Designation: D2196 – 20

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 s^{-1} to 50 s^{-1} using a rotational viscometer operating in a fluid contained in a 600 mL low form Griffin beaker.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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 a 600 mL low form Griffin beaker.

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 viscometer with a spindle operating in a fluid contained in a 600 mL low form Griffin beaker. 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 instrument will have the following capabilities at a minimum:

4.1.1 A *drive motor*, to apply a unidirectional rotational displacement to the spindle immersed in the specimen for rotational speeds between 0.307 rad/sec and 10.24 rad/sec (0.3 r/min and 100 r/min) constant to within 0.1 %.

4.1.2 A *force sensor* to measure the torque required to drive the spindle immersed in the specimen at each of the defined speed settings to within 0.1 %.

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

4.1.4 A *rotational element, spindle, or tool*, such as the shapes shown in Fig. 1 to fix the specimen between the spindle and a stationary surface. The protective bracket, which attaches to the viscometer and protects the spindle, provides the stationary surface described in the preceding sentence.

NOTE 1—Each spindle can measure a range of almost four decades in viscosity for the speed settings specified in this method. The spindle is selected so that the measured torque value is between 10 % and 100 %.

[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 Dec. 1, 2020. Published December 2020. Originally approved in 1963. Last previous edition approved in 2018 as D2196 – $18^{\varepsilon 1}$. DOI: 10.1520/D2196-20.

[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.

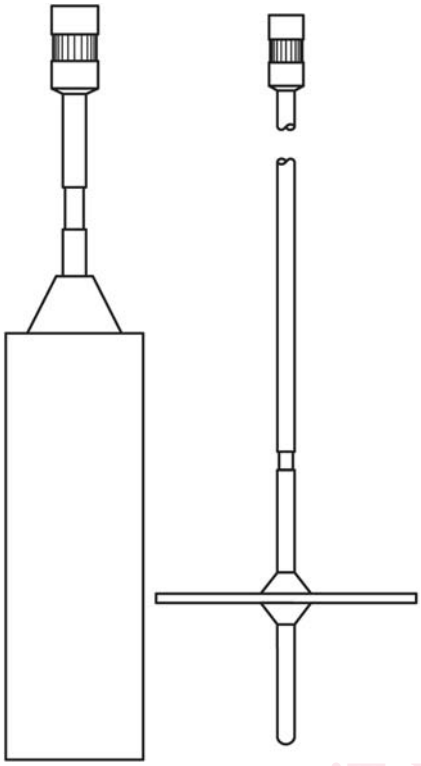

FIG. 1 Cylindrical and Disc Rotational Element Configuration

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 a viscosity measurement are rotational speed of the spindle and torque. Best practice is to record output signals for time of spindle rotation when making the viscosity measurement and the temperature of the specimen.

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

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°C to 70°C to within 0.1°C (see Note 2).

4.3 A 600 mL low form Griffin beaker or equivalent cylindrical container with minimum volume capacity of 500 mL, minimum diameter of 85 mm (3.35 in.), and minimum depth of 100 mm (3.94 in.) to contain the 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 scientific units of either Pascal-seconds, milliPascal-seconds, Poise, or centipoises.

6. Calibration Verification of Apparatus

6.1 Select one viscosity reference oil within the viscosity range of the material being measured. Condition the oil to 25.0°C ± 0.1°C (or other agreed-upon temperature) for 1 h in a 600 mL low form Griffin beaker (or equivalent container). Select an appropriate spindle, connect it to the viscometer, and attach the bracket. Immerse the spindle and bracket into the oil and allow these items to equilibrate to temperature during the 1 h period. Measure the oil viscosity at three increasing rotational speeds which give torque readings between 10 % and 100 %.

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

NOTE 4—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 Each measured viscosity value must be within the following calculation for the viscometer to pass the calibration check.

6.2.1 Determine the full scale viscosity range for the spindle/speed combination being used. Calculate 1 % of this number.

6.2.2 Calculate 1 % of the viscosity value for the reference oil.

6.2.3 Add the viscosity values obtained in the two previous calculations. Add and subtract this sum from the actual viscosity value for the reference oil. The measured viscosity value must fall between these calculated limits for the viscometer to pass the calibration check.

6.2.4 If any of the three viscosity measurements do not pass, repeat the test. If the test is still not successful, contact the instrument manufacturer for service of the rotational viscometer.

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 5—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°C ± 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 6—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 7—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 100 %. Allow the viscometer to run until reading stabilizes. Record the torque and the viscosity reading.

NOTE 8—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:

$$V = fs \quad (1)$$

where:

V = viscosity of sample in mPa·s,
f = conversion factor for spindle/speed combination furnished with instrument,
s = % torque 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°C ± 0.5°C, or other agreed upon temperature.

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

12.3 Decide upon the different rotational speeds to be used for the test; a minimum of three speeds is recommended. Set the viscometer at the slowest rotational speed chosen for the test (Note 8). Initiate the spindle rotation and record the reading after ten revolutions (or other agreed-upon number of revolutions).

NOTE 9—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.

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. If using a dial reading viscometer, calculate the equivalent viscosity value for each torque value as shown in Section 10.

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

13.2.1 *Shear Thinning Index* (sometimes called the thix 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 r/min and 2 r/min (2 r/min and 20 r/min), 0.5 r/min and 5 r/min (5 r/min and 50 r/min), 0.6 r/min and 6 r/min (6 r/min 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: