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Designation: ~~D3236 – 88 (Reapproved 2009)~~ D3236 – 88 (Reapproved 2014)

Standard Test Method for Apparent Viscosity of Hot Melt Adhesives and Coating Materials[1]

This standard is issued under the fixed designation D3236; 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 hot melt adhesives and coating materials compounded with additives and having apparent viscosities up to 200 000 millipascal second (mPa·s) (Note 3) at temperatures up to 175°C (347°F).

NOTE 1—Although precision has not been studied, this procedure may be adaptable to viscosities higher than the present 200 000-mPa·s limit and temperatures above 175°C (347°F). Equipment described in this test method permits testing of materials having viscosities as high as 16×10^6 mPa·s and provides temperatures up to 260°C (500°F).

NOTE 2—For petroleum waxes and their blends having apparent viscosities below 15 mPa·s, Test Method D445 is especially applicable.

NOTE 3—One pascal second (Pa·s) = 1000 centipoise (CP); one millipascal second = one centipoise.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

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.*

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

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3.1.2 *viscosity*—the ratio of shear stress to shear rate. The 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.

4. Summary of Test Method

4.1 A representative sample of the molten material to be tested is maintained in a thermally controlled sample chamber. Apparent viscosity is determined under temperature equilibrium conditions using a precision rotating spindle type viscometer. Data obtained at several temperatures can be plotted on appropriate semi-logarithmic graph paper and apparent viscosity at intermediate temperatures can be estimated.

5. Significance and Use

5.1 This test method 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 test method may not correlate well with end-use applications where high shear rates are encountered.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum ~~Products~~ Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.10.0A on Physical/Chemical Properties.

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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.

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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 method 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.

5.3 Approximate shear rates using various spindles are shown in Table A1.1.

6. Apparatus

6.1 *Viscometer,* rotating spindle type with leveling stand.[3]

6.2 *Viscometer Spindles,* stainless steel.[3]

6.3 *Sample Chamber,* with precision proportional temperature controller[3,4] that provides control accuracy of ~~±1.0°C (1.8°F)~~ ±1.0°C (1.8°F) or better through the range from 100 to 200°C (212 to 392°F).

6.4 *Graph Paper,* semi-logarithmic.

7. Calibration

7.1 The viscometer is precalibrated using Newtonian fluids by the manufacturer. No zero adjustment is provided, since experience has shown that the zero point will not vary due to changes in the spring. The viscometer and spindles are precision equipment and should be kept from undue shock and mishandling. Physical damage to the instrument will often reveal itself as erratic or no oscillation of the pointer when the instrument, with or without the spindle in place, is operated in air. When operating normally, the pointer will be stable and have free oscillation about the zero point in air.

7.2 The instrument may be further calibrated using standard reference fluids. Suitable fluids are available in nominal viscosities up to 15 000 mPa·s at 149°C (300°F).[5] The procedure for instrument calibration using standard reference fluids is that encompassed by this test method. Results obtained using standard reference fluids should not deviate from the nominal viscosity by more than 2 %.

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7.3 The temperature controller of the type recommended for this procedure is factory calibrated and has control capability of ±0.5 % of the control point (±1.0°C at 175°C). To further check the controller and further establish controller settings, use the following procedure: Place a sufficient quantity of low viscosity (500 mPa·s or less) hot melt in the sample container to permit immersion of the appropriate ASTM thermometer to the proper depth. Do not permit the thermometer bulb to rest on the bottom of the sample container. Suitable thermometers are shown in Table 1.

NOTE 4—Particular care must be taken not to overflow the sample chamber when using the 100°C, 76-mm immersion thermometer.

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7.3.1 Insert the thermometer through the insulating cover of the sample container and hold it in place at the point required for proper immersion depth. Adjust the thermal controller to provide the desired test temperature. Rotate the thermometer during temperature reading to minimize the effect of thermal gradients in the sample. Continue temperature readings and controller adjustment until minimum deviation from test temperature is obtained. Minimum deviation may vary between laboratories, depending upon the controller, but should in no case exceed ±0.5°C (0.9°F). Repeat this procedure for any test temperature desired within the scope of this test method.

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[3] The sole source of supply of the viscometers and accessories known to the committee at this time is Brookfield Engineering Laboratories, Inc., Stoughton, MA 02072. 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.

[4] The sole source of supply of the temperature controller known to the committee at this time is Athena Controls, Inc., 2 Union Road, West Conshohocken, PA 19428. 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] The sole source of supply of the calibration fluids known to the committee at this time is Brookfield Engineering Laboratories, Inc., Stoughton, MA 02072 or Cannon Instrument Co., P. O. Box 16, State College, PA 16801. 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.

