



# Standard Test Method for Apparent Viscosity of Hot Melt Adhesives and Coating Materials<sup>1</sup>

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 ( $\epsilon$ ) 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  $16 \times 10^6$  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.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

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

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

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<sup>2</sup> 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.

may vary with the spindle and rotational speed selected because many hot melts are non-Newtonian.

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.

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.

## 6. Apparatus

6.1 *Viscometer, Concentric Cylinder Rotational*—The essential instrumentation required providing the minimum rotational viscometer analytical capabilities include:

6.1.1 *Drive motor*, to apply a unidirectional rotational displacement to the specimen at a rate of 0.5 r/min to 60 r/min constant to  $\pm 1$  %.

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

6.1.2 *Force sensor*, to measure the torque developed by the specimen in response to the rotational displacement.

6.1.3 *Coupling shaft*, or other means to transmit the rotational displacement from the motor to the specimen.

NOTE 4—It is helpful to have a mark on the shaft to indicate appropriate test fluid level.

6.1.4 *Stainless steel rotational element, spindle, or tool*, for the type shown in Fig. 1 to fix the specimen between the draft shaft and a stationary position.

6.1.5 *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 viscometry are torque, rotational speed, temperature, and time.

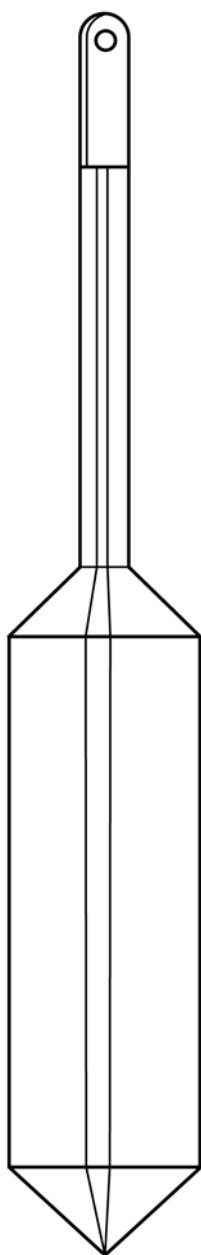


FIG. 1 Spindle Configuration

NOTE 5—Manual observation and recording of data are acceptable.

6.1.6 *Temperature measuring device*, to provide an indication of the specimen temperature over their range of 100 °C to 200 °C to within  $\pm 0.2$  °C.

6.1.7 *Stand*, to support, level, and adjust the height of the drive motor, shaft and rotational element.

6.1.8 *Specimen container*, fitted with an insulated cover, to contain the test specimen during testing.

6.1.9 Auxiliary instrumentation considered necessary or useful in conducting this method includes:

6.1.9.1 Data analysis capability to provide viscosity, stress, or other useful parameters derived from measured signals.

6.1.9.2 Level to indicate the vertical plumb of the drive motor, shaft, and rotational element.

6.1.9.3 A guard to protect the rotational element from mechanical damage.

6.2 *Temperature Bath* and precision proportional temperature controller<sup>3</sup> that provides a controlled isothermal temperature environment over the range of 100 °C to 200 °C (212 °F to 392 °F) constant to accuracy of  $\pm 1.0$  °C (1.8 °F).

6.3 *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 torque sensor. 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 torque indication or no oscillation of the shaft when the instrument, with or without the spindle in place, is operated in air. When operating normally, the torque indication 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).<sup>4</sup> 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 %.

7.3 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.

<sup>3</sup> The sole source of supply of the temperature controller known to the committee at this time is Athena Controls, Inc., 2 Union Rd., 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,<sup>1</sup> which you may attend.

<sup>4</sup> 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,<sup>1</sup> which you may attend.

NOTE 6—Do not permit the temperature sensor to rest on the bottom of the sample container.

7.3.1 Insert the temperature sensor 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 temperature sensor 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  $\pm 0.5$  °C (0.9 °F). Repeat this procedure for any test temperature desired within the scope of this test method.

## 8. Procedure

8.1 *Selection of Spindle*—The measurement range of the rotational viscometer is determined by the dimensions of the spindle, the rotational speed, the size and shape of the container and the full scale range of the motor. From the estimated viscosity of the sample and the operations manual for the viscometer, select a spindle and rotational speed combination that will produce readings in the desired range.

