and Standardization

9.1 Instead of direct calibration of the spectrophotometer with known naphthalenes, the average absorptivity of the C_{10} to C_{13} naphthalenes at 285 nm can be taken at 33.7 L/g·cm. The data used to calculate this average are given in Table 2.

10. Procedure A—Serial Dilution

NOTE 4—The user may use alternative Procedure B if preferred.

10.1 For recommended techniques, refer to Practices E169. Check carefully sections on handling and cleaning of cells and glassware, instrument adjustments, and method of absorbance measurement.

10.2 Prepare three dilutions of the sample as follows:

10.2.1 *First Dilution*—If the sample is more volatile than *iso*octane, add 10 to 15 mL of spectroscopic *iso*octane to a clean, dry, glass-stoppered, 25 mL volumetric flask. Weigh out approximately 1 g of sample in the flask, dilute to volume with spectroscopic solvent, and mix thoroughly. If the sample is less volatile than *iso*octane, weigh out approximately 1 g of sample in the flask, dilute to volume with spectroscopic solvent, and mix thoroughly.

10.2.2 *Second Dilution*—Pipet 5.00 mL of the first dilution into a 50-mL glass-stoppered volumetric flask, dilute to volume with spectroscopic *iso*octane, and mix thoroughly.

10.2.3 *Third Dilution*—Dilute 5.00 mL of second dilution to 50 mL in the same manner as in 10.2.2.

10.3 *Determination of Cell Correction*—Measure and record the absorbance of the spectroscopic *iso*octane-filled sample cell as compared to the spectroscopic *iso*octane-filled solvent cell.

10.4 *Measurement of Absorbance*—Transfer portions of the final dilution into the sample cell of the spectrophotometer. Cover the cells immediately to prevent transfer of aromatic hydrocarbons from the sample cell to the solvent cell. Check the windows of the absorption cells and make certain they are clean. Measure the absorbance as recommended in Practices E169. Record the absorbance of the sample as compared to spectroscopic *iso*octane at 285 nm.

TABLE 2 Data Issued by API Research Project 44

Compound	API Serial Number	L/g·cm
Naphthalene	605	28.5
1-methyl Naphthalene	539	32.0
2-methyl Naphthalene	572	22.9
1,2-dimethyl Naphthalene	215	37.3
1,3-dimethyl Naphthalene	216	36.4
1,4-dimethyl Naphthalene	217	43.5
1,5-dimethyl Naphthalene	218	54.0
1,6-dimethyl Naphthalene	219	36.4
1,7-dimethyl Naphthalene	220	36.0
1,8-dimethyl Naphthalene	221	46.0
2,3-dimethyl Naphthalene	222	22.0
2,6-dimethyl Naphthalene	226	21.3
2,7-dimethyl Naphthalene	224	23.5
1-isopropyl Naphthalene	203	31.7

NOTE 5—The dilution of the sample should be controlled so that absorbance readings fall within a range of 0.2 to 0.8 for maximum reproducibility of results. To accomplish this it may be necessary to use an alternative third dilution than the one specified in 10.2.3, such as 10 mL of the second dilution to 25 mL with solvent.

11. Procedure B—Alternative 100-mL Dilution

11.1 *Discussion*—The incorporation of the single dilution procedure has been included as an alternative procedure to reduce: test time, glassware, cleaning, and dilution errors.

11.2 For recommended techniques, refer to Practices E169. Check carefully sections on handling and cleaning of cells and glassware, instrument adjustments, and method of absorbance measurement.

11.3 *Sample Preparation*—Add an appropriate weight of sample to a clean, dry, tared 100-mL volumetric flask. Record the weight to the nearest 0.0001 g. Dilute to the mark with spectroscopic grade *iso*octane, stopper, and mix thoroughly.

11.3.1 Refer to Table 3 for lists of sample weights associated with naphthalene(s) concentrations that give 0.2 to 0.8 absorbance readings as directed in Note 7. A 60-mg sample will be appropriate for typical jet fuels in the range of 0.8 to 3.0 % volume naphthalenes.

