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An American National Standard

Standard Test Methods for Hydrocarbon Waxes Used for Electrical Insulation[1]

This standard is issued under the fixed designation D 1168; 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 These test methods cover a compendium of tests that apply to mineral waxes of petroleum origin in general, but more specifically to the so-called microcrystalline types used as either electrical insulation or moisture-proofing mediums, or both, for treating, impregnating, coating, and filling electrical apparatus. These test methods are also applicable to other waxes of natural or synthetic origin, provided that their characteristics are similar to those of the so-called microcrystalline waxes.

NOTE 1—There is no equivalent ISO or IEC standard.

1.2 The values stated in SI units are the 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 precautions, see Section 5.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 6 Test Method for Loss on Heating of Oil and Asphaltic Compounds

D 70 Test Method for Specific Gravity and Density of Semi-Solid Bituminous Materials (Pycnometer Method)

D 87 Test Method for Melting Point of Petroleum Wax (Cooling Curve)

D 88 Test Method for Saybolt Viscosity

D 92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester

D 94 Test Method for Saponification Number of Petroleum Products

D 127 Test Method for Drop Melting Point of Petroleum Wax, Including Petrolatum

D 176 Test Methods for Solid Filling and Treating Compounds Used for Electrical Insulation

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)

D 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration

D 937 Test Method for Cone Penetration of Petrolatum

D 938 Test Method for Congealing Point of Petroleum Waxes, Including Petrolatum

D 974 Test Method for Acid and Base Number by Color-Indicator Titration

D 1321 Test Method for Needle Penetration of Petroleum Waxes

D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)

D 1711 Terminology Relating to Electrical Insulation

D 2161 Practice for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity

E 28 Test Method for Softening Point of Resins Derived from Naval Stores by Ring-and-Ball Apparatus

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology D 1711.

4. Significance and Use

4.1 The significance and use of the individual test methods are to be found in the individual methods referenced. For significance specifically applicable to electrical insulation materials, refer to Test Methods D 176.

5. Safety Precautions

5.1 Ovens in which waxes are heated should have low-temperature heating elements, forced exhaust, and safety door latches to minimize the hazard of explosion of vapors.

6. Test Methods

6.1 Use the following methods for testing hydrocarbon waxes as specified for the individual material:

6.1.1 *Color*—Test Method D 1500.

[1] These methods are under the jurisdiction of ASTM Committee D09 on Electrical and Electronic Insulating Materials and are the direct responsibility of Subcommittee D09.01 on Electrical Insulating Varnishes, Powders, and Encapsulating Compounds.

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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.

6.1.2 *Melting and Softening Properties*:

6.1.2.1 Determine melting point by Test Method D 127.

6.1.2.2 Determine softening point by the ring-and-ball Test Method E 28. Make three measurements. If any measurement differs from the average by more than 1°C, the significance of the test is doubtful.

6.1.2.3 Use Test Method D 87 for melting point of waxes having a plateau in their cooling curve.

6.1.2.4 Determine the congealing point by Test Method D 938. (The test value will usually be lower than the melting point determined by Test Method D 127.)

6.1.3 *Penetration*:

6.1.3.1 Use Test Method D 1321 for all but very soft waxes.

6.1.3.2 Use Test Method D 937 for soft waxes below the range for Test Method D 1321.

6.1.3.3 Report the test method used.

6.1.4 *Viscosity*:

6.1.4.1 Unless otherwise specified, measure Saybolt Universal Viscosity at 99°C (210°F) using Test Method D 88.

6.1.4.2 When specified, Test Method D 445 or measurements at other temperatures may be made.

6.1.4.3 Use Practice D 2161 for conversion of viscosity values.

6.1.5 *Flash and Fire Points*—Test Method D 92.

6.1.6 *Loss on Heating*:

6.1.6.1 Determine by Test Method D 6.

6.1.6.2 For some waxes subject to oxidation on heating and due to lack of close control of air circulation, the reproducibility of results may be variable.

