



Designation: D6267/D6267M – 13

# Standard Test Method for Apparent Viscosity of Hydrocarbon Resins at Elevated Temperatures<sup>1</sup>

This standard is issued under the fixed designation D6267/D6267M; 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 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 SI unit of (dynamic) viscosity is the pascal second. The centipoise (cP) is one millipascal second (mPa·s) and is frequently used as a viscosity unit.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

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>

- D6440 Terminology Relating to Hydrocarbon Resins
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- E1 Specification for ASTM Liquid-in-Glass Thermometers

## 3. Terminology

3.1 The definitions in Terminology D6440 are applicable to this test method.

### 3.2 Definition Specific to This Standard:

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.34 on Pine Chemicals and Hydrocarbon Resins.

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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.

3.3 *apparent viscosity, n*—of a hydrocarbon resin, the viscosity determined by this test method, expressed in millipascal seconds.

3.3.1 *Discussion*—Its value may vary with the spindle and rotational speed selected.

## 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 viscosity values of a hydrocarbon resin may be related to the properties of coatings, adhesives and the like, containing such a resin.

5.2 For hydrocarbon resins, values of apparent viscosity will usually be a function of shear rate under the conditions of test. Although the type of viscometer described in this test method operates under conditions of relatively low shear rate, shear rate depends on the spindle and rotational speed selected for a determination; therefore, comparisons between apparent viscosity values should be made only for measurements made with similar viscometers under conditions of equivalent shear rate.

## 6. Apparatus

6.1 *Rotational Viscometer*—rotating-spindle type with leveling stand.

6.2 *Viscometer Spindles*, stainless steel. (**Warning**—Care must be taken in the storage and handling of spindles and assemblies. Protect them from scratches, dust, corrosion or 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 cleaning procedure is included in the procedure.)

6.3 *Temperature Controller, Thermocontainer (a heater for the sample chamber), and Sample Chamber*, designed for use with the viscometer in 6.1, complete with locating ring, leveling screws, safety guard, spindle extension, insulating cap, alignment bracket, cooling plug (optional) and extracting tool. The precision temperature controller shall provide control accuracy of  $\pm 1.0^\circ\text{C}$  or better through the range from 100 to  $150^\circ\text{C}$  [212 to  $302^\circ\text{F}$ ] and  $\pm 2.0^\circ\text{C}$  or better through the range from 150 to  $300^\circ\text{C}$  [302 to  $572^\circ\text{F}$ ].

## 7. Assembly of Apparatus

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

## 8. Calibration

8.1 A digital viscometer should be zeroed according to the manufacturer's instructions. For a dial-reading viscometer, no zero adjustment is required, since experience has shown that the zero point will not vary due to changes in the spring.

NOTE 2—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 reading when the instrument, with or without the spindle in place, is operated in air. When operating normally, the reading in air will be stable and have free oscillation about the zero point.

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  $149^\circ\text{C}$  [ $300^\circ\text{F}$ ]. The procedure for instrument calibration using standard reference fluids shall be that described 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 the results deviate by more than this value, the instrument should be removed from use and repaired.

8.3 To check the controller and verify the calibration of the controller settings, use the procedure in 8.3.1 and 8.3.2.

8.3.1 Place enough silicone oil (or other high-boiling material that is liquid under the conditions of the determination) in the sample container to permit immersion of the appropriate ASTM thermometer to the proper depth. Suitable thermometers are shown in Table 1 in accordance with Specification E1. Temperature measuring devices such as RTDs, thermocouples

**TABLE 1 Suitable ASTM Thermometers**

Temperature Range	Immersion mm	Scale Error, max
$90^\circ\text{C}$ - $170^\circ\text{C}$	51	$0.2^\circ\text{C}$
$94^\circ\text{F}$ - $338^\circ\text{F}$	51	$0.5^\circ\text{F}$
$145^\circ\text{C}$ - $206^\circ\text{C}$	76	$0.4^\circ\text{C}$

and liquid-in-glass thermometers with temperature range and accuracy consistent with thermometers in Table 1 may be used. Adjust the thermal controller setpoint to provide the desired temperature.

8.3.2 Insert the thermometer through the insulating cover of the sample container, into the liquid, and hold it in place at the level required for proper immersion depth. *Do not permit the thermometer bulb to rest on the bottom of the sample container.* Adjust the thermal controller set point to provide the desired temperature. Repeat this procedure for each test temperature desired.

NOTE 3—Particular care must be taken not to overflow the sample chamber when using the  $100^\circ\text{C}$ , 76-mm immersion thermometer, since the volume of the immersed stem is relatively large.

## 9. Procedure

9.1 *Selection of Spindle*—From the estimated viscosity of the sample and the manufacturer's instructions, select a viscometer and spindle combination that will produce readings in the desired range.

NOTE 4—Use only spindles 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 display or dial 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 or a smaller spindle, or both. 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 display or dial reading less than 10 % of full scale for some temperatures, 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 desired test volume (see Table 2), into the sample chamber. Insert the sample chamber into the thermocontainer, preheated to the desired test temperature.

NOTE 6—Use a fresh sample for each temperature for which a determination is to be made. The sample should be uniform in appearance and free of foreign material.

9.3 *System Alignment and Spindle Insertion*—Raise the viscometer to clear the top of the thermocontainer. Connect the spindle extension to the spindle and to the coupling nut. If necessary, connect the coupling nut to the viscometer shaft

**TABLE 2 Sample Size Guideline**

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