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Standard Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield type) Viscometer[1]

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This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover the determination of the apparent viscosity and the shear thinning and thixotropic properties of non-Newtonian materials in the shear rate range from 0.1 to 50 s^{-1}.

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 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]

E 1 Specification for ASTM Liquid-in-Glass Thermometers

3. Summary of Test Method

3.1 Test Method A consists of determining the apparent viscosity of coatings and related materials by measuring the torque on a spindle rotating at a constant speed in the material.

3.2 Test Methods B and C consist of determining the shear thinning and thixotropic (time-dependent) rheological properties of the materials.[3] The viscosities of these materials are determined at a series of prescribed speeds of a rotational-type viscometer. The agitation of the material immediately preceding the viscosity measurements is carefully controlled.

4. Significance and Use

4.1 Test Method A is used for determining the apparent viscosity at a given rotational speed, although viscosities at two or more speeds give better characterization of a non-Newtonian material than does a single viscosity measurement.

4.2 With Test Methods B and C, the extent of shear thinning is indicated by the drop in viscosity with increasing viscometer speed. The degree of thixotropy is indicated by comparison of viscosities at increasing and decreasing viscometer speeds (Test Method B), viscosity recovery (Test Method B), or viscosities before and after high shear (combination of Test Methods B and C). The high-shear treatment in Test Method C approximates shearing during paint application. The viscosity behavior measured after high shear is indicative of the characteristics of the paint soon after application.

5. Apparatus

5.1 Rotational-type viscometers having at least four speeds, such as:

5.1.1 *Brookfield Dial-Reading (Analog) Viscometer,* or equivalent having multiple rotational speeds with set of spindles; or

5.1.2 *Brookfield Digital Viscometer*, or equivalent having multiple rotational speeds, with set of spindles.

5.2 *Thermometer*—ASTM thermometer having a range from 20 to 70°C and conforming to the requirements for Thermometer 49C as prescribed in Specification E 1.

5.3 *Containers*, round 0.5-L (1-pt) can, 85 mm (3⅜ in.) in diameter, or 1-L (1-qt) can, 100 mm (4 in.) in diameter.

5.4 *Shaker,* or equivalent machine capable of vigorously shaking the test specimen.

6. Materials

6.1 *Standard Oils,* calibrated in absolute viscosity, millipascal seconds.

7. Calibration of Apparatus

7.1 Select at least two standard oils of viscosities differing by at least 0.5 Pa·s (5P) within the viscosity range of the material being measured and in the range of the viscometer. Condition the oils as closely as possible to 25.0°C (or other

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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.

[3] Pierce, P. E., "Measurement of Rheology of Thixotropic Organic Coatings and Resins with the Brookfield Viscometer," *Journal of Paint Technology*, Vol 43, No. 557, 1971, pp. 35–43.

agreed-upon temperature) for 1 h in a 0.5-L (1-pt) can, 85 mm (3⅜ in.) in diameter. Measure the viscosities of each oil as described in Test Method B (Section 13) taking readings only at increasing speeds (13.4). Make certain that the spindle is centered in the container prior to taking measurements.

NOTE 1—The Brookfield LV and RV series viscometers are equipped with a spindle guard leg. The spindle/speed multiplying factors (Table 1) are designed for use with the guard leg in place except for the following conditions: RV series when the factors are the same with or without the guard leg for spindles No. 3 through 7; or LV series when the factors are the same with or without the guard leg for spindles No. 3 and 4.

7.1.1 Calibration in a 0.5-L (1-pt) can is always possible with the LV series viscometer with the guard leg attached. Calibration of the RV series viscometer in the 1-pt can must be done with spindles No. 3 through 7 without the guard leg. If the No. 1 or No. 2 spindles are to be used, calibration is carried out in the 1-L (1-qt) can with the guard leg attached.

7.2 Combining the tolerance of the viscometer (±1 %, equal to the spindle/speed factor) and the tolerance of the temperature control (typically ±0.5°C at 25°C) it is reasonable to assume that a viscometer is calibrated if the calculated viscosities are within ±5 % of the stated values (see Table 2 for examples of the considerable change in viscosity with temperature exhibited by standard oils). If measurements are not made at 25°C, then the stated viscosities should be corrected to the temperature at which they are measured. If the viscosities determined in 7.1 differ from the stated values of the viscosity standard by more than 5 %, calculate new factors for each spindle/speed combination as follows:

$$f = V/s \tag{1}$$

TABLE 1 Factors for Converting Brookfield Dial Readings to Millipascal Seconds (Centipoises)

NOTE 1—M = 1000.