TABLE 1 Suitable ASTM Thermometers

Temperature Range	Immersion, mm	Scale Error, max	ASTM Thermometer Number
90°C to 170°C	51	0.2°C	35C-62
194°F to 338°F	51	0.5°F	35F-62
145°C to 205°C	76	0.4°C	100C-68

8. Procedure

8.1 *Selection of Spindle*—From the estimated viscosity of the sample and Table A1.1 in the Annex, select a viscometer and spindle combination that will produce readings in the desired range.

NOTE 5—Use only the spindle shown to be appropriate for the viscometer to be used.

8.1.1 Where more than one spindle is available for the range selected, choose the spindle that produces results nearest the midpoint of the measurable viscosity range. Viscometer scale readings must be within the 10 to 95 range.

NOTE 6—Care must be taken in the storage and handling of spindles and assemblies. Protect them from dust, corrosive deposits, and mechanical abuse. Avoid touching the calibrated section of the spindle with the hands. Clean the spindle and sample chamber thoroughly after each use. A recommended cleaning procedure is included in Annex A2.

8.2 *Preparation of Sample*—Place the required amount of representative sample (see Table 2) measured to the nearest 0.005 g (or 0.05 mL if handled in the molten state) in the sample chamber. Melt the sample in an oven set at the desired test temperature or in the thermo-container preheated to the desired test temperature. Avoid excessive or prolonged heating of the sample to minimize thermal and oxidative effects. Use a fresh sample for each temperature for which a determination is to be made.

8.3 *System Alignment and Spindle Insertion*—After the sample is completely melted, lower the properly aligned and leveled viscometer until the tips of the alignment bracket just touch the top of the thermo-container, making contact directly behind the locating ring. Raise the viscometer, positioning the tips of the alignment bracket 2 mm (1⁄16 in.) above the top of the thermo-container. Using both hands, gently slide the thermo-container base until the tips of the alignment bracket *just touch* the locating ring. *Do not* forcibly displace the alignment bracket (see Fig. 1). Screw the link coupling nut onto the viscometer coupling nut (note left-hand thread). Connect the coupling link to the spindle (and the coupling nut). Lower the spindle into the sample chamber and connect the link coupling nut to the viscometer coupling nut, noting the left-hand thread. Pick up the insulating cap and place it over the sample chamber (see Fig. 1).

8.4 *Viscosity Determination*—Ensure that the material in the sample chamber is completely molten and that temperature controller settings are proper. Turn on the viscometer and allow the spindle to rotate at the lowest spindle speed available to minimize temperature gradients in the sample as well as possible shear effects. When temperature equilibrium is indicated, turn off the viscometer, remove the insulating cap, raise the viscometer and spindle, and inspect the liquid level on the spindle shaft. It should extend about 3 mm (⅛ in.) up the spindle shaft beyond the upper, tapered portion of the spindle. If the liquid level varies significantly from this, add or remove sample to provide this level. Replace the insulating cap, and allow the unit to reestablish temperature equilibrium with the spindle rotating at the lowest available speed. Continue spindle rotation for 15 min after apparent equilibrium. Increase the spindle speed to that required to produce a scale reading nearest the midpoint of the scale, but in no case outside the 10 to 95-unit range. Engage the pointer clutch and stop the viscometer motor when the pointer is in view. Record the scale reading. Restart the viscometer motor, and allow at least five additional revolutions of the spindle. Engage the pointer clutch and stop the viscometer motor with the pointer in view. Record the second dial reading. Repeat the above operation until three consecutive scale readings are obtained that differ by no more than 0.5 unit.

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9. Calculation

9.1 Determine the average of the three consecutive scale readings which differ by no more than 0.5 scale unit. To convert to millipascal seconds, multiply this value by the appropriate factor taken from either the instrument instruction manual or Table A1.2. Repeat this for each temperature.

NOTE 7—If it is necessary to interpolate for viscosity values at intermediate temperatures, plot a series of observed apparent viscosity values on the logarithmic scale and the corresponding test temperatures on the linear scale of appropriate semi-logarithmic paper, using a series of at least three different temperatures. From the plot, determine the apparent viscosity at any temperature requested, within the range of test temperatures.

TABLE 2 Sample Size Requirement

Spindle	Approximate Volume, mL	Approximate Sample Weight, g[A]
SC 4-18	8.00	6.40
SC 4-21	8.00	6.40
SC 4-27	10.50	8.40
SC 4-28	11.50	9.20
SC 4-29	13.00	10.40
SC 4-31	10.00	8.00
SC 4-34	9.50	7.60

[A] Based on typical molten specific gravity of 0.800. If the specific gravity of the material to be tested varies greatly from this value, sample size must be adjusted to ensure proper liquid level on the spindle shaft.

