



Designation: D1795 – 13 (Reapproved 2021)

Standard Test Method for Intrinsic Viscosity of Cellulose¹

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1. Scope

1.1 This test method covers the determination of the intrinsic viscosity of purified celluloses such as bleached wood pulps, cotton linters, and regenerated cellulose. It is applicable to all cellulose samples with an intrinsic viscosity of 15 dL/g or less. Most native (unpurified) celluloses have intrinsic viscosity values too high for measurement by this test method.

NOTE 1—The use of cuprammonium hydroxide solution for regular viscosity determination is described in Method T 206 m-55 of the Technical Association of Pulp and Paper Industry on “Cuprammonium Disperse Viscosity of Pulp,” and Joint Army-Navy Specifications JAN-C-206.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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. Referenced Documents

2.1 *ASTM Standards:*²

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D629 Test Methods for Quantitative Analysis of Textiles

E1 Specification for ASTM Liquid-in-Glass Thermometers

¹ This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.36 on Cellulose and Cellulose Derivatives.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

3. Summary of Test Method

3.1 A weighed sample of the material is dissolved in a 0.5 M cupriethylenediamine hydroxide solution. The viscosity of this solution, and also that of the solvent, is determined at 25°C by means of a calibrated glass capillary-type viscometer. The relative viscosity is calculated and the corresponding intrinsic viscosity is read from a table.

4. Significance and Use

4.1 This test is a sensitive measure of the degradation of cellulose resulting from the action of heat, light, acids, alkalies, oxidizing and reducing agents, and the like, used in its processing or purification. The intrinsic viscosity value may be converted to degree of polymerization (DP) or to intrinsic fluidity, if desired.

4.2 Solutions of cellulose are not Newtonian liquids; that is, their viscosity depends upon the rate-of-shear or velocity gradient during measurement. This effect is smaller for samples of low molecular mass (DP) and at low concentrations than for high-DP samples and at high concentrations. For the celluloses and concentrations included within the limits set forth in this test method, the effect of rate-of-shear is assumed to be negligible for referee purposes. For other conditions and for research purposes this assumption may be invalid, but to discuss ways of accounting for this effect is beyond the scope of the present test method.

5. Apparatus

5.1 *Viscometer, Glass, Capillary Type*—The Cannon-Fenske, Ubbelohde, or similar capillary type instrument as described in Test Method **D445** is recommended. In order to avoid correction for the kinetic energy effect, choose a viscometer with a small enough capillary to give an outflow time of 80 s or more for the Cannon-Fenske type. (A size 100 viscometer is normally used for the sample solution and a size 50 for the solvent.)

5.2 *Thermometer*—ASTM Kinematic Viscosity Thermometer for use at 25°C, having a range from 19 to 27°C and conforming to the requirement for Thermometer 17C as prescribed in Specification **E1**. Thermometric devices such as

RTDs, thermistors and liquid-in-glass thermometers (as specified in Specification E2251) of equal or better accuracy in the specified temperature range, may be used.

5.3 Bath—A constant-temperature bath at 25°C suitable for immersion of the viscometer so that the reservoir or the top of the capillary, whichever is uppermost, is immersed at least 50 mm, and with provision for visibility of the instrument and the thermometer. Firm supports for the viscometer shall be provided; or the viscometer may be sealed in as an integral part of the bath. Either a liquid bath with thermostatic regulation and a stirrer or a vapor bath with pressure regulation is permissible. The efficiency of the stirring and the balance between heat losses and heat input must be such that the temperature of the bath medium does not vary by more than $\pm 0.1^\circ\text{C}$ over the length of the viscometer, or from viscometer to viscometer in the various bath positions. If a vapor bath is used, there must be no temperature gradient over the length of the viscometer greater than that permitted in a liquid bath.

5.4 Timer—A stop watch or other spring-activated timing device or electrical timing device shall be used, graduated in divisions of 0.2 s or less, and accurate to within 0.05 % when tested over not less than a 10-min period. Such electrical timing devices shall be used only on electrical circuits of continuously controlled frequency. Frequency-controlled devices of suitable capacity for laboratory purposes, accurate to within 1 part in 10 000 should be used. Errors exceeding 0.05 % of a 10-min interval may occur in timing devices actuated by electrical synchronous motors driven by most public power systems, which are intermittently and not continuously controlled.

