Designation: D2196 – 15

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

This standard is issued under the fixed designation D2196; 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.

This standard has been approved for use by agencies of the U.S. 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} using a rotational viscometer operating in a fluid of "infinite" dimensions.

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. Summary of Test Method

2.1 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.

2.2 Test Methods B and C consist of determining the shear thinning and thixotropic (time-dependent) rheological properties of the materials.[2] The viscosities of these materials are determined at a series of prescribed speeds of a rotational-type viscometer operating in a fluid of "infinite" dimensions. The agitation of the material immediately preceding the viscosity measurements is carefully controlled.

3. Significance and Use

3.1 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.

3.2 With Test Methods B and C, the extent of shear thinning is indicated by the drop in viscosity with increasing rotational speed. The degree of thixotropy is indicated by comparison of viscosities at increasing and decreasing rotational 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.

4. Apparatus

4.1 *Rotational Viscometer*—The essential instrumentation required providing the minimum rotational viscometer analytical capabilities for this method include:

4.1.1 A *drive motor*, to apply a unidirectional rotational displacement to the specimen at at least for rotational speeds between 0.05 and 6 rad/s (0.5 and 60 r/min) constant to within 1 %.

4.1.2 A *force sensor* to measure the torque developed by the specimen to the rotational displacement of the rotational element to within 1 %.

4.1.3 A *coupling shaft*, or other means, to transmit the rotational displacement from the motor to the rotational element.

4.1.4 A *rotational element, spindle, or tool*, such as the cylindrical shape shown in Fig. 1, to fix the specimen between the drive shaft and a stationary position.

NOTE 1—Each rotational element covers a range of about 1.5 decades of viscosity. The rotational element is selected so that the measured viscosity (or torque) is between 10 and 90 % of the range of the rotational element.

4.1.5 A *data collection device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for rotational viscosity are torque, rotational speed, temperature, and time.

NOTE 2—Manual observation and recording of data are acceptable.

[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints & Paint Materials.

Current edition approved Feb. 15, 2015. Published April 2015. Originally approved in 1963. Last previous edition approved in 2010 as D2196 – 10. DOI: 10.1520/D2196-15.

[2] 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.

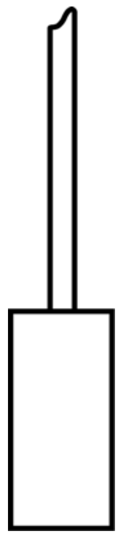

FIG. 1 Cylindrical Rotational Element Configuration

4.1.6 A *stand*, to support, level, and adjust the height of the drive motor, shaft and rotational element.

4.1.7 A *level* to indicate the vertical plumb of the drive motor, shaft and rotational element.

4.1.8 Auxiliary instrumentation considered useful in conducting this method includes:

4.1.8.1 *Data analysis capability* to provide viscosity, stress or other useful parameters derived from the measured signals.

4.2 A *temperature measuring and recording device* to provide specimen temperature of the fluid near the rotational element over the range of 20 to 70°C to within 0.1°C (see Note 2).

4.3 A cylindrical *container* with a capacity of 0.5-L (1-pt), 85 mm (3⅜ in.) in diameter, or 1-L (1-qt), 100 mm (4 in.) in diameter to contain the test specimen during testing.

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

5. Materials

5.1 *Viscosity Reference Oils,* calibrated in absolute viscosity, milliPascal seconds.

6. Calibration of Apparatus

6.1 Select at least two reference 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 to 25.0°C ±0.5°C (or other agreed-upon temperature) for 1 h in a 0.5-L (1-pt) container. Measure the viscosities of each oil as described in Test Method B (Section 12) taking readings only at increasing speeds (12.4).

NOTE 3—Ensure that the spindle is centered in the container prior to taking measurements.

NOTE 4—Combining the tolerance of the viscometer (±1 %, equal to the spindle/speed combination 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.

NOTE 5—Reference oils can exhibit a change in viscosity of about 7 %/°C. If measurements are not made at 25°C, then the stated viscosities shall be corrected to the temperature at which they are measured.

