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Standard Test Method for Apparent Viscosity of Petroleum Waxes Compounded with Additives (Hot Melts)[1]

This standard is issued under the fixed designation D2669; 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.

ε[1] NOTE—Added mercury caveat editorially in April 2012.

1. Scope

1.1 This test method covers the determination of the apparent viscosity of petroleum waxes compounded with additives (hot melts). It applies to fluid hot melts having apparent viscosities up to about 20 Pa·s at temperatures up to 175°C (347°F).

NOTE 1—For petroleum waxes and their blends having low apparent viscosities, below about 15 mPa·s, Test Method D445, is especially applicable.

1.2 The values stated in SI units shall be regarded as the standard. One pascal second (Pa·s) = 1000 centipoises (cP). One millipascal second (mPa·s) = 1 centipoise (cgs units).

1.3 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

E1 Specification for ASTM Liquid-in-Glass Thermometers

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *viscosity*—the ratio of shear stress to shear rate. Viscosity of a liquid is a measure of the internal friction of the liquid in motion. The unit of dynamic viscosity is the pascal second. For a Newtonian liquid, the viscosity is constant at all shear rates. For a non-Newtonian liquid, viscosity will vary depending on shear rate.

3.1.2 *viscosity, apparent*—the viscosity determined by this method, expressed in pascal seconds. Its value may vary with the spindle and rotational speed selected because many hot melts are non-Newtonian.

4. Summary of Test Method

4.1 Approximately 800 g of sample are melted on a hot plate or in an oven. An 800-mL glass beaker which is jacketed with an electric heating mantle is filled with the melted sample to a level of about 25 mm (1 in.) from its top. The viscometer, with attached spindle and guard, is properly positioned. Stirring is begun and continued while the temperature of the

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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] The last approved version of this historical standard is referenced on www.astm.org.

sample is brought to slightly above the highest desired test temperature. Heating is discontinued and stirring is maintained until the sample cools to the chosen temperature. At this time, stirring is stopped and the apparent viscosity is determined. Additional determinations are made over a range of temperatures as the sample cools. Results of temperature and apparent viscosity determinations are plotted on semilog paper, and values at any particular temperature are determined from the curve.

5. Significance and Use

5.1 This test distinguishes between hot melts having different apparent viscosities. It is believed that apparent viscosity determined by this procedure is related to flow performance in application machinery operating under conditions of low shear rate. Apparent viscosity as determined by this method may not correlate well with end use applications where high shear rates are encountered.

5.2 Materials of the type described in this procedure may be quite non-Newtonian and as such the apparent viscosity will be a function of shear rate under the conditions of test. Although the viscometer described in this test generally operates under conditions of relatively low shear rate, differences in shear effect can exist depending upon the spindle and rotational speed conditions selected for the test program. Maximum correlation between laboratories, therefore, depends upon testing under conditions of equivalent shear.

6. Apparatus

6.1 *Viscometer*—Brookfield Synchro-Lectric Viscometer, Models LVF or LVT with numbers 1, 2, 3, and 4 stainless steel spindles and V-shaped stand with leveling screws.

6.2 *Glass Beaker,* 800-mL Griffin standard form.

6.3 *Glass Heating Mantle,* for 800-mL beaker.

6.4 *Autotransformer,* variable.[4]

6.5 *Thermometers,* ASTM Precision Thermometer 88C having a range from 10 to 200°C or Thermometer 88F having a range from 50 to 392°F as prescribed in Specification E1 are suitable.

6.6 *Laboratory Stirrer Motor,* variable speed.

6.7 *Propeller and Shaft,* stainless steel 51 mm (2 in.) in diameter, three blades to fit 7.9 by 475-mm (5⁄16 by 18-in.) stainless steel shaft.

6.8 *Hot Plate,* with continuously adjustable temperature control.

6.9 *Laboratory Jack,* scissors-type.

6.10 *Ring Stands and Clamps,* for mounting stirrer and thermometer.

6.11 *Semilog Graph Paper,* two cycles.

[4] The sole source of supply of the apparatus known to the committee at this time is the Variac Type W5 MT, IET Labs, Inc. 534 Main Street, Westbury, NY 11590. 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.

7. Procedure

7.1 *Selection of Spindle*—From the estimated viscosity of the sample and Table A1.1, select a spindle size and speed combination that preferably will produce readings within the range recommended by the manufacturer. Attach the spindle to the viscometer, with guard attached and mount the instrument on its stand.

NOTE 2—Care must be taken while storing and handling the spindle. It should be protected from dust, corrosive deposits, and mechanical abuse. Avoid touching the calibrated section of the spindle with the hands. Thoroughly clean it and the guard after each use.

7.2 *Preparation of Sample*—In a suitable container, melt approximately 800 g representative of the sample to be tested on a hot plate or in an oven. Bring the temperature of the sample to 120 to 150°C (250 to 300°F) and stir to ensure homogeneity, taking care not to whip air into the melted sample. Fill the 800-ml test beaker with the melted sample to a level about 25 mm (1 in.) from the top. Place the filled beaker into the heating mantle which is supported by the laboratory jack in its lowered position. Connect the heating mantle to the autotransformer which, in turn, is connected to the proper ac supply. Connect the viscometer to its proper ac supply. Position the viscometer with spindle and guard attached, the stirrer, and thermometer as shown in Figs. A1.1 and A1.2. Mount the thermometer so that the center of its bulb is in the same horizontal plane as the center of the test section of the spindle, and spaced approximately the same distance as the guard from the spindle, about 13 mm (½ in.). Position the stirring propeller about midway between the bottom of the guard and the bottom of the beaker. Position the complete assembly so that the test portion of the spindle is spaced approximately 19 mm (¾ in.) from the side of the beaker when the beaker containing the sample is in the operating position. Raise the beaker containing the sample by means of the laboratory jack so that the spindle is covered to about 6 mm (¼ in.) below its immersion mark. Adjust the stirrer speed to give maximum agitation of the test sample without permitting vortex or air bubble inclusion. Apply heat to the sample by adjusting the autotransformer, and raise its temperature to about 5°C (10°F) above the highest test temperature. Maintain stirring throughout the heating cycle, being careful to prevent air entrainment in the sample.

