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.

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[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.

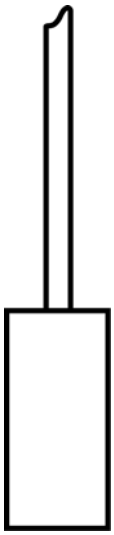

FIG. 1 Cylindrical Rotational Element Configuration

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.