Designation: D2669 – 16

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.

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.

1.2.1 *Exception*—Alternative units in parentheses are for information purposes only.

NOTE 2—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)

3. Terminology

3.1 *Definitions:*

3.1.1 *viscosity, n*—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, n*—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 container is filled with the melted sample to a level of about 25 mm (1 in.) from its top and placed in a temperature bath. The viscometer, with attached spindle and guard, is positioned in the test specimen. Stirring is begun and continued while the temperature of the 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

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

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, Rotational*—The essential instrumentation required providing the minimum rotational viscometer analytical capability includes:

6.1.1 A *drive motor*, to supply a unidirectional displacement to the specimen at a rate between 0.5 r⁄min and 60 r⁄min constant to ±1 %.

6.1.2 A force *sensor* to measure the torque developed by the specimen to within 1 %.

6.1.3 A *coupling shaft*, or other means to transmit the rotation displacement from the motor to the specimen.

NOTE 3—It is helpful to have a mark on the shaft to indicate appropriate test fluid level.

6.1.4 A *rotational element, spindle or tool*, composed of stainless steel or other insulating material, to fix the specimen between the draft shaft and a stationary position of the type shown in Fig. 1.

NOTE 4—Each spindle typically covers a range of about 2 decades of viscosity. The spindle is selected so that the measured viscosity is between 10 % and 90 % of the range of that spindle.

6.1.5 A *specimen container* to contain 800 mL of the test specimen during testing.

NOTE 5—A low form, glass Griffin beaker has been found suitable for this purpose.

6.1.6 A *data collection device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for rotational viscosity are torque, rotational speed, temperature and time.

NOTE 6—Manual observation and recoding of data are acceptable

6.1.7 A *stand* to support, level and adjust the height of the drive motor, shaft and spindle.

6.1.8 Auxiliary instrumentation considered useful in conducting this method includes:

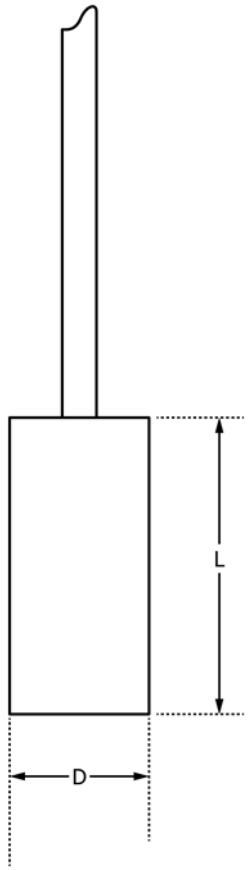

FIG. 1 Spindle Configuration

6.1.8.1 A *level* to indicate the vertical plumb of the drive motor, shaft and spindle.

6.1.8.2 A *guard* to protect the rotational element from mechanical damage.

6.2 A *temperature bath and controller* to provide a controlled isothermal temperature environment for the specimen over the temperature range of 100 °C to 175 °C constant to within ±1 °C.

NOTE 7—A glass heating mantle of suitable size for the container and an autotransformer have been found suitable for this purpose.

6.3 A *temperature sensor or temperature measuring device* to provide an indication of the specimen temperature over the range of 100 °C to 200 °C to within ±0.1 °C.

6.4 *Laboratory Stirrer Motor,* variable speed.

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

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

6.7 *Laboratory Jack,* scissors-type.

6.8 *Ring Stands and Clamps,* for mounting stirrer and temperature measuring device.

6.9 *Semilog Graph Paper,* two cycles.

7. Procedure

7.1 *Selection of Spindle*—From the estimated viscosity of the sample and Table 1, select a spindle size and speed combination that will produce readings within the range of 10 % to 90 % full scale. Attach the spindle to the viscometer, with guard attached and mount the instrument on its stand.

NOTE 8—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 °C to 150 °C (250 °F to 300 °F) and stir to ensure homogeneity, taking care not to whip air into the melted sample.

7.2.1 Fill the 800 mL container with the melted sample to a level about 25 mm (1 in.) from the top. Place the filled container into the temperature bath so that it is supported in its position. Position the viscometer with spindle and guard attached, the stirrer, and temperature sensor as shown in Fig. 2 and Fig. 3. Mount the temperature sensor so that it 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 container. Position the viscometer assembly so that the test portion of the spindle is spaced approximately 19 mm (¾ in.) from the side of the container when in the operating position. Raise the container with the sample 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 temperature bath and raise its temperature to about 5 °C (10 °F) above the highest operator selected test temperature. Maintain stirring throughout the heating cycle, being careful to prevent air entrainment in the sample.

NOTE 9—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:*

7.3.1 When the temperature of the sample reaches 5 °C (10 °F) above the highest test temperature, shut off the temperature bath, start the viscometer motor, and continue stirring.

7.3.2 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.

7.3.3 Allow the spindle to make three complete additional rotations. Record the torque reading.

7.3.4 Make three additional spindle revolutions. Record the second torque reading.

7.3.5 Repeat 7.3.4 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.

7.3.6 Immediately after the final reading, start the stirrer motor and maintain the viscometer rotation.

7.3.7 Permit the temperature of the sample to drop about 15 °C (25 °F), and repeat 7.3.3 to 7.3.6 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 10—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.

8.2 Plot the apparent viscosity values obtained on the logarithmic 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 identification and speed used to obtain the data as:

Apparent viscosity is 325 mPa·s at 120 °C using a No. 2 spindle and 30 r/min.

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.

TABLE 1 Viscometer Spindle Dimensions, Speed, Viscosity Relationship

Spindle No.	Nominal Diameter,[A] mm	Nominal Length,[B] mm	Maximum Viscosity, mPa·s			
			at 60 r/min	at 30 r/min	at 12 r/min	at 6 r/min
1	19	65	100	200	500	1000
2	10	54	500	1000	2500	5000
3	5.9	43	200	4000	10 000	20 000
4	3.2	31	10 000	20 000	50 000	100 000

[A] D in Fig. 1.
[B] L in Fig. 1.

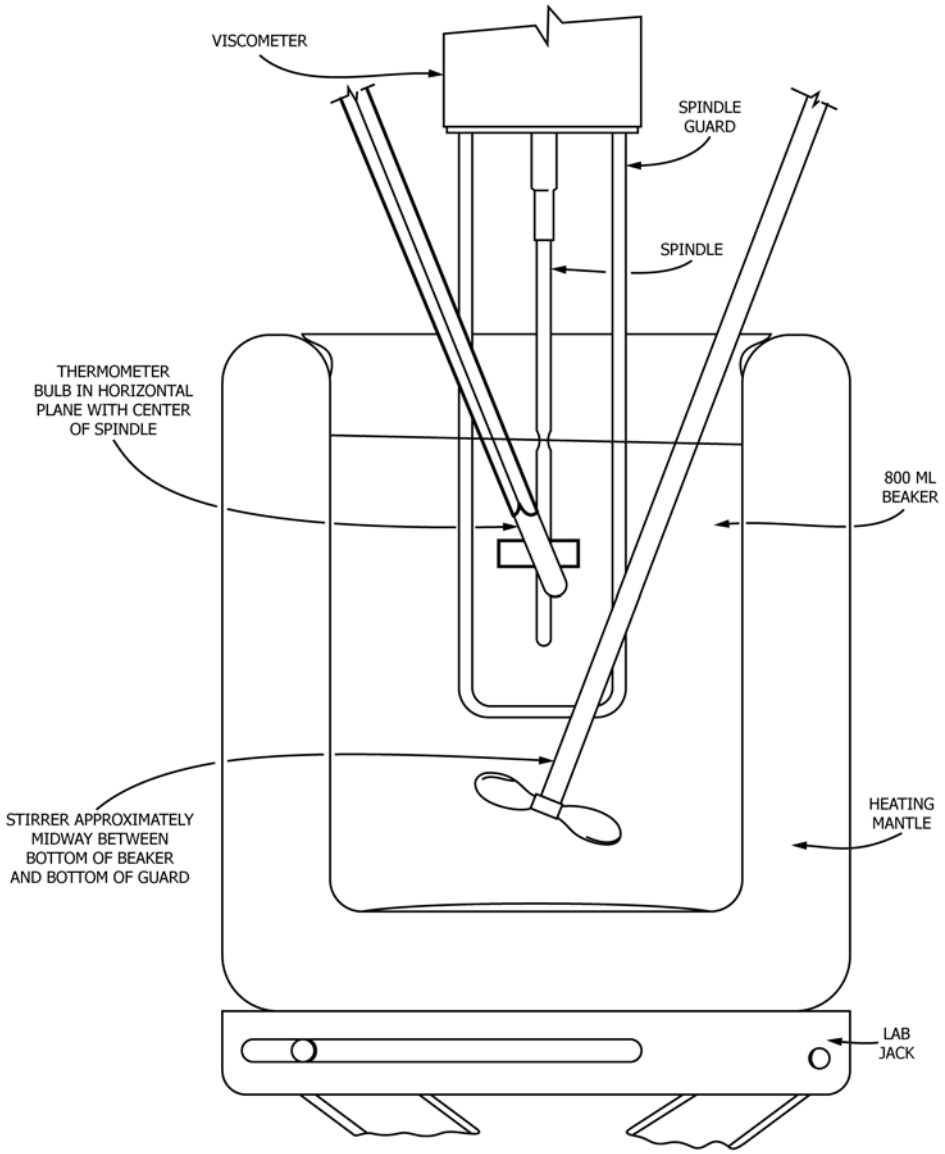

FIG. 2 Viscosity Test Apparatus, Side View

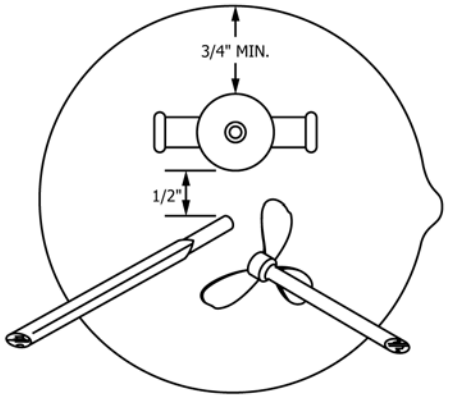

FIG. 3 Viscosity Test Apparatus, Top View

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 % by mass of a 68 °C (155 °F) melting point wax.

42 % by mass 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 % by mass of a 61 °C (142 °F) melting point wax.

28 % by mass 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 % by mass of a 77 °C (170 °F) melting point microcrystalline wax.

2.7 % by mass 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 % by mass of a 68 °C (154 °F) melting point wax.

20 % by mass of a 5000 g/mol molecular weight polyethylene having a melting point from 107 °C to 111 °C (224 °F 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

SUMMARY OF CHANGES

Subcommittee D02.10 has identified the location of selected changes to this standard since the last issue (D2669 – 06 (2012)$^{\varepsilon 1}$) that may impact the use of this standard. (Approved June 1, 2016.)

(1) Revised 4.1, 6.1 and subsections, Section 7, and 9.1.
(2) Table 1 moved from Annex to subsection 7.1 and revised.
(3) Added new Fig. 1; moved Fig. 2 and Fig. 3 from Annex to subsection 7.2.1.
(4) Deleted Annex.
(5) Revised SI unit formatting throughout.