



Designation: ~~D2196~~—~~10~~ D2196 – 15

## Standard Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield type) 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 to 50 s<sup>-1</sup>; 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. Referenced Documents

2.1 ~~ASTM Standards:~~<sup>2</sup>

~~E1 Specification for ASTM Liquid-in-Glass Thermometers~~

### 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.<sup>2</sup> The viscosities of these materials are determined at a series of prescribed speeds of a rotational-type viscometer; 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 viscometer rotational speed. The degree of thixotropy is indicated by comparison of viscosities at increasing and decreasing viscometer 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—Rotational-type viscometers having at least four speeds, such as: The essential instrumentation required providing the minimum rotational viscometer analytical capabilities for this method include:

<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.

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 Brookfield Dial-Reading (Analog) Viscometer, A coupling shaft, or equivalent having multiple rotational speeds with set of spindles; or other means, to transmit the rotational displacement from the motor to the rotational element.

4.1.4 Brookfield Digital Viscometer, A or equivalent rotational element, spindle, or tool, having multiple rotational speeds, with set of such as the cylindrical shape shown in Fig. 1 spindles; 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.

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 Thermometer—ASTMA thermometer having a range from temperature measuring and recording device 20 to 70°C and conforming to the requirements for Thermometer 49C as prescribed in Specification to provide specimen temperature of the fluid near the rotational element over the range of **E1**. In addition, temperature measuring devices such 20 to 70°C to within 0.1°C (see Note 2 as non-mercury liquid-in-glass thermometers, thermocouples, or platinum resistance thermometers that provide equivalent or better accuracy and precision, that cover the temperature range for thermometer 49C, may be used.).

4.3 Containers, round A cylindrical container 0.5-L (1-pt) can, with a capacity of 0.5-L (1-pt), 85 mm (3 3/8 in.) in diameter, or 1-L (1-qt) can, (1-qt), 100 mm (4 in.) in diameter; 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 Standard Viscosity Reference Oils, calibrated in absolute viscosity, millipascal/second.

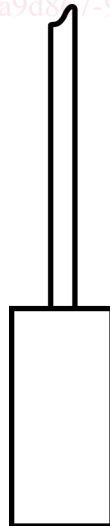


FIG. 1 Cylindrical Rotational Element Configuration

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 %) —

## 6. Calibration of Apparatus

6.1 Select at least two ~~standard 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 as closely as possible to  $25.0^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  (or other agreed-upon temperature) for 1 h in a 0.5-L (1-pt) ~~can, 85 mm (3 container, 3/4 in.) in diameter.~~ Measure the viscosities of each oil as described in Test Method B (Section ~~13.12~~) taking readings only at increasing speeds (~~13.412.4~~). ~~Make certain that the spindle is centered in the container prior to taking measurements.~~

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

NOTE 4—The Brookfield LV and RV series viscometers are equipped with a spindle guard leg. The spindle/speed multiplying factors (Combining ~~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; 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  $25^{\circ}\text{C}$ ) it is reasonable to assume that a viscometer is calibrated if the calculated viscosities are within  $\pm 5\%$  of the stated values.

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.

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