6.1.7 *Saponification Number*:

6.1.7.1 Determine using Test Method D 94, modified as specified in 6.1.7.2-6.1.7.4.

6.1.7.2 Use solvent mixtures appropriate to the melting point of the wax being tested as follows:

74.8°C	ethanol-methyl ethyl ketone
77.3°C	isopropanol-methyl ethyl ketone
80.6°C	isopropanol-toluol
92.6°C	N-propanol-toluol

6.1.7.3 Do not use the ASTM precipitation naphtha.

6.1.7.4 Reheat the solution when necessary during titration.

6.1.8 *Acid and Base Number*:

6.1.8.1 Determine using Test Method D 664.

6.1.8.2 When the color of the material permits, and when specified for the material, Test Method D 974 may be used.

6.1.8.3 The test results on a given sample may vary depending upon the method used.

6.1.9 *Electrical Properties*—Determine using methods specified in Test Methods D 176.

6.1.10 *Density and Volume Changes*:

6.1.10.1 Measure specific gravity at 25°C (77°F) by Test Method D 70 using the procedure for cements and pitches.

6.1.10.2 Measure volume contraction on cooling from liquid to solid using the procedures in Sections 7-10 of these test methods.

6.1.10.3 Measure density at specified temperatures (either below or above the melting point) using procedures in Sections 11-18 of these test methods. From the density measurement, specific gravity and specific volume may be calculated. When specified for a given material, density measurements can be made at several temperatures from which volume coefficient of expansion can be calculated.

VOLUME CONTRACTION ON COOLING

7. Scope

7.1 This test method measures the volume contraction of microcrystalline wax to be used for electrical insulation when cooled from 5.5°C (10°F) above its melting point to 27.8°C (50°F) below its melting point.

7.2 The total contraction from a temperature of 5.5°C (10°F) above the melting point to a temperature of 27.8°C (50°F) below the melting point has been used in defining a crystallinity index, which may be employed to classify micro- and macrocrystalline waxes.[3]

7.3 This test method can also be used to determine the contraction occurring between temperatures other than those specified in this section, but in this case the temperature limits should be stated.

8. Apparatus

8.1 *Mixing Cylinder*, 100-mL capacity.

8.2 *Water Bath*, capable of maintaining the test temperature within ±0.5°C (±1°F), and permitting submersion of at least the graduated portion of a 100-mL mixing cylinder.

9. Procedure

9.1 Heat the sample to 5.5°C (10°F) above its melting point (Test Method D 127) and pour exactly 100 mL of the heated sample into a 100-mL mixing cylinder that has been brought to the same temperature. Allow the wax to cool for 2 h, protected from drafts.

9.2 If a thin wax layer covers the cavity formed on cooling, pierce the wax layer at the center with a pointed glass rod to make an opening 2 to 3 mm in diameter.

9.3 Immerse the mixing cylinder in a water bath maintained at 27.8°C (50°F) below the melting point of the wax for 2 h. Remove the cylinder from the bath and add a 50 % aqueous solution of glycerin from a buret to reach the 100-mL mark. Apply a slight vacuum to the cylinder to remove any trapped air, and add more glycerin solution if necessary. Note the total millilitres of glycerin solution added, and report as "percentage contraction." The percentage expansion is 100 times the reciprocal of (100 − percentage contraction) multiplied by the percentage contraction.

10. Precision and Bias

10.1 Operators familiar with this method estimate that duplicate determinations by the same operator should differ by no more than 2 % of the value, and by different operators using different apparatus, by no more than 5 %. This precision applies to the usual waxes and over the range from 5.5°C (10°F) above to 27.8°C (50°F) below the melting point.

10.2 A statement of bias is not applicable since a standard reference material for this property is unavailable.

[3] See Kinsel, A., and Phillips, J., "Method for Classification of Petroleum Waxes," *Industrial and Engineering Chemistry*, IECHA, Vol 17, March 15, 1945, p. 152.