



Designation: D2196 – 20

# Standard Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational Viscometer<sup>1</sup>

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 ( $\epsilon$ ) 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  $0.1 \text{ s}^{-1}$  to  $50 \text{ s}^{-1}$  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.<sup>2</sup> 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 %.

<sup>1</sup> 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.

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<sup>2</sup> 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.

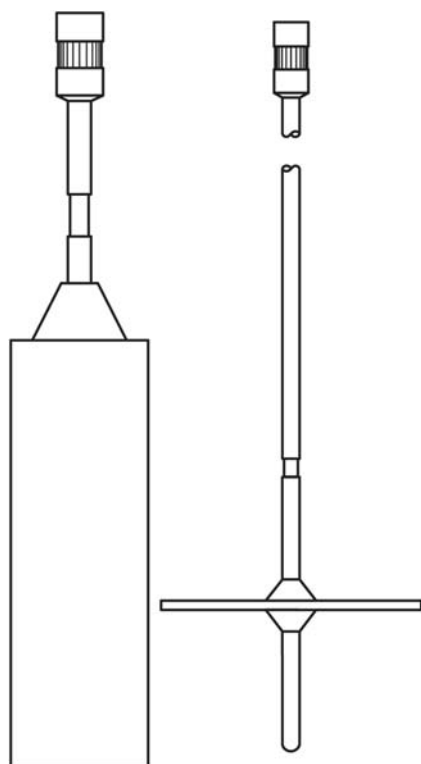


FIG. 1 Cylindrical and Disc Rotational Element Configuration

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  $25.0^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$  (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  $25^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  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.