Speed, rpm	RV Series Factors Spindles						
	1	2	3	4	5	6	7
0.5	200	800	2000	4000	8000	20M	80M
1	100	400	1000	2000	4000	10M	40M
2	50	200	500	1000	2000	5M	20M
2.5	40	160	400	800	1600	4M	16M
4	25	100	250	500	1000	2.5M	10M
5	20	80	200	400	800	2M	8M
10	10	40	100	200	400	1M	4M
20	5	20	50	100	200	500	2M
50	2	8	20	40	80	200	800
100	1	4	10	20	40	100	400

Speed, rpm	LV Series Factors Spindles			
	1	2	3	4
0.3	200	1000	4000	20M
0.6	100	500	2000	10M
1.5	40	200	800	4M
3.0	20	100	400	2M
6	10	50	200	1M
12	5	25	100	500
30	2	10	40	200
60	1	5	20	100

TABLE 2 Viscosity Variation of Cannon Viscosity Standards About the 25°C Temperature Point

Cannon Viscosity Standard	Viscosity at 25°C, mPa·s (cP)	Viscosity Change With + 1°C at 25°C, mPa·s (cP)
S-600	1 400	87.7 (6.26 %)
S-2000	4 900	332 (6.77 %)
S-8000	20 000	1462.3 (7.31 %)

where:

f = new factor for converting scale reading to viscosity, mPa·s (cP),
V = viscosity of standard oil, mPa·s, and
s = scale reading of the viscometer.

7.3 Prepare a table of new factors similar to that furnished with the viscometer (Table 1) for the spindle/speed combinations worked out in 7.2. Spindle/speed factors vary inversely with speed.

8. Preparation of Specimen

8.1 Fill a 1-pt or 1-qt can with sample to within 25 mm (1 in.) of the top with the sample and bring it as close as possible to a temperature of 25°C or other agreed-upon temperature prior to test.

8.2 Vigorously shake the specimen on the shaker or equivalent for 10 min, remove it from the shaker, and allow it to stand undisturbed for 60 min at 25°C prior to testing (Note 2). Start the test no later than 65 min after removing the can from the shaker. Do not transfer the specimen from the container in which it was shaken.

NOTE 2—Shake time may be reduced if necessary, or as agreed upon between the purchaser and manufacturer, but, in any case, should not be less than 3 min.

TEST METHOD A—APPARENT VISCOSITY

9. Procedure

9.1 Make all measurements as close as possible to 25°C, or other agreed-upon temperature.

9.2 Place the instrument on the adjustable stand. Lower the viscometer to a level that will immerse the spindle to the proper depth. Level the instrument using the attached spirit level.

9.3 Tilt the selected spindle (Note 3), insert it into one side of the center of the surface of the material, and attach the spindle to the instrument as follows: Firmly hold the upper shaft coupling with thumb and forefinger; screw left-hand thread spindle coupling securely to the upper shaft coupling being very careful when connecting to avoid undue side pressure which might affect alignment. Avoid rotating the dial so that pointer touches the stops at either extreme of the scale.

NOTE 3—Select the spindle/speed combination that will give a minimum scale reading of 10 but preferably in the middle or upper portion of the scale. The speed and spindle to be used may differ from this by agreement between user and producer.

9.4 Lower the viscometer until the groove (immersion mark) on the shaft just touches the material. Adjust the viscometer level if necessary. Move the container slowly in a horizontal plane until the spindle is located in approximately the center of the container so that the test will be run in a region undisturbed by the lowering of the spindle.

9.5 *Dial-Reading Viscometer*—Turn on the viscometer. Adjust the viscometer to the rpm selected (Note 3) for the material under test. Allow the viscometer to run until the pointer has stabilized (Note 4). After the pointer has stabilized, depress the clutch and switch off the motor so that when it stops, the pointer will be in view (Note 5).

9.6 *Digital Viscometer*—Turn on the viscometer. Adjust the viscometer to the rpm selected for the material under test. Allow the viscometer to run until the digital reading has stabilized. The digital viscometer gives a direct reading in centipoises, mPa.