NOTE 7—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 torque scale readings shall be within the 10 % to 95 % range.

NOTE 8—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 A1](#).

8.2 *Preparation of Sample*—Using a fresh sample for each measurement, place the required amount of representative sample measured to the nearest 0.005 g (or 0.05 mL if handled in the molten state) in the specimen container. Melt the sample in an oven set at the desired test temperature or in the temperature bath preheated to the desired test temperature.

NOTE 9—Avoid excessive or prolonged heating of the sample to minimize thermal and oxidative effects.

8.3 *System Alignment and Spindle Insertion*—After the sample is completely melted, lower the properly aligned and leveled viscometer spindle into the specimen container. Place the insulating cap over the specimen container.

### 8.4 Viscosity Determination:

8.4.1 Ensure that the material in the sample chamber is completely molten and that the test specimen is equilibrated to the selected temperature controller settings.

8.4.2 Initiate the spindle rotation at the lowest spindle speed available to minimize temperature gradients in the sample as well as possible shear effects.

8.4.3 When temperature equilibrium is indicated, stop the spindle rotation, remove the insulating cap, raise the viscometer and spindle, and inspect the liquid level on the spindle shaft. It should extend about 3 mm ( $\frac{1}{8}$  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.

8.4.4 Lower the spindle back into the test specimen. Replace the insulating cap. Initiate the spindle rotation at the lowest available speed, and allow the temperature equilibrium to be reestablished.

8.4.5 Continue spindle rotation for 15 min after apparent equilibrium.

8.4.6 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-unit to 95-unit range. Record the scale reading.

8.4.7 After at least five additional revolutions of the spindle, record the second indicator reading.

8.4.8 Repeat 8.4.6 to until three consecutive scale readings are obtained that differ by no more than 0.5 unit.

## 9. Calculation

9.1 Determine the average of the three consecutive scale readings which differ by no more than 0.5 scale units from 8.4.7. Convert the indicatory reading to viscosity using the procedure described in the instrument instruction manual. Repeat this for each temperature.

NOTE 10—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.

## 10. Report

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

## 11. Precision and Bias

11.1 The precision of this test method was determined in an interlaboratory study using six representative wax-based hot melt materials. Tests were conducted at three temperatures by seven to eleven laboratories using four instrument models and seven spindles from a single manufacturer. Each spindle was appropriately tailored to the viscometer.

11.1.1 *Repeatability*—Within laboratory variability may be described using the repeatability value ( $r$ ) obtained by multiplying the repeatability relative standard deviation by 2.8. The difference between two test results, obtained by the same operator with the same apparatus 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:

$$8.8\% \text{ of the mean of the two results.} \quad (1)$$

11.1.2 *Reproducibility*—The between laboratory variability may be described using the reproducibility value ( $R$ ) obtained by multiplying the reproducibility relative standard deviation by 2.8. 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:

$$25.4\% \text{ of the mean of the two results.} \quad (2)$$

11.1.3 A review of that portion of the data which can be considered comparable at equal shear rates indicates that those laboratories capable of comparing data at equal shear rates can expect improvement in reproducibility. It is estimated that under conditions of equal shear rate reproducibility, the values would in the long run, exceed the following in one case in twenty:

$$15\% \text{ of the mean of the two results.} \quad (3)$$

11.1.4 A summary of data generated in this interlaboratory test program is shown in **Table 1** and **Table 2**.

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

## 12. Keywords

12.1 adhesives; apparent viscosity; coating materials; hot melt adhesives; rotational viscometry; viscosity

