



Designation: ~~D2196~~—~~15~~ D2196 – 18

Standard Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational Viscometer Viscountess¹

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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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 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 the material 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-type viscometer rotational viscometer with a spindle operating in a fluid of “infinite” dimensions 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 instrumentation required providing the minimum rotational viscometer analytical capabilities for this method include: instrument will have the following capabilities at a minimum:

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 June 1, 2018. Published April 2015 August 2018. Originally approved in 1963. Last previous edition approved in 2010 2015 as ~~D2196~~—~~10~~ D2196 – 15. DOI: 10.1520/D2196-15.10.1520/D2196-18.

² Pierce, P. E., “Measurement of Rheology of Thixotropic Organic Coatings and Resins with the Brookfield ~~Viscometer~~ Viscountess,” *Journal of Paint Technology*, Vol 43, No. 557, 1971, pp. 35–43.

4.1.1 A *drive motor*, to apply a unidirectional rotational displacement to the specimen at at least spindle immersed in the specimen for rotational speeds between 0.05 and 6 rad/s (0.5 and 60 r/min) 0.307 and 10.24 rad/sec (0.3 and 100 rpm) constant to within ~~±0.01%~~ 0.01%.

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% 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 rotational element spindle.

4.1.4 A *rotational element, spindle, or tool*, such as the cylindrical shape shapes shown in Fig. 1; to fix the specimen between the drive shaft spindle and a stationary position 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 rotational element coverspindle can measure a range of about 1.5 decades of viscosity. The rotational element almost four decades in viscosity for the speed settings specified in this method. The spindle is selected so that the measured viscosity (or torque) torque value is between 10 and 90% of the range of the rotational element. 10% and 100%.

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. 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 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 a minimum volume capacity of 0.5 L (1 pt), 500 mL, minimum diameter of 85 mm (3.35 in.) in diameter, or 1 L (1 qt), in., and minimum depth of 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.

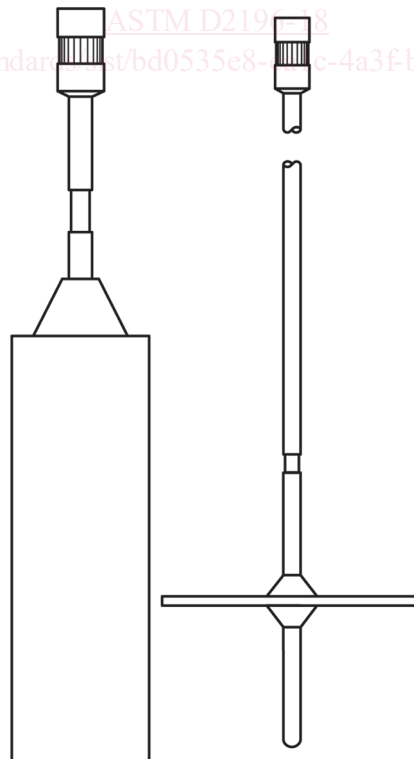


FIG. 1 Cylindrical and Disc Rotational Element Configuration

5. Materials

5.1 *Viscosity Reference Oils*, calibrated in ~~absolute viscosity, milliPascal-seconds, scientific units of either Pascal-seconds, milliPascal-seconds, Poise, or centipoises.~~

6. Calibration Verification of Apparatus

6.1 Select ~~at least two reference oils of viscosities differing by at least 0.5 Pa·s (5P)~~ one viscosity reference oil 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 measured.~~ Condition the oil to 25.0°C ± 0.1°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 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 ~~take~~ take readings only at increasing speeds (give torque readings between 10 % and 100 %. ~~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 ($\pm 1\%$, equal to the spindle/speed combination factor) and the tolerance of the temperature control (typically $\pm 0.5^\circ\text{C}$ at 25°C) it is reasonable to assume that a viscometer is calibrated if the calculated viscosities are within $\pm 5\%$ of the stated values.

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