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

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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 troque 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

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.

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

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 thermo-container. Connect the spindle extension to the spindle and to the coupling nut.

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; 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. An etched 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 thermo-container 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 settings are 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. The liquid level *on the spindle shaft*, should be about 3 mm (⅛ in.) above the upper cone. *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 the spindle speed to *maximize the reading at the test temperature*. 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. For digital viscometers, record the display reading. Repeat this operation until 3 consecutive readings differ by no more than 0.5 units.

NOTE 7—**Caution:** The spindle extension link should not be in contact with the insulating cap when rotating. The rotating spindle must not be 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 thermo-container using the extracting tool and discard the sample in an appropriate manner. With the sample chamber removed, cool the thermo-container by inserting the cooling plug into the sample chamber well and circulating a cooling medium (tap water) through it. Clean the stainless steel 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 consecutive 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 manual or Table A1.2 in the Annex A1. Repeat this for each temperature.

NOTE 8—If desired, the viscosity at intermediate temperatures may be calculated by fitting 1/T versus log viscosity to a straight line or by use of curve fitting software.

11. Report

11.1 Report the apparent melt viscosity at a given temperature along with the particulars of the instrument model, the spindle number, and rotational speed.

NOTE 9—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.

12. Precision and Bias

12.1 *Precision*—An interlaboratory study of the viscosity of a standard oil at one temperature and the melt viscosity of three resins each at 2 different temperatures was run in 1997 by 10 laboratories. The precision, characterized by repeatability, *Sr, r,* and reproducibility, *SR* and *R*, as specified in Practice E 691, is shown in Table 3.

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

12.2 *Bias*—Since there is no accepted reference material, method or laboratory suitable for determining the bias for the procedure in this test method for measuring softening point, no statement on bias is being made.

13. Keywords

13.1 Brookfield viscometer; rotational viscometer; apparent viscosity; viscosity; thermosel

ANNEX

(Mandatory Information)

A1. VISCOMETER RANGE DATA AND VISCOMETER-SPINDLE FACTORS

A1.1 Table A1.1 and Table A1.2 illustrate viscometer range data and viscometer-spindle factors.

NOTE A1.1—For each range, the first number represents the minimum *recommended* measurable viscosity (10 % of the lowest full scale of each viscometer).

TABLE A1.1 Viscometer Range Data

LV Series Viscometers

Spindle	Viscosity Range (cP) LVT	LVF	5XLVT	Shear Rate (sec^{-1})	Sample Volume (ml)
SC4-18	5-10 000	5-500	25-50 000	1.32N[A]	8.0
SC4-31	5-100 000	50-5 000	250-250 000	0.34N	10.0
SC4-34	100-200 000	100-10 000	500-1 000 000	0.28N	9.5

RV Series Viscometers

Spindle	Viscosity Range (cP) RVT	RVF	RVF-100	Shear Rate (sec^{-1})	Sample Volume (ml)
SC4-21	50-1000 000	250-25 000	50-5 000	0.93N	8.0
SC4-27	250-500 000	1 250-125 000	250-25 000	0.34N	10.5
SC4-28	500-1 000 000	2 500-250 000	500-50 000	0.28N	11.5
SC4-29	1 000-2 000 000	5 000-500 000	1 000-100 000	0.25N	13.0

HA Series Viscometers

Spindle	Viscosity Range (cP) HAT	HAF	Shear Rate (sec^{-1})	Sample Volume (ml)
SC4-21	10-200 000	1 000-100 000	0.93N	8.0
SC4-27	500-1 000 000	5 000-500 000	0.34N	10.5
SC4-28	1 000-2 000 000	10 000-1 000 000	0.28N	11.5
SC4-29	2 000-4 000 000	20 000-2 000 000	0.25N	13.0

HB Series Viscometers

Spindle	Viscosity Range (cP) HBT	HBF	Shear Rate (sec^{-1})	Sample Volume (ml)
SC4-21	400-800 000	3 000-400 000	0.93N	8.0
SC4-27	2 000-4 000 000	200 000-2 000 000	0.34N	10.5
SC4-28	4 000-8 000 000	40 000-4 000 000	0.28N	11.5
SC4-29	8 000-16 000 000	80 000-8 000 000	0.25N	13.0

[A] N = RPM

TABLE A1.2 Viscometer-Spindle Factors

Speed (RPM)	LVF and LVT Viscometer Spindle Number 18	31	34	5XLVF and 5XLVT Viscometer Spindle Number 18	31	34
60	0.5	5	10	2.5	25	50
30	1	10	20	5	50	100
12	2.5	25	50	12.5	125	250
6	5	50	100	25	250	500
3	10	100	200	50	500	1M[A]
1.5	20	200	400	100	1M	2M
0.6	50	500	1M	250	2.5M	5M
0.3	100	1M	2M	500	5M	10M

Speed (RPM)	RVF and RVT Viscometer Spindle Number 21	27	28	29
100	5	25	50	100
50	10	50	100	200
20	25	125	250	500
10	50	250	500	1M
5	100	500	1M	2M
4	125	625	1.25M	2.5M
2.5	200	1M	2M	4M
2	250	1.25M	2.5M	5M
1	500	2.5M	5M	10M
0.5	1M	5M	10M	20M

Speed (RPM)	HAT Viscometer Spindle Number 21	27	28	29
100	10	50	100	200
50	20	100	200	400
20	50	250	500	1M
10	100	500	1M	2M
5	200	1M	2M	4M
2.5	400	2M	4M	8M
2	500	2.5M	5M	10M
1	1M	5M	10M	20M
0.5	2M	10M	20M	40M

Speed (RPM)	HBT Viscometer Spindle Number 21	27	28	29
100	40	200	400	800
50	80	400	800	1.6M
20	200	1M	2M	4M
10	400	2M	4M	8M
5	800	4M	8M	16M
2.5	1.6M	8M	16M	32M
2	2M	10M	20M	40M
1	4M	20M	40M	80M
0.5	8M	40M	80M	160M

[A] M = 1000 To calculate viscosity in centipoise (cP) multiply the dial reading by the factor corresponding to the viscometer spindle and speed combination utilized.