NOTE 3—As the temperature of the sample increases, its liquid level will approach the immersion mark on the spindle. Be careful to prevent the sample level from rising above the immersion mark on the spindle. Final immersion adjustment shall be made just before viscometer readings are determined.

7.3 *Viscosity Determination*—When the temperature of the sample reaches 5°C (10°F) above the highest test temperature, shut off the autotransformer, start the viscometer motor, and continue stirring. The temperature of the sample will begin to lower, and when it becomes 0.5°C (1°F) above the intended test temperature, stop the stirrer, but continue the spindle rotation. Wait 5 s, and readjust the viscometer to the immersion mark on the spindle. Allow the spindle to make three complete additional rotations. Engage the pointer clutch, and stop the viscometer motor when the pointer is in view. Record the dial reading. Restart the viscometer motor, release the clutch, make three additional spindle revolutions, engage the clutch, and

stop the viscometer motor when the pointer is in view. Record the second dial reading, and repeat the above operation to obtain a total of three readings which should be completed within a period of about 1 min. During this time the temperature of the sample should fall no lower than 0.5°C (1°F) below the intended test temperature. Record the three test readings and the test temperature. Immediately after the final reading, start the stirrer motor and viscometer with the clutch engaged. Permit the temperature of the sample to drop about 15°C (25°F), and repeat the same procedure at a lower temperature. Continue this sequence to produce dial readings at four or more different temperatures, each spaced approximately 15°C (25°F) lower than the preceding test temperature.

NOTE 4—The range of test temperatures shall include all temperatures at which apparent viscosity values are desired. Minor vertical adjustments of the spindle may be required to maintain its proper immersion as the volume of the sample decreases with lower temperatures.

8. Calculation

8.1 Determine the averages of the three scale readings made for each test temperature. Calculate the apparent viscosities, in pascal seconds or millipascal seconds, by multiplying the average viscometer scale reading for each test temperature by the factor indicated in Table A1.1 for the spindle and speed combination used.

8.2 Plot the apparent viscosity values obtained on the log scale, and the corresponding test temperatures on the linear scale of appropriate semilog paper. From the plot, determine the apparent viscosity of the sample at any temperature within the range of the test temperatures.

9. Report

9.1 Report the apparent viscosity at a given temperature with the spindle number and speed used to obtain the data as:

Apparent viscosity, 325 mPa·s at 120°C (1)

(No. 2 spindle, 30 rpm)

10. Precision and Bias

10.1 The composition of a hot melt influences the precision to be expected when testing different types of samples. The following data should be used to judge the acceptability of results (95 % probability) for four different types of hot melts according to the concept of precision as given in Practice E180.

10.2 Duplicate results should be considered suspect if they differ by more than the following amounts for each of the four types listed: below:

10.2.1 *High-Viscosity Sample, M1-65-20:*

58 weight % of a 68°C (155°F) melting point wax.

42 weight % of an ethylene-vinyl acetate copolymer containing 27 to 29 % vinyl acetate and having a melt index of from 12 to 18.

Viscosity, mPa·s	Temperature, °C	(°F)	Repeatability, mPa·s	Reproducibility, mPa·s
11 200	121	(250)	1900	2400
7500	134	(275)	1200	1700
5100	149	(300)	660	1500

10.2.2 *Medium-Viscosity Sample, MI-65-21:*

72 weight % of a 61°C (142°F) melting point wax.

28 weight % of the same copolymer used in sample MI-65-20.

Viscosity, mPa·s	Temperature, °C	(°F)	Repeatability, mPa·s	Reproducibility, mPa·s
1200	121	(250)	81	240
840	134	(275)	63	150
610	149	(300)	47	120

10.2.3 *Low-Viscosity Sample, MI-65-22:*

96.3 weight % of a 77°C (170°F) melting point microcrystalline wax.

2.7 weight % of butyl rubber.

Viscosity, mPa·s	Temperature, °C	(°F)	Repeatability, mPa·s	Reproducibility, mPa·s
68	121	(250)	15	32
52	134	(275)	11	29
41	149	(300)	7.9	22

10.2.4 *Low-Viscosity Sample, MI-65-23:*

80 weight % of a 68°C (154°F) melting point wax.

20 weight % of a 5000 molecular weight polyethylene having a melting point from 107 to 111°C (224 to 232°F), a specific gravity of 0.92 and a typical viscosity at 140°C of 4 Pa·s.

Viscosity, mPa·s	Temperature, °C	(°F)	Repeatability, mPa·s	Reproducibility, mPa·s
25	121	(250)	1.9	4.4
20	134	(275)	1.2	4.0
16	149	(300)	1.5	3.8

10.3 *Bias*—The procedure in this test method has no bias because the value of apparent viscosity can be defined only in terms of a test method.

11. Keywords

11.1 apparent viscosity; hot melts; petroleum waxes; waxes