6.2 If the viscosities determined in 6.1 differ from the stated values of the viscosity standard by more than 5 %, calculate new calibration factors for each spindle/speed combination as follows:

$$f = V / s \quad (1)$$

where:

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

6.3 Prepare a table of new calibration factors similar to that furnished with the viscometer for the spindle/speed combinations in 6.2. Spindle/speed factors vary inversely with speed.

7. Preparation of Specimen

7.1 Fill a 0.5-L (1-pt) or 1-L (1-qt) container with sample to within 25 mm (1 in.) of the top with the sample and bring it to a temperature of 25°C ± 0.5°C or other agreed-upon temperature prior to test.

7.2 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. Start the test no later than 65 min after removing the container from the shaker. Do not transfer the specimen from the container in which it was shaken. Shake time may be reduced if necessary, or as agreed upon between the purchaser and manufacturer, but, in any case, shall not be less than 3 min.

NOTE 6—Shake time may be reduced if necessary, if agreed upon between the purchaser and manufacturer, but, in any case, shall not be less than 3 min.

TEST METHOD A—APPARENT VISCOSITY

8. Procedure

8.1 Make all measurements at 25 ± 0.5°C, or other agreed-upon temperature.

8.2 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.

8.3 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.

NOTE 7—When connecting the spindle to the viscometer avoid undue side pressure which might affect alignment. Avoid rotating the spindle so that the viscometer indicator touches the stops at either extreme of the scale.

NOTE 8—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.

8.4 Lower the viscometer until the immersion mark on the shaft just touches the specimen. Adjust the viscometer level if necessary. Move the container slowly in a horizontal plane until the spindle is located in the approximate center of the container.

8.5 Initiate the rotation of the spindle. Adjust the rotational speed so that the torque reads between 10 and 90 % of full. Allow the viscometer to run until reading stabilizes. Record the torque or viscosity reading.

NOTE 9—In thixotropic paints, the 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.

9. Calculation (Dial Reading Viscometer)

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

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

where:

V = viscosity of sample in mPa·s,
f = calibration factor furnished with instrument or determined in Section 6, and
s = reading of viscometer.

10. Report

10.1 Report the following information:

10.1.1 The viscometer manufacturer, model and spindle,

10.1.2 The viscosity at the spindle and speed utilized,

10.1.3 The specimen temperature in degrees Celsius, and

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

11. Precision and Bias

11.1 *Precision*—See Section 22 for precision, including that for measurement at a single speed.

11.2 *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

12. Procedure

12.1 Make all viscosity (or torque) measurements at 25 ± 5°C, or other agreed upon temperature.

12.2 Adjust the instrument and attach the spindle as in 8.2 – 8.4.

12.3 Set the viscometer at the slowest rotational speed (Note 9). Initiate the spindle rotation and record the reading after ten revolutions (or other agreed-upon number of revolutions).

NOTE 10—A higher initial rotational speed may be used upon agreement between producer and user.

12.4 Increase the rotational speed in steps and record the 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 rotational speed in steps to the slowest speed, recording the reading after ten revolutions (or equivalent time) at each speed.

NOTE 11—It is preferable to change speed when the motor is running.

12.5 After the last reading has been taken at the slowest speed, stop the rotation and allow the specimen to stand undisturbed for an agreed-upon rest period typically 1 minute. At the end of the rest period, start the spindle rotation at the slowest speed and record the reading after ten revolutions (or other agreed-upon number of revolutions).

13. Calculations and Interpretation of Results

13.1 Calculate the apparent viscosity at each speed as shown in Section 8.

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

13.2.1 *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 0.2 and 2 rad/s (2 and 20 r/min), 0.5 and 5 rad/s (–5 and 50 r/min), 0.6 and 6 rad/s (–6 and 60 r/min) 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.

13.2.2 A regular or log-log plot of viscosity versus rotational speed 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.

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

13.3.1 Calculate the ratio of the viscosity at the slowest rotational speed with increasing speed to that with decreasing speed. The higher the ratio, the greater the thixotropy.

