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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

~~1.1This test method covers the determination of the apparent viscosity of hydrocarbon resins having apparent viscosities up to 2 000 000 millipascal seconds (mPa·s) (~~*

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 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 *ASTM Standards:* [2]

D 6440 Terminology Relating to Hydrocarbon Resins

E~~1Specification for ASTM Liquid-in-Glass Thermometers~~ 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E~~691Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method~~ 2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

3. Terminology

3.1 The definitions in Terminology D 6440 are applicable to this test method.

3.2 *Definition Specific to This Standard*:

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.

[1] 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 D01.38 on Hydrocarbon Resins.

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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.**

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.

~~5.3Approximate 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.

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 ±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 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°C (300°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:

$$\sqrt{a^2 + b^2} \tag{1}$$

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~~E 2251. 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°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 ~~Table A1.1, Annex A1;~~the manufacturer's instructions, select a viscometer and spindle combination that will produce readings in the desired range.

TABLE 1 Suitable ASTM Thermometers

Temperature Range	Immersion mm	Scale Error, max~~ASTM Thermometer Number~~
~~90°C - 170°C~~	~~51~~	~~0.2°C35C 62~~
90°C - 170°C	51	0.2°C
~~94°F - 338°F~~	~~51~~	~~0.5°F35F 62~~
94°F - 338°F	51	0.5°F
~~145°C - 206°C~~	~~76~~	~~0.4°C100C 68~~
145°C - 206°C	76	0.4°C

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 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 (note left-handed thread). With the viscometer aligned and leveled, lower the entire assembly until the spindle touches the sample in the chamber. Do not force the spindle into the sample, since this may result in bending the spindle extension or causing it to detach from the spindle shaft. Allow the sample to melt completely, but avoid prolonged heating—to minimize thermal and oxidative changes to the test material. Lower the assembly so that the tips of the alignment bracket are 2 mm (1⁄16 in.) above the horizontal surface of the locating ring, making contact with the vertical curve. A scribed line on the back of the vertical curve is the 2 mm (1⁄16 in.) reference point. Do not forcibly displace the alignment bracket. Verify that the viscometer and thermocontainer are level. Place the insulating cap over the sample chamber inlet.

9.4 *Viscosity Determination*—Ensure that the material in the sample chamber is completely molten and that temperature controller setting is proper. Turn on the viscometer, and allow the spindle to rotate. When temperature equilibrium is indicated, typically after about 10 to 15 min., turn off the viscometer, remove the insulating cap, and inspect the liquid level on the spindle shaft. This level should be about 3 mm (1⁄8 in.) above the upper cone of the spindle. *Do not overfill.* Replace the insulating cap, and allow the unit to reestablish temperature equilibrium. ~~Continue spindle rotation for 15 min after apparent equilibrium. Increase~~ As needed, adjust the spindle speed to *maximize the on-scale reading at the test temperature*. Continue spindle rotation for 15 min after apparent equilibrium. For digital viscometers, record the display reading. For dial-reading viscometers, engage the pointer clutch and stop the viscometer motor when the pointer is in view. Record the dial reading. ~~For digital viscometers, record the display reading.~~ Repeat this operation until 3 consecutive readings differ by no more than 0.5 scale units (or 0.5 % of the viscosity value for a direct-reading viscometer.) (**Warning** —The spindle extension link should not come in contact with the insulating cap when rotating. The rotating spindle must not come in contact with the inside wall of the sample chamber.)

9.5 *Cleaning the Viscometer*—Remove the insulating cap and turn off the motor. Unhook the spindle extension from the coupling nut and remove the spindle from the sample chamber. Lift the sample chamber from the thermocontainer using the extracting tool and discard the sample in an appropriate manner. With the sample chamber removed, (optionally) cool the thermocontainer by inserting the cooling plug into the sample chamber well and circulating a cooling medium (tap water) through it. Clean the spindle and sample chamber using an appropriate solvent. Care must also be exercised to avoid scratching or deforming the spindles.

10. Calculation

10.1 Determine the average of the three acceptable scale readings that differ by no more than 0.5 scale unit. If necessary, to convert to millipascal seconds, multiply the scale reading by the appropriate factor taken from ~~either~~ the instrument instruction

TABLE 2 Sample Size Guideline

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