



Standard Test Methods for Hydrocarbon Waxes Used for Electrical Insulation¹

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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 $\pm 0.5^\circ\text{C}$ ($\pm 1^\circ\text{F}$), 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.

DENSITY

11. Scope

11.1 This test method determines density of wax and like material, in both the solid and liquid phases. The results are obtained by determining the mass and volume of a liquid of known density, and the wax in a dilatometer, over a temperature range from 15.5 to 99°C (60 to 210°F). From these data the density, specific volume, specific gravity, and cubical expansion of the wax can be calculated.

12. Apparatus and Materials

12.1 *Dilatometer*, constructed of heat-resistant glass as shown in Fig. 1.

12.2 *Glass-Walled Constant-Temperature Water or Oil Bath*, of such depth as to permit observation of the submerged active length of the capillary arm of the dilatometer.

12.3 *Analytical Balance*.

12.4 *Wooden Frames and Cellulose Film or Shallow Metal Container*, for casting wax specimens.

12.5 *Glycerin*, cp.

13. Standardization of Dilatometer

13.1 Standardize the dilatometer as follows so that its exact volumetric capacity at each of the calibration points will be known:

13.1.1 Clean the dilatometer with chromic acid, and rinse a number of times with distilled water. Dry, either by heating in an oven at 121°C (250°F) for 30 min or by rinsing with acetone. If acetone is used, remove all traces of vapor by suction.

13.1.2 Assemble the dilatometer, using a minimum amount of stopcock lubricant.

13.1.3 Determine the mass of the instrument on a balance, using a suitable hanger for suspending the dilatometer from the pan stirrup. Note the position of the dilatometer, and replace it in a like position for each successive weighing.

13.1.4 Deaerate distilled water by boiling vigorously. Cool slowly, without stirring, to approximately 15°C (60°F).

13.1.5 Charge the dilatometer with the prepared distilled water. Draw the water up into the capillary to approximately the 1.8-mL point by applying gentle suction. Replace the plug, with stopcock open, and permit the excess water to drain out. Close the stopcock and remove all traces of water from the stopcock outlet tube and from the hole through the body and plug of the stopcock. Dry the outside of the dilatometer and determine the mass of the water.

13.1.6 Place in a constant-temperature bath at $20 \pm 0.05^\circ\text{C}$ ($68 \pm 0.1^\circ\text{F}$) and, when temperature equilibrium is attained as indicated by the constancy of volume readings at three successive 5-min intervals, record the volume reading.

13.1.7 Remove the dilatometer from the bath and change the mass of water by opening the stopcock and drawing off a portion, lowering the volume in the capillary to about the 0.2 point. Remove all moisture, and weigh as described in 13.1.5.

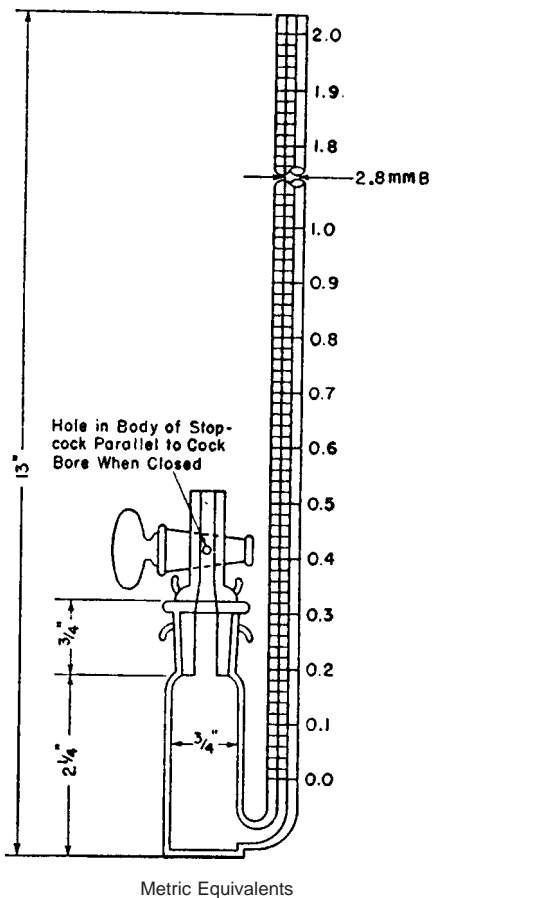
13.1.8 Repeat the procedure described in 13.1.6.

13.1.9 Remove the dilatometer from the bath and repeat the procedure described in 13.1.1, 13.1.2, 13.1.3, and 13.1.5, and charging the dilatometer so that the capillary arm will be filled to the 1.3-mL point, and repeating 13.1.6 and 13.1.7. In repeating the procedure in 13.1.7, lower the capillary volume to the 0.6 point.

13.1.10 From the volume readings, mass of water, and the standard tabulated value of the density of water, calculate the volume at each of the determined points. The four volume readings, when plotted, shall fall on a straight line. From this graph determine the volumetric capacity of the dilatometer at each test temperature.

