Designation: D2269 – 10

Standard Test Method for Evaluation of White Mineral Oils by Ultraviolet Absorption[1]

This standard is issued under the fixed designation D2269; 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.

1. Scope*

1.1 This test method describes a procedure for the examination and evaluation of NF and USP grade white mineral oils.

1.2 This test method is not applicable to oils containing additives soluble in dimethyl sulfoxide (DMSO) that exhibit fluorescence or fluorescence quenching properties.

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

1.4 *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 7.1.1-7.1.3.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1840 Test Method for Naphthalene Hydrocarbons in Aviation Turbine Fuels by Ultraviolet Spectrophotometry

E131 Terminology Relating to Molecular Spectroscopy

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

2.2 *Other Standard:*

U.S. Pharmacopeia USP XIII/National Formulary (NF XVIII)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms and symbols relating to absorption spectroscopy see 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*—the 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 logarithm to the base 10 of the reciprocal of the transmittance, T. In symbols:

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

where *T* is the transmittance as defined in 3.2.5.

3.2.2 *absorptivity, a*, *n*—the absorbance divided by the product of sample pathlength and concentration. In symbols:

$$a = A/bc$$

where A is the absorbance as defined in 3.2.1, b is the sample pathlength as defined in 3.2.4, and c is the concentration as defined in 3.2.3.

3.2.3 *concentration, c*, *n*—the quantity of sample expressed in grams per litre.

3.2.4 *sample pathlength, 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.

3.2.5 *transmittance, T*, *n*—the ratio of the radiant power transmitted by the mineral oil extract in its cell to the radiant power transmitted by the solvent control in its cell. Expressed by:

$$T = P_e/P_c$$

where P_e is the radiant power transmitted by the mineral oil extract and P_c is the radiant power transmitted by the solvent control.

4. Summary of Test Method

4.1 A sample of oil is extracted with dimethyl sulfoxide and the ultraviolet absorbance of the extract is determined in the range from 260 to 350 nm. The absorbance is compared with that of a naphthalene standard.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products 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.

[3] Available from The United States Pharmacopeia (USP), 12601 Twinbrook Parkway, Rockville, MD 20852.

***A Summary of Changes section appears at the end of this standard.**

5. Significance and Use

5.1 The ultraviolet absorption of white mineral oils is used to determine their suitability for use in food, drug, and cosmetic applications.

5.2 The U.S. Pharmacopeia and the National Formulary specifications for mineral oil require the measurement of ultraviolet absorption.

6. Apparatus

6.1 *Spectrophotometer*, equipped to handle liquid samples in 1-cm path length cells. The instrument shall be capable of measuring absorbance with a repeatability of ±1.0 % or better from an average at the 0.4 absorbance level in the spectral region near 290 nm with a nominal band width of 1 nm or less.

NOTE 1—For recommended methods of testing spectrophotometers to be used in this test method refer to Practice E275.

6.2 *Fused Quartz Cells*, two, having path lengths of 1.00 ± 0.005 cm, or better. The distance in centimetres does not include the thickness of the cell in which the sample is contained.

6.3 *Separatory Funnels*, glass-stoppered, of sufficient capacity to perform the necessary extractions in the procedure, equipped with TFE-fluorocarbon stopcocks or other suitable stopcocks that will not contaminate the solvents used.

7. Reagents and Materials

7.1 *Solvents*:

7.1.1 *Normal Hexane*—Pure grade, having an ultraviolet light absorbance not exceeding 0.02 down to 260 nm when measured in a 1-cm cell (**Warning**—Normal hexane is extremely flammable, harmful if inhaled. May produce nerve cell damage.) The purity shall be such that the solvent control as defined in 8.3 shall have an absorbance curve compared to water showing no extraneous impurity peaks and no absorbance exceeding that of dimethyl sulfoxide compared to water at any wavelength in the range 260 to 350 nm, inclusive.

7.1.2 *Spectroscopic Grade 2,2,4–Trimethylpentane (Isooctane)*—(**Warning**—Isooctane is extremely flammable, harmful if inhaled.)

NOTE 2—For a suitable *iso*octane and a procedure for preparing spectroscopic solvents from commercially available stocks, see Test Method D1840.

7.1.3 *Dimethyl Sulfoxide*—(**Warning**—Dimethyl sulfoxide is combustible. Also it is rapidly absorbed through skin.) For use as spectroscopic solvent (see Note 3). Pure grade, clear, water white, 99.9 % dimethyl sulfoxide, m.p. 18.5°C, with an absorbance curve compared to water not exceeding 1.0 at 264 nm and showing no extraneous impurity peaks in the wavelength range up to 350 nm when measured through a path length of 1 cm.