**TABLE 1 Summary of Precision Estimates, Total Interlaboratory Study Data**

Sample No.	Average Viscosity, mPa·s	S <sub>a</sub>	deg freedom	S <sub>a</sub> + b	deg freedom	S <sub>a</sub> %	S <sub>a</sub> + b %
MI69-28							
100 °C	65.2	2.02	11	3.64	10	3.10	5.58
125 °C	38.7	1.07	10	2.39	9	2.76	6.18
150 °C	25.2	0.27	10	2.20	9	1.07	8.73
MI69-29							
100 °C	170.2	4.39	10	19.7	9	2.58	11.58
125 °C	93.4	2.54	9	9.10	8	2.72	9.74
150 °C	55.8	1.00	9	4.23	8	1.79	7.59
MI69-30							
125 °C	232 100	8540	6	9040	5	3.68	3.88
150 °C	128 167	4280	6	7380	5	3.34	5.76
175 °C	74 021	1840	7	5310	6	2.49	7.17
MI69-31							
125 °C	3416	117	11	207	10	3.43	6.06
150 °C	1456	43.2	11	70.0	10	2.96	4.81
175 °C	756	16.7	11	46.2	10	2.21	6.11
MI69-32							
125 °C	66 560	2850	9	7410	8	4.28	11.13
150 °C	26 800	1130	9	2750	8	4.22	10.26
175 °C	11 850	449	9	1030	8	3.79	8.69
MI69-33							
125 °C	165 300	3730	7	7320	6	2.26	4.43
150 °C	74 590	1880	8	2650	7	2.52	3.78
175 °C	35 840	1820	9	2420	8	5.07	6.75

*Pooled Data:*

$$\begin{aligned} S_a \text{ (overall)} &= 3.14\% \text{ at } 162 \text{ deg freedom} \\ 3.14 \times 2.80 &= 8.8\% \text{ relative} \end{aligned}$$

$$\begin{aligned} S_a + b \text{ (overall)} &= 7.60\% \text{ at } 7 \text{ deg freedom} \\ 7.60 \times 3.34 &= 25.4\% \text{ relative} \end{aligned}$$

**TABLE 2 Summary of Precision Estimates, Selected Shear Equalized Data**

Sample No.	Average Viscosity, mPa·s	Sa	deg freedom	Sa + b	deg freedom	Sa %	Sa + b %
MI69-28							
100 °C	64.4	1.33	7	2.15	6	2.07	3.34
125 °C	38.0	0.56	5	0.75	4	1.47	1.97
150 °C	24.1	0.51	6	0.47	5	2.12	1.95
MI69-29							
100 °C	163.2	0.63	6	4.09	5	0.38	2.51
125 °C	90.3	0.75	5	1.90	4	0.90	2.11
150 °C	53.7	0.83	5	1.30	4	1.55	2.42
MI69-30							
125 °C	229 900	10 240	4	15 700	3	4.59	7.04
150 °C	130 900	5200	5	5200	4	3.97	3.97
175 °C	74 640	830	5	4750	4	1.11	6.36
MI69-31							
125 °C	3429	40	6	77.6	5	1.17	2.26
150 °C	1474	12.3	6	28.3	5	0.83	1.92
175 °C	770	15.6	5	21.0	4	2.03	2.73
MI69-32							
125 °C	68 680	1450	5	3440	4	2.11	5.00
150 °C	28 000	255	6	1670	5	0.91	5.96
175 °C	12 350	361	6	911	5	2.92	7.38
MI69-33							
125 °C	167 700	1730	5	5000	4	1.03	2.98
150 °C	75 700	756	7	1645	6	1.00	2.17
175 °C	37 350	685	5	1070	4	1.83	2.86

*Pooled Data:*

Sa (overall) = 1.83 % at 99 deg freedom  
 $1.83 \times 2.82 = 5.16$  % relative

Sa + b (overall) = 3.81 % at 4 deg freedom  
 $3.81 \times 3.92 = 14.93$  % relative

## ANNEX

### (Mandatory Information)

#### A1. APPARATUS AND EQUIPMENT CLEANING

A1.1 The spindle and specimen containers may be cleaned with most commercial chlorinated or hydrocarbon solvents. If the solvents are heated, adequate precautions must be taken to avoid toxicity, flammability, or explosive hazards. Spindles and

containers must not come in contact with sulfuric acid, hydrofluoric acid, hydrochloric acid, and ferric chloride. Care must also be exercised to avoid scratching or deforming the spindles.

#### SUMMARY OF CHANGES

Subcommittee D02.10 has identified the location of selected changes to this standard since the last issue (D3236 – 88 (2014)) that may impact the use of this standard. (Approved April 1, 2015.)

(1) Revisions made throughout to include generic rotational viscometer apparatus information.

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