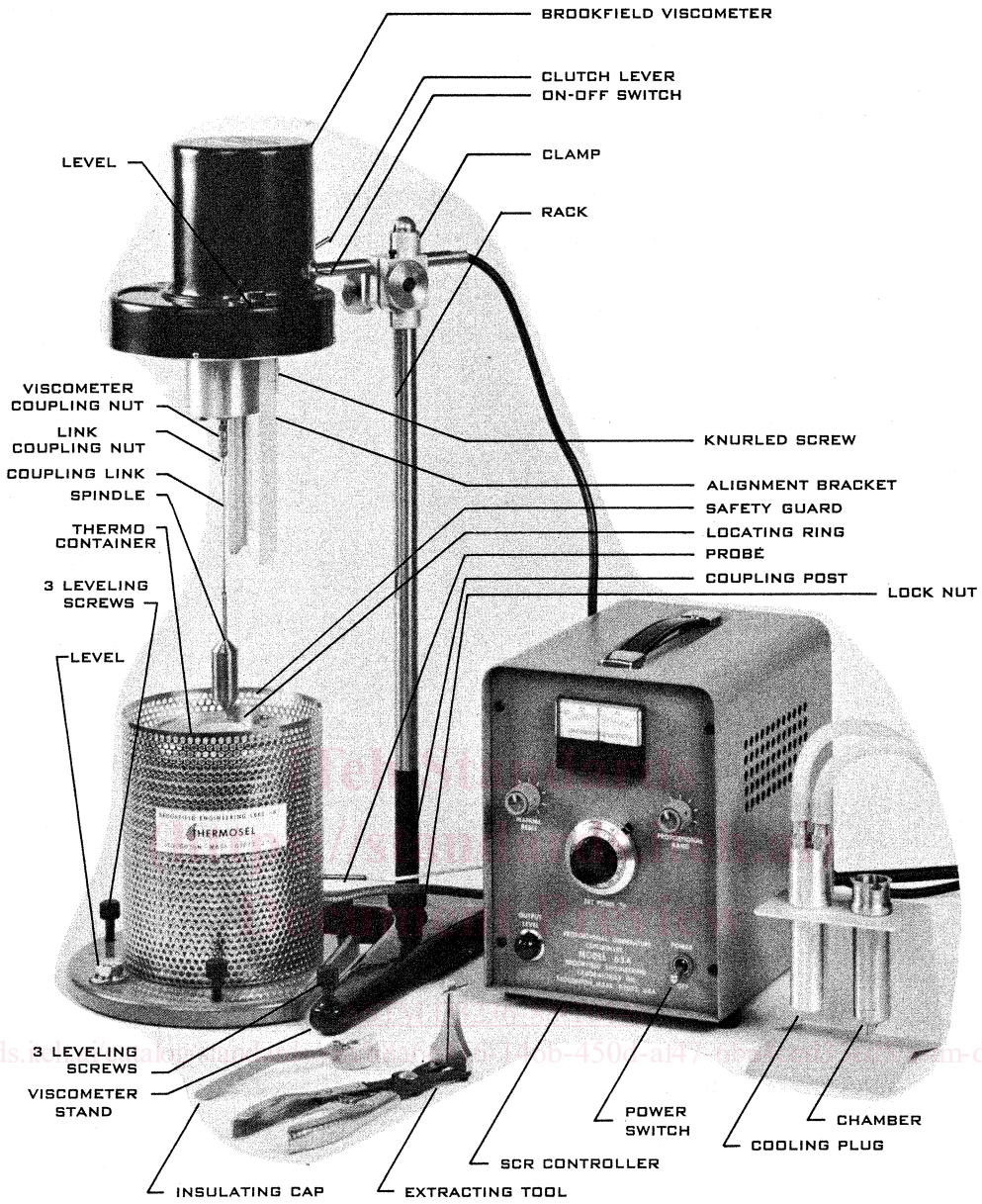

FIG. 1 Apparatus for Viscosity Determination

10. Report

10.1 Report the apparent viscosity at a given temperature along with the particulars of the instrument model, the spindle number and rotational speed. *Example:* Apparent viscosity at 125°C (RVT, SC 4-28, 20 rpm)—20 000 mPa·s.

NOTE 8—If it is desired to report the shear rate corresponding to the instrument/spindle/speed combination, refer to Table A1.1 for the appropriate calculation.

11. Precision and Bias

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

11.1.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparataus under constant operating conditions on identical test material would, in the normal and correct operation of the test method, exceed the following values in one case in twenty:

$$8.8\,\%\text{ of the mean of the two results.} \tag{1}$$

11.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories in identical test material would, in the long run, exceed the following value only in one case in twenty:

$$25.4\,\%\text{ of the mean of the two results.} \tag{2}$$