Designation: D 6267 – 98

Standard Test Method for Apparent Viscosity of Hydrocarbon Resins at Elevated Temperatures[1]

This standard is issued under the fixed designation D 6267; 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 hydrocarbon resins having apparent viscosities up to 2 000 000 millipascal seconds (mPa·s) (Note 1) at temperatures up to 300 °C (572 °F).

NOTE 1—The cgs unit of viscosity is the poise (dyne-sec/cm^2) and is equivalent to 0.1 Pa·s. The centipoise (cP) is one millipascal second (mPa·s) and is frequently used as a viscosity unit.

1.2 The values stated in SI units are to be regarded as the standard. The values given 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 E 1 Specification for ASTM Thermometers[2]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *viscosity*—the ratio of shear stress to shear rate.

3.1.1.1 *Discussion*—The viscosity of a liquid is a measure of the resistance to flow of the liquid. The SI unit of dynamic viscosity is the pascal second. For a Newtonian liquid, the viscosity is constant at a range of shear rates. For a non-Newtonian liquid, viscosity will vary depending on shear rate.

3.1.2 *apparent viscosity*—the viscosity determined by this test method, expressed in millipascal seconds.

3.1.2.1 *Discussion*—Its value may vary with the spindle and rotational speed selected because many resins are non-Newtonian.

4. Summary of Test Method

4.1 The viscometer described in this test method can be used to determine the apparent viscosity of hydrocarbon resins at elevated temperatures. Apparent viscosity is determined under temperature equilibrium conditions using a rotating spindle type viscometer. The torque on a spindle rotating in a thermostatted sample holder containing a small amount of sample is used to measure the relative resistance to rotation. A factor is applied to the torque reading to yield the viscosity in mPa·s.

5. Significance and Use

5.1 This test method is used to measure the apparent viscosity of hydrocarbon resins *at elevated temperatures. Elevated temperature viscosities of hydrocarbon resin may be related to the properties of coatings, adhesives and the like, containing such a resin.*

5.2 Materials of the type described in this procedure may be 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 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. Comparisons between non-Newtonian viscosity values should be made only for measurements made with similar viscometers under conditions of equivalent shear.

5.3 Approximate shear rates using various spindles are shown in Table A1.1 in Annex A1. to this procedure.

6. Apparatus

6.1 *Rotational Viscometer*—rotating spindle type with leveling stand.[4]

6.2 *Viscometer Spindles*, stainless steel[3]

NOTE 2—**Caution:** Care must be taken in the storage and handling of spindles and assemblies. Protect them from scratches, dust, corrosive-deposits, and mechanical abuse. Replace the spindle extension if it is bent. Avoid touching the calibrated section of the spindle with hands. Clean the spindle and sample chamber thoroughly after each use. A recommended

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[2] *Annual Book of ASTM Standards*, Vol 14.03.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Supporting data are available from ASTM Headquarters. Request RR: D01–1109.

cleaning procedure is included in the procedure.

6.3 *Temperature Controller Thermocontainer, and Sample Chamber*, designed for use with the viscometer in 6.1, complete with locating ring, leveling screws, safety guard, spindle extension, insulating cap, upright rod, cooling plug (optional) and extracting tool. The precision temperature controller provides control accuracy of ±1.0°C or better through the range from 100 to 150°C (212 to 302°F) and ± 2.0°C or better through the range from 150 to 300°C (302 to 572°F).

7. Assembly of Apparatus

7.1 Assemble the apparatus according to the manufacturer's instructions.

8. Calibration

8.1 For dial reading viscometers, no zero adjustment is provided since experience has shown that the zero point will not vary due to changes in the spring. A digital viscometer should be zeroed according to the manufacturer's instructions. 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 (dial reading models) when the instrument, with or without the spindle in place, is operated in air. When operating normally, the reading will be stable and have free oscillation about the zero point in air.

8.2 The calibration of the instrument may be verified using standard reference fluids. Suitable fluids are available in nominal viscosities up to 15 000 mPa·s at 149°C (300°F).[3] 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 the following amount:

$$\sqrt{a^2 + b^2} \quad (1)$$

where "*a*" is 1 % of the full measurement range under the conditions of the test, and "*b*" is 1 % of the nominal viscosity of the calibration fluid. If results deviate by more than this value, the instrument should be removed from use and repaired.

8.3 The temperature controller is factory calibrated and has control capability of ± 0.5 % of the control point (± 1.0°C at 175°C). To check the controller and verify the calibration of the controller settings, use the procedure in 8.3.1.

8.3.1 Place a sufficient quantity of silicone oil in the sample container to permit immersion of the appropriate ASTM thermometer to the proper depth. See Specification E 1. *Insert the thermometer into the oil and hold it in place at the point required for proper immersion depth.* Do not permit the thermometer bulb to rest on the bottom of the sample container. Suitable thermometers are shown in Table 1.

NOTE 3—Particular care must be taken not to overflow the sample chamber when using the 100C, 76-mm immersion thermometer.

8.3.2 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 set point to provide the desired temperature.* Repeat this procedure for any test temperature desired within the scope of this procedure.

TABLE 1 Suitable ASTM Thermometers

Temperature Range	Immersion mm	Scale Error, max	ASTM Thermometer Number
90°C - 170°C	51	0.2°C	35C-62
94°F - 338°F	51	0.5°F	35F-62
145°C - 206°C	76	0.4°C	100C-68

TABLE 2 Sample Size Guideline

Spindle	Approximate Volume, mL	Approximate Sample Weight, g
SC 4-18	8.0	6.4
SC 4-21	8.0	6.4
SC 4-27	10.5	6.4
SC 4-28	11.5	9.2
SC 4-29	13.0	10.4
SC 4-31	10.0	8.0
SC 4-34	9.5	7.6

TABLE 3 Interlaboratory Precision Study Results

Material	Average, cP	Sr	SR	r	R
Standard Oil	4657.3	23.0	58.2	64.4	162.9
Resin A, Temperature 1	9657.6	191.6	925.9	536.4	2592.6
Resin A, Temperature 2	764.8	12.8	64.2	35.8	179.8
Resin B, Temperature 1	1513.4	26.2	94.8	73.3	654.4
Resin B, Temperature 2	480.2	6.6	31.5	18.4	88.3
Resin C, Temperature 1	4420.0	58.6	172.8	164.2	438.8
Resin C, Temperature 2	149.1	4.5	7.10	12.6	19.9

9. Procedure

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

NOTE 4—Use only spindles shown to be appropriate for the viscometer to be used.

9.1.1 Where more than one spindle is available for the range selected, choose the spindle that produces a dial or display reading between 10 and 100 *% of full scale*. The goal is to select a combination whose range brackets the estimated viscosity of the sample.

NOTE 5—Accuracy improves as the reading approaches 100 *% of full scale*. If the reading is over 100 *% of full scale*, select a lower speed and/or a smaller spindle. If the reading is under 10 *% of full scale*, select a higher speed or a larger spindle, or both. Whenever possible, when conducting multiple comparative tests, the same spindle/speed combination should be used for all tests. When a test must be performed at several speeds, select a spindle that produces on-scale readings at all required speeds. This may necessitate using a dial or display reading less than 10 *% of full scale*, which is acceptable as long as the reduced accuracy of such a reading is recognized.

9.2 *Preparation of Sample*—Weigh the amount of representative sample, which when melted will be equivalent to the test volume (see Table 2), into the sample chamber, Insert the sample chamber into the thermo-container, preheated to the desired test temperature.