NOTE 6—A micropipette is a convenient tool for adding an appropriate volume. If the fuel density is not known at the time of sample preparation, use 0.8 as an approximation.

11.4 *Determination of Cell Correction*—Proceed as written in 10.3.

11.5 *Measurement of Absorbance*—Proceed as written in 10.4.

12. Calculations

12.1 Calculate the mass percentage of naphthalenes in the sample as follows:

$$\text{Naphthalenes, mass \%} = \left[\left(A \times K\right)/\left(33.7 \times W\right)\right] \times 100 \qquad (5)$$

where:

A = corrected absorbance (observed absorbance minus cell correction) of the dilution measured,

For Procedure A in Section 10 using serial dilutions,

TABLE 3 Estimated Sample Weight and Volume to Take for the Volume % Naphthalene Content of the Sample in the Single Dilution Procedure to Keep the Absorption Values Between 0.2 and 0.8 Units (Assuming a Density of 0.8)

Sample Volume (mL)	Sample Weight (mg)	Volume % Naphthalenes for Expected Absorbance of 0.2 units	Volume % Naphthalenes for Expected Absorbance of 0.8 units
0.050	40	1.2	4.8
0.075	60	0.8	3.2
0.100	80	0.6	2.4
0.150	120	0.4	1.6
0.200	160	0.3	1.2
0.300	240	0.2	0.8

K = equivalent volume of solvent, in litres, if the dilution had been made in a single step. For the first dilution K = 0.025, for the second dilution K = 0.25, for the third dilution K = 2.5. For the suggested alternative third dilution K = 0.625,

For Procedure B in Section 11 using 100-mL dilution,

K = 0.10,
W = grams of sample used, and
33.7 = the average absorptivity of C_{10} to C_{13} naphthalenes in litres per gram-centimetre.

12.2 Calculate the volume percentage of naphthalenes as follows:

$$\text{Naphthalenes, volume \%} = M \times (B/C) \tag{6}$$

where:

M = percentage of naphthalenes by mass,
B = relative density of the total fuel (15°C/15°C), and
C = relative density of the naphthalenes (15°C/15°C) = 1.00.

13. Report

13.1 Report numerical values of volume percent naphthalene to the nearest 0.01 %.

14. Reference Spectra

14.1 Absorptivities of individual naphthalene hydrocarbons at 285 nm are derived from data in the API catalog of ultraviolet spectral data issued by Research Project 44 as given in Table 2.

NOTE 7—The arithmetic average of the above absorptivities is 33.7. The reliability of the average absorptivity as a measure of selected individual naphthalenes can be estimated from the above table.

15. Precision and Bias

15.1 *Precision*[3,4]—The precision of this test method was determined by the statistical examination of interlaboratory test results. The precision for Procedure A was determined based on examination of interlaboratory test results for samples covering the range from 0.03 to 4.25 volume % naphthalenes. The precision for Procedure B was determined based on examination of interlaboratory test results for samples covering the range from 0.08 to 5.6 volume % naphthalenes. The precisions are as follows:

15.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method exceed the following values only in one case in twenty.

$$\text{Repeatability for Procedure A} = 0.0222\,(1.00 + X) \tag{7}$$

$$\text{Repeatability for Procedure B} = 0.056\,X^{0.6} \tag{8}$$

where:

X = average of two results, volume %.

15.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method exceed the following values only in one case in twenty.

$$\text{Reproducibility for Procedure A} = 0.0299\,(1.00 + X) \tag{9}$$

$$\text{Reproducibility for Procedure B} = 0.094\,X^{0.6} \tag{10}$$

where:

X = average of two results, volume %.

NOTE 8—Instruments not conforming to the equipment specifications in 7.1 can result in much poorer precision.

15.2 *Bias*—Bias cannot be determined for the procedure in this test method for measuring naphthalene hydrocarbon because the absorptivity will vary with composition of the individual naphthalene derivatives in samples.

16. Keywords

16.1 aviation turbine fuels; naphthalene hydrocarbons; ultraviolet spectrophotometry

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[3] Supporting data for Procedure A (Section 10) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1375.

[4] Supporting data for Procedure B (Section 11) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1525.