NOTE 4—In thixotropic paints, the pointer or digital reading does not always stabilize. On occasion it reaches a peak and then gradually declines as the structure is broken down. In these cases, the time of rotation or number of revolutions prior to reading the viscometer should be agreed to between user and manufacturer.

NOTE 5—Always release the clutch while the spindle is still immersed so that the pointer will float, rather than snap back to zero.

10. Calculation (Dial Reading Viscometer)

10.1 Calculate the apparent viscosity at each speed, as follows:

$$V = fs \quad (2)$$

where:

V = viscosity of sample in centipoises, mPa·s,
f = scale factor furnished with instrument (see Table 1), and
s = scale reading of viscometer.

11. Report

11.1 Report the following information:

11.1.1 The viscometer manufacturer, model and spindle,

11.1.2 The viscosity at the spindle/speed utilized,

11.1.3 The specimen temperature in degrees Celsius, and

11.1.4 The shake time and rest period, if other than specified.

12. Precision and Bias

12.1 *Precision*—See Section 23 for precision, including that for measurement at a single speed.

12.2 *Bias*—No statement of bias is possible with this test method.

TEST METHOD B—VISCOSITY UNDER CHANGING SPEED CONDITIONS, DEGREE OF SHEAR THINNING AND THIXOTROPY

13. Procedure

13.1 Make all measurements with the viscometer as close as possible to 25°C, or other agreed upon temperature.

13.2 Adjust the instrument and attach the spindle as in 9.2-9.4.

13.3 Set the viscometer at the slowest rotational speed (Note 5 and Note 6). Start the viscometer and record the scale reading after ten revolutions (or other agreed-upon number of revolutions).

NOTE 6—A higher initial rotational speed may be used upon agreement between producer and user.

13.4 Increase the viscometer speed step-wise and record the scale reading after ten revolutions (or equivalent time for each spindle/speed combination) at each speed. After an observation has been made at the top speed, decrease the speed in steps to the slowest speed, recording the scale reading after ten revolutions (or equivalent time) at each speed.

NOTE 7—It is preferable to change speed when the motor is running.

13.5 After the last reading has been taken at the slowest speed, shut off the viscometer and allow it and the specimen to stand undisturbed for an agreed-upon rest period. At the end of the rest period, start the viscometer at the slowest speed and record the scale reading after ten revolutions (or other agreed-upon number of revolutions).

14. Calculations and Interpretation of Results

14.1 Calculate the apparent viscosity at each speed as shown in Section 9.

14.2 If desired, determine the degree of shear thinning by the following method:

14.2.1 *Shear Thinning Index (sometimes erroneously called the thixotropic index)*—Divide the apparent viscosity at a low rotational speed by the viscosity at a speed ten times higher. Typical speed combinations are 2 and 20 rpm, 5 and 50 rpm, 6 and 60 rpm but selection is subject to agreement between producer and user. The resultant viscosity ratio is an index of the degree of shear thinning over that range of rotational speed with higher ratios indicating greater shear thinning.

14.2.2 A regular or log-log plot of viscosity versus viscometer speed in rpm may also be useful in characterizing the shear-thinning behavior of the material. Such plots may be used for making comparisons between paints or other materials.

14.3 If desired, estimate the degree of thixotropy (under conditions of *limited* shearing-out of structure) by one of the following methods:

14.3.1 Calculate the ratio of the slowest speed viscosity taken with increasing speed to that with decreasing speed. The higher the ratio, the greater the thixotropy.

14.3.2 Calculate the ratio of the slowest speed viscosity taken after the rest period to that before the rest period. The higher the ratio, the greater the thixotropy.

15. Report

15.1 Report the following information:

15.1.1 The viscometer manufacturer, model and spindle,

15.1.2 The viscosities at increasing and decreasing spindle speeds,

15.1.3 The rest period time and the viscosity at the end of that time,

15.1.4 The specimen temperature in degrees Celsius, and

15.1.5 The shake time if other than that specified.

15.2 *Optional Reporting*:

15.2.1 *Degree of Shear Thinning*—Shear thinning index and speeds over which it was measured (14.2).

15.2.2 *Estimated Degree of Thixotropy (under conditions of shearing-out of structure)*—Ratio of the lowest speed viscosities, for both increasing and decreasing speeds; or ratio of the lowest speed viscosities before and after the rest period, and speed at which they were measured (14.3).