NOTE 3—This solvent can be purified by percolation through a 1.2-m column of type CAL 1.68 by 0.420 mm (12 by 40 mesh) activated charcoal.[4] The column is 25 mm in diameter, drawn to 6.4 mm in diameter at the bottom and has a reservoir at the top for containing the liquid. Glass wool is placed in the bottom of the column, and about 13 mm of 0.707 to 0.074 (25 to 200 mesh) or 0.0149 to 0.074 (100 to 200 mesh) silica gel is placed on top of it. The column is filled with activated charcoal and the spectroscopic solvent poured into the reservoir and allowed to percolate through the charcoal at atmospheric pressure. The purified solvent is collected at the bottom of the column and stored in glass-stoppered bottles as it is very hygroscopic and reacts with some metal containers in the presence of air.

7.2 *Naphthalene*—high-purity, 99+ %.

7.3 *Standard Reference Solution*—A solution containing 7.0 mg of naphthalene per litre of purified *iso*octane.

7.4 *Standard Reference Spectrum*—The absorbance curve obtained by scanning the standard reference solution in the range from 260 to 350 nm against *iso*octane of the same spectral purity as that used to prepare the standard (7.1.2).

7.5 *Standard Reference Absorbance*— The absorbance at 275 nm of the standard reference spectrum.

NOTE 4—This absorbance will be approximately 0.30.

8. Procedure

8.1 Transfer 25 mL of the mineral oil and 25 mL of hexane to a separatory funnel and mix. Add 5.0 mL of dimethyl sulfoxide and shake the mixture vigorously for at least 1 min. Allow to stand until the lower layer is clear.

8.2 Transfer the lower layer to a separatory funnel, add 2 mL of hexane and shake the mixture vigorously. Allow to stand until the lower layer is clear. Draw off the lower layer, designated as *mineral oil extract*, into a 1-cm cell.

8.3 Add 5.0 mL of dimethyl sulfoxide to 25 mL of hexane in a separatory funnel. Shake vigorously for at least 1 min and allow to stand until the lower layer is clear. Draw off this layer, designated as *solvent control*, into a 1-cm cell.

8.4 Determine the absorbance of the mineral oil extract compared to the solvent control through the range 260 to 350 nm inclusive.

9. Correction for Inhibitor Content[5]

9.1 A correction for the absorbance due to inhibitor may be made as described below if a sufficient amount of the same inhibitor contained in the sample and the inhibited sample are available to prepare a blend. The concentration of additional inhibitor to be added to the inhibited sample should be equal to the concentration contained in the inhibited sample.

[4] The sole source of supply of the activated charcoal known to the committee at this time is Calgon Carbon Dr., Robinson Township, PA 15205. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[5] Correction for inhibitor content has not been cooperatively tested.

NOTE 5—The inhibitor content is usually expressed in pounds per thousand barrels. Pound per thousand barrels × 0.00285 = grams per litre. For example, 3 lb/1000 barrels × 0.00285 = 0.00855 g/litre.

9.2 Weigh a minimum of 50 mg of inhibitor in a volumetric flask, fill to volume with inhibited sample and mix thoroughly. Make further dilutions with inhibited sample, as necessary, to obtain the desired concentration of added inhibitor.

9.3 Run the original inhibited sample in accordance with Test Method D2269 and label it Run A.

9.4 Run the blend containing known amount of added inhibitor in accordance with Test Method D2269 and label it Run B.

9.5 Calculate the difference in absorbance between Run B and Run A at corresponding wavelengths as follows:

$$\Delta A = A_b - A_a \quad (1)$$

where:

ΔA = difference in absorbance at a given wavelength,
A_b = absorbance of Run B at the same wavelength, and
A_a = absorbance of Run A at the same wavelength.

9.5.1 Correct the absorbance at each wavelength as follows:

$$A_c = A_a - \Delta A \quad (2)$$

where:

A_c = corrected absorbance at each wavelength.

9.6 Mark the points of inflection on the spectrum from Run A at wavelengths lower and higher than those which were increased.

9.6.1 Draw a baseline tangent to the curve connecting the two points.

9.6.2 Read the absorbance, A_d, along the baseline at each wavelength.

9.7 Compare A_d and A_c at each position. If A_c is less than A_d it is indicated that the inhibitor content of the original sample is less than the amount added.

9.8 Use the absorbance A_c or A_d, whichever is greater, to compare to the solvent control through the range 260 to 350 nm, inclusive.

10. Report

10.1 Report the difference between the absorbance of the mineral oil extract and the solvent control at the wavelength desired.

NOTE 6—Calculations can be found in U.S. Pharmacopeia (USP XXIII)/National Formulary (NF XVIII).

11. Precision and Bias

11.1 The precision of this test method as obtained by statistical examination of interlaboratory test results is as follows:

11.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed 0.014 absorbance units only in one case in twenty.

11.1.2 *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 0.044 absorbance units only in one case in twenty.

11.1.3 These precision data were obtained on absorbances in the wavelength region 275 to 280 nm.

11.2 *Bias*—This procedure has no bias because the value of absorbance can be defined only in terms of a test method.

12. Keywords

12.1 ultraviolet spectroscopy; white mineral oil

SUMMARY OF CHANGES

Subcommittee D02.04 has identified the location of selected changes to this standard since the last issue (D2269–99(2005)) that may impact the use of this standard.

(*1*) Revised 6.3.