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

14. Report

14.1 Report the following information:

14.1.1 The viscometer manufacturer, model and spindle,

14.1.2 The viscosities at increasing and decreasing spindle speeds,

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

14.1.4 The specimen temperature in degrees Celsius, and

14.1.5 The shake time if other than that specified.

14.2 *Optional Reporting:*

14.2.1 *Degree of Shear Thinning*—Shear thinning index and speeds over which it was measured (13.2).

14.2.2 *Estimated Degree of Thixotropy* (under conditions of shearing-out of structure)—Ratio of the viscosities at the lowest speed, for both increasing and decreasing speeds; or ratio of the viscosity at the lowest speeds before and after the rest period, and speed at which they were measured (13.3).

15. Precision and Bias

15.1 *Precision*—See Section 22 for precision, including that for measurement of the shear thinning index (ratio of viscosity at 0.5 rad/s to that at 5 rad/s (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.

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

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

16. Apparatus

16.1 High-speed laboratory stirrer with speeds of at least 200 rad/s (2000 r/min) and equipped with a 50-mm (2-in.) diameter circular dispersion blade.[3]

17. Preparation of Specimen

17.1 Insert the 50-mm (2-in.) blade into the center of the container (7.1) so that the blade is about 25 mm (1 in.) from the bottom. Run the mixer at 200 rad/s (2000 r/min) (Note 12) for 1 min.

NOTE 12—Materials may be sheared at other speeds using other size blades upon agreement between producer and user.

18. Procedure

18.1 Immediately insert the same spindle used in Test Method B into the sheared material in the same manner as in Section 8.

18.2 Initiate the spindle rotation at the highest speed used in Test Method B (12.5). Record the scale reading after ten revolutions (or other agreed-upon number of revolutions).

18.3 Decrease the rotational speed (Note 11) in steps and record the readings at each speed down to the lowest speed used in Test Method B, recording the reading after ten revolutions at each speed (or other agreed-upon number of revolutions).

19. Calculations and Interpretation of Results

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

19.2 If desired, calculate the degree of shear thinning by the method given in Test Method B, 13.2. The measured viscosity behavior after shearing is essentially that of the paint immediately after application (disregarding changes in solids).

19.3 If desired, estimate the degree of thixotropy (under conditions of *complete* shearing-out of structure) by calculating the ratio of the viscosities at the lowest speeds before and after shear. The viscosity at the lowest speed before-shearing is taken from Test Method B, 13.1, at the lowest increasing speed. The viscosity at lowest speed after-shear is taken from 19.1. The higher the ratio, the greater the thixotropy.

[3] Cowles or Shar type mixer/disperser.

20. Report

20.1 Report the following information:

20.1.1 The viscometer manufacturer, model and spindle,

20.1.2 The viscosities at decreasing spindle speeds,

20.1.3 The specimen temperature in degrees Celsius, and

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

20.2 *Optional Reporting:*

20.2.1 *Degree of Shear Thinning*—Shear thinning index and speed over which it was measured (13.2).

20.2.2 *Estimated Thixotropy*—Ratio of viscosities at lowest speed viscosities before and after shearing and the rotational speed at which they were measured.

21. Precision and Bias

21.1 *Precision*—The precision for individual viscosity measurements is the same as for Test Method A in Section 22. 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 15.1.

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

22. Summary of Precision

22.1 In an interlaboratory study of Test Methods A and B, eight operators in six laboratories using dial reading apparatus from a single supplier (Brookfield Engineering) 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 0.5, 1.0, 2.0, and 5.0 rad/s (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 2.5 % with 121 df and for Test Method B (Shear Thinning Index) 3.3 % with 31 df. The corresponding between-laboratories coefficients are 7.7 % with 105 df and 7.6 % with 27 df. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

22.1.1 *Repeatability*—Two results obtained by the same operator at different times should be considered suspect if they differ by more than 7.0 % relative for single speed viscosity and 9.5 % relative for shear thinning index.

22.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 22 % relative, respectively, for the same two test methods.

NOTE 13—Measurements made by digital apparatus from the same supplier or apparatus from other suppliers may have different precision

23. Keywords

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