14. Preparation of Specimens

14.1 Heat the sample to approximately 17°C (30°F) above its melting point and pour into a shallow container to a liquid depth of approximately 3 mm (1/8 in.). A suitable container for use in the preparation of test specimens consists of a membrane of regenerated cellulose film stretched across a rectangular opening between a four-sided wooden frame, approximately 3 by 5 in. (76 by 127 mm), made from 12 by 31-mm (1/2 by 1 1/4-in.) maple wood. The film may be fastened to the wooden frame by pressure-sensitive tape or thumb tacks. After mounting the film, place a second wooden frame, identical in size



Metric Equivalents	
in.	mm
13	330
2 1/4	57
3/4	19

FIG. 1 Dilatometer

with the first, on top of the film and line up the two frames. Give the lower inner faces of the upper frame a thin coating of clear amber petrolatum to facilitate removal of the cake of solidified wax. Cover the sample with a sheet of metal or cardboard to reduce heat losses, and permit it to cool until it solidifies. When the wax has cooled to room temperature, remove it from the container by rapidly chilling it in an ice-water bath. This will cause the sample to contract and permit its easy removal in thin cake form. Strip the film from the wax sample. Select a section of the cake that is free from all visible entrained air bubbles and cut in 50 by 9.5-mm (2 by 3/8-in.) pieces. Smooth cut sides to eliminate all voids either by heating the cut sides or by mechanical means.

15. Determination of Density of Glycerin

15.1 To ensure the effective cancellation of buoyancy, meniscus, and other minor errors, determine the density of the glycerin over the entire temperature range as follows:

15.1.1 Repeat the procedure described in 13.1.1, 13.1.2, and 13.1.3.

15.1.2 Carefully charge the dilatometer with the glycerin to avoid entrainment of air bubbles. When properly charged, the meniscus of the glycerin in the capillary arm will be slightly above the first calibration mark at the bottom of the capillary. Carefully remove all excess glycerin from the exterior and from the stopcock as described in 13.1.5.

15.1.3 Determine the mass of the glycerin, taking cognizance of the position of the dilatometer as pointed in 13.1.3.

15.1.4 Place the charged dilatometer in a constant-temperature bath operating at 15.6°C (60°F), and proceed otherwise as in 13.1.6.

15.1.5 When the foregoing data are obtained, raise the bath temperature in 11°C (20°F) increments, and repeat the steps prescribed in 15.1.3 and 15.1.4.

15.1.6 From the mass and volume data, calculate the density of the glycerin at the various temperatures.

NOTE 2—When the glycerin is standardized, it can be used over a period of several months if it is properly stored and care is taken against contamination. Glycerin is hygroscopic.

16. Procedure

16.1 Repeat the procedure described in 13.1.1, 13.1.2, and 13.1.3.

16.2 Charge the dilatometer with four or five pieces of the prepared sample of wax, and determine the mass of the sample.

16.3 Charge the dilatometer with the standardized glycerin, being careful to avoid or eliminate all entrained air bubbles on or between the pieces of sample and on the glass wall. Replace the plug, with stopcock open, and permit the excess glycerin to drain out. Close the stopcock, and clean and dry the body and plug. Hold the plug firmly in place by the use of helical steel springs or other suitable media bound around the lugs provided on the body and plug. Observe similar care in keeping the stopcock tight, so as to avoid any loss of glycerin, particularly at the upper temperatures.

16.4 Determine the mass of the glycerin.

16.5 Place the dilatometer in a constant-temperature bath at the initial test temperature. When temperature equilibrium is attained, as indicated by the constancy of volume readings at

three successive 5-min intervals, record the volume reading. Raise the bath to the next test temperature, and read the volume at temperature equilibrium as before. When a curve of specific volume against temperature is required, read the temperature and volume at 5.5°C (10°F) intervals or at such other points as will furnish a smooth curve of specific volume against temperature.

17. Calculation

17.1 Calculate the density of the material as follows:

$$D_1 = M_1/V_1 \quad (1)$$

where:

D_1 = density of sample at test temperature,

M_1 = mass of sample, and

V_1 = volume of sample at test temperature.

$$V_1 = V_2 - V_3 \quad (2)$$

where:

V_2 = volumetric capacity of dilatometer at test reading and temperature, and

V_3 = volume of glycerin at test temperature.

$$V_3 = M_3/D_3 \quad (3)$$

where:

M_3 = mass of glycerin, and

D_3 = density of glycerin at test temperature.

17.2 Calculate the specific gravity of the material as follows:

$$\text{Specific gravity, } t/t = D_1/D_2 \quad (4)$$

where:

D_1 = density of sample at the desired temperature, and

D_2 = density of water at the identical temperature of D_1 .

17.3 Calculate specific volume as the reciprocal of the density at the test temperature. Report the specific volume at a number of test temperatures, in either tabular or graphical form.

17.4 Calculate the cubical expansion or the coefficient of thermal expansion, as desired, from the specific volumes at the test temperatures. The coefficient for the solid state should be calculated from the lowest test temperature to a point 10°C (18°F) below the melting point, and the coefficient for the liquid state should be calculated from a temperature 5°C (9°F) above the melting point to 99°C (210°F), or the highest test temperature.

18. Precision and Bias

18.1 Operators familiar with this method estimate that the density, specific gravity, or specific volume may be calculated with a precision well within 1%. The precision of the coefficient of expansion is about 2%.

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

19. Keywords

19.1 acid and base number; color; density; fire point; flash point; hydrocarbon waxes; loss on heating; melting properties; mineral waxes; penetration; saponification number; softening

properties; viscosity; volume changes; volume contraction; waxes

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