16. Precision and Bias

16.1 *Precision*—See Section 23 for precision, including that for measurement of the shear thinning index (ratio of viscosity at 5 r/min to that at 50 r/min). It has not been possible to devise a method for determining precision for viscosities at increasing and decreasing speeds other than as individual measurements. No attempt was made to determine the precision of the measurement of the degree of thixotropy because this parameter is dependent on the material, the time of the test, and other variables.

16.2 *Bias*—No statement of bias is possible with this test method.

TEST METHOD C—VISCOSITY AND SHEAR THINNING OF A SHEARED MATERIAL

17. Apparatus

17.1 High-speed laboratory stirrer with speeds of at least 2000 rpm and equipped with a 50-mm (2-in.) diameter circular dispersion blade.[4]

18. Preparation of Specimen

18.1 Insert the 50-mm (2-in.) blade into the center of the can (8.1) so that the blade is about 25 mm (1 in.) from the bottom. Run the mixer at 2000 rpm (Note 8) for 1 min.

NOTE 8—Materials may be sheared at other speeds using other size blades upon agreement between producer and user.

19. Procedure

19.1 Immediately insert the same spindle used in Test Method B into the sheared material in the same manner as in Section 9.

19.2 Start the viscometer and adjust to the highest speed used in Test Method B (13.5). Record the scale reading after ten revolutions (or other agreed-upon number of revolutions).

19.3 Decrease the viscometer speed (Note 7) step-wise and record the scale readings at each speed down to the lowest speed used in Test Method B, recording the scale reading after ten revolutions at each speed (or other agreed-upon number of revolutions).

20. Calculations and Interpretation of Results

20.1 As in Test Method B, calculate the viscosities at each decreasing speed.

20.2 If desired, calculate the degree of shear thinning by the method given in Test Method B, 14.2. The measured viscosity behavior after shearing is essentially that of the paint immediately after application (disregarding changes in solids).

20.3 If desired, estimate the degree of thixotropy (under conditions of *complete* shearing-out of structure) by calculating the ratio of the lowest speed viscosities before and after shear. The lowest speed before-shear viscosity is taken from Test Method B, 14.1, at the lowest increasing speed. The lowest speed after-shear viscosity is taken from 20.1. The higher the ratio, the greater the thixotropy.

21. Report

21.1 Report the following information:

21.1.1 The viscometer manufacturer, model and spindle,

21.1.2 The viscosities at decreasing spindle speeds,

21.1.3 The specimen temperature in degrees Celsius, and

21.1.4 The speed of the high-speed mixer, size of blade, and time of mixing if different from method.

21.2 *Optional Reporting*:

21.2.1 *Degree of Shear Thinning—Shear thinning index and speed over which it was measured (*14.2*).*

21.2.2 *Estimated Thixotropy*—Ratio of lowest speed viscosities before and after shear and the speed at which they were measured.

22. Precision and Bias

22.1 *Precision*—The precision for individual viscosity measurements is the same as for Test Method A in Section 23. No attempt has been made to determine the precision of the shear thinning index or degree of thixotropy for Test Method C for the reasons given in 16.1.

22.2 *Bias*—No statement of bias is possible with this test method.

23. Summary of Precision

23.1 In an interlaboratory study of Test Methods A and B, eight operators in six laboratories measured on two days the viscosities of four architectural paints comprising a latex flat, a latex semi-gloss, a water-reducible gloss enamel, and an alkyd semi-gloss, that covered a reasonable range in viscosities and were shear thinning. Measurements at increasing speeds of 5, 10, 20, and 50 r/min (equivalent to eight operators testing 16 samples) were used to obtain the precision of Test Method A. The within-laboratory coefficient of variation for Test Method A (single speed) was found to be 2.49 % with 121 df and for Test Method B (Shear Thinning Index) 3.3 % with 31 df. The corresponding between-laboratories coefficients are 7.68 % with 105 df and 7.63 % with 27 df. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

23.1.1 *Repeatability*—Two results obtained by the same operator at different times should be considered suspect if they differ by more than 7 % relative for single speed viscosity and 9.5 % relative for shear thinning index.

23.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 21.6 and 22.1 % relative, respectively, for the same two test methods.

[4] Cowles or Shar type mixer/disperser.

24. Keywords

24.1 Brookfield viscometer; non-Newtonian; rheological properties; rheology; rotational; shear thinning; thixotropic; thixotropy; viscometer; viscosity