6. Reagent

6.1 Cupriethylenediamine Hydroxide Solution ($1.00 \pm 0.01 M$), in copper, with the molar ratio of ethylenediamine to copper of 2 ± 0.1 to 1. This solvent may be prepared in the laboratory. It is also commercially available.³

7. Reference Materials

7.1 Viscosity Oil Standards—Calibrating oils in the specified ranges of viscosity.³ Aqueous solutions of glycerol may be used instead of standardized oils; the compositions for various viscosities are given in chemical handbooks. The applicable viscosity oil standards (Note 2) are listed in Table 1.

NOTE 2—The viscosity oil standards are available only as 4.7-m³ (1-pt) samples. More than 1 pt of any given oil (for example, duplicate samples) are supplied only when it is established that 1 pt is inadequate.³

8. Calibration of Viscometer

8.1 The following directions apply to the Cannon-Fenske viscometer (Note 3). They should be modified according to the operating instructions for other types of viscometers. The viscometers shall be calibrated (Note 4) by means of liquids

TABLE 1 Viscosity Oil Standards

Viscometer		Viscosity Oil Standard	
Size	Absolute Viscosity Range, cP ^A	Designation	Approximate Absolute Viscosity at 25°C, cP
50	0.9 to 3.5	S-3	3.3
100	3.3 to 13.3	S-6	7.7

^A For solution with density of 0.9.

having known viscosities approximately equal to those of the solvent and cellulose solutions respectively (1.2 and 12 cP, approximately).

NOTE 3—Detailed specifications and directions for filling, calibrating, and measurement with types of capillary viscometers most used are given in Test Method D445.

NOTE 4—Calibration of the viscometers may be avoided if both solvent and solution are measured in the same instrument. Then the relative viscosity is nearly the ratio of outflow times for solution and solvent, respectively. This simplification involves two assumptions. The first, that the densities of solution and solvent are equal, holds very well for the dilute solutions used in these tests. The second, that the kinetic energy correction is zero, depends upon the choice of viscometer. If the one used gives convenient outflow times for the solution of less than 150 s, then it will be too fast for the solvent. The kinetic energy correction is zero, depending upon flow. On the other hand, if one is chosen so that the outflow time for the solvent is large enough (80 s or more), then the times for the solutions will in most cases be inconveniently long. For some work, however, it may be desirable to make some sacrifice in accuracy or in convenience during measurement in order to avoid calibration and using two sizes of viscometers.

8.2 By means of a pipet, add 7.0 mL of the calibrating liquid to the viscometer, in a constant-temperature bath at $25 \pm 0.1^\circ\text{C}$ (or fill as described in Test Method D445, Appendix A).

8.3 When the liquid has reached temperature equilibrium with the bath (in about 5 min), determine the outflow time t by drawing the top meniscus of the liquid above the mark between the two bulbs and measuring the time required for the meniscus to pass from this mark to the mark below the lower bulb. Take the average of two or more observations, which should differ by not more than 0.2 s.

8.4 Determine the viscometer constant C by the equation:

$$C = \eta/dt \quad (1)$$

where:

η = viscosity of the calibrating liquid, cP,
 d = density, g/mL, and
 t = time, s.

9. Preparation of Sample

9.1 To avoid undesirable effects from long heating at high temperature, samples should be air-dried and the moisture content determined on a portion that is not used for measurement of viscosity. The mass of air-dried samples is then corrected for moisture to obtain the mass of oven-dried cellulose used to calculate concentration.

9.2 Soft, sheeted pulp should be picked apart with tweezers or scraped with a dull knife. Hard-pressed or harsh pulp should be slurried in water, formed into thin sheets on a Büchner funnel, and dried at a temperature below 100°C (preferably room temperature). Loose pulp should be picked apart by hand

³ The sole source of supply of the viscosity oil standards known to the committee at this time is Cannon Instrument Co., P. O. Box 16, State College, PA 16801. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.