Designation: D6188 – 17 (Reapproved 2022)

Standard Test Method for Viscosity of Cellulose by Cuprammonium Ball Fall[1]

This standard is issued under the fixed designation D6188; 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.

1. Scope

1.1 This test method describes the procedure for estimating the molecular weight of cellulose by determining the viscosity of cuprammonium (CuAm) solutions of cellulosic materials, such as wood pulp, cotton, and cotton linters. This test method is suitable for rapid, routine testing of large numbers of samples with high accuracy and precision. This test method updates and extends the procedure reported by the American Chemical Society (ACS).[2]

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. Referenced Documents

2.1 *ASTM Standards:*[3]

D1193 Specification for Reagent Water
D1695 Terminology of Cellulose and Cellulose Derivatives
E438 Specification for Glasses in Laboratory Apparatus

3. Terminology

3.1 This standard terminology of cellulose and cellulose derivatives, see Terminology D1695.

4. Summary of Test Method

4.1 An in-process or finished product sample is taken. All cooking and bleaching chemicals must be washed out of in-process samples. Dry samples are wetted with demineralized water. Samples are either squeezed or pressed to 20 to 40 % consistency as necessary, then passed through a picker.

4.2 The wet pulp sample is dried with air whose maximum temperature is 120 °C and weighed under conditions that cause the specified quantity of sample to be obtained. The weighed sample is placed in a glass 120-mL (4-oz) bottle, steel shot are added, a vacuum is pulled on the bottle, and 97 mL of cuprammonium solution are added to the bottle. The bottle is placed on a shaker to mix and dissolve the pulp sample in the CuAm solution.

4.3 The dissolved sample is transferred to a glass viscosity tube. The tube is mounted vertically with a bright light behind the tube. A special glass bead (see 7.13) is dropped into the center of the solution in the tube. The time is measured in seconds (s) for the glass bead to pass between two marks on the tube which are 20 cm apart. This time (s) is the uncorrected "as is" cuprammonium ball fall viscosity. The temperature of the solution is determined, and the correction factor for this temperature is multiplied by the uncorrected viscosity of the sample. This gives the "as is" cuprammonium ball fall viscosity value.

4.4 The "as is" viscosity value for the sample size used is converted to the 2.50-g ACS viscosity by the equations provided in 14.4. The viscosity is reported in "ACS seconds."

5. Significance and Use

5.1 This test method is suitable for use as a rapid control test for pulp manufacture or for careful determination of the viscometric molecular weight of purified cotton or wood derived pulps.

5.2 This test method is applicable over a very large range of cellulose molecular weights because seven sample sizes are defined. (Sample weights are reduced as cellulose molecular weight increases.)

[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] Carver et al., "A Standard Method for Determining the Viscosity of Cellulose in Cuprammonium Hydroxide," *Industrial and Engineering Chemistry, Analytical Edition*, Vol 1, No 1, 1929, pp. 49-51.

[3] 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.

5.3 Cotton and high molecular weight pulps may be difficult to dissolve. (**Warning—**This test method is only valid if the sample dissolves completely without gels.)

6. Interferences

6.1 High temperature drying of pulp causes a reduction in viscosity. Therefore, limit the maximum temperature of the air used to dry the sample to 120 °C and the maximum drying time to 20 min to keep viscosity loss to a minimum. All in-process samples must be washed to remove cooking and bleaching chemicals, because the presence of chemicals while drying will increase viscosity loss.

6.2 The weight of sample used with this test method is critical. The effect of incorrect sample weight on viscosity is shown in Table 1.

6.2.1 If the pulp sample is properly weighed but a small amount fails to dissolve, the viscosity will be incorrect by at least the percentage of the sample that failed to dissolve.

6.3 The volume of cuprammonium solution used is also critical. The effect of incorrect volume on viscosity is shown in Table 2.

6.4 Use the temperature correction factors given in Table 3 to correct the cuprammonium viscosities to 25 °C, assuming that a 1 °C increase causes a 3 % decrease in the measured viscosity of the solution. Correction for temperatures off by more than 5 °C is not recommended. Samples should be retested, ensuring than the CuAm solution is within temperature limits.

7. Apparatus

7.1 *Testing Laboratory,* maintained at 25 ± 2 °C.

7.2 *Picker,* suitable for shredding pulp without damaging it. The picker must have provisions that permit sample remaining after picking is completed to be blown out with compressed air.

7.3 *Drier,* suitable for pulp sample that dries the pulp with hot air whose temperature is never permitted to get higher that 120 °C.

7.4 *Analytical Balance,* capable of weighing to ±0.001 g.

7.5 *Bottles,* wide mouth, glass, for use with an approximately No. 5 rubber stopper, and with a capacity of at least 120 mL (4 oz). The type of bottle must be selected such that it is suitable for dissolving pulps in cuprammonium solution as specified in this test method.

7.6 *Steel Balls,* chrome alloy, Grade 25, 3.2-mm (⅛-in.) diameter.

7.7 *Automatic Pipet,* special made, capable of delivering 97 ± 1 mL of CuAm solution, which is part of the cuprammonium solution filling system (see Fig. 1).

TABLE 1 Effect of Weight Errors on Viscosity Error

Pulp Weight	Percent Viscosity Error	
Error, %	Underweight	Overweight
1	3.8	3.9
2	7.4	8.0
5	17.4	21.1
10	31.8	45.6

TABLE 2 Effect of Volume Errors on Viscosity Error

Volume	Percent Viscosity Error	
Error, (mL)	Low Volume	High Volume
1	4.0	3.9
2	8.2	7.6
5	21.8	17.9
10	48.3	32.6

TABLE 3 Temperature Correction Factors

Temperature	Correction Factor	
Error, °C	Low Temperature	High Temperature
1	0.971	1.030
2	0.943	1.061
3	0.915	1.093
4	0.888	1.126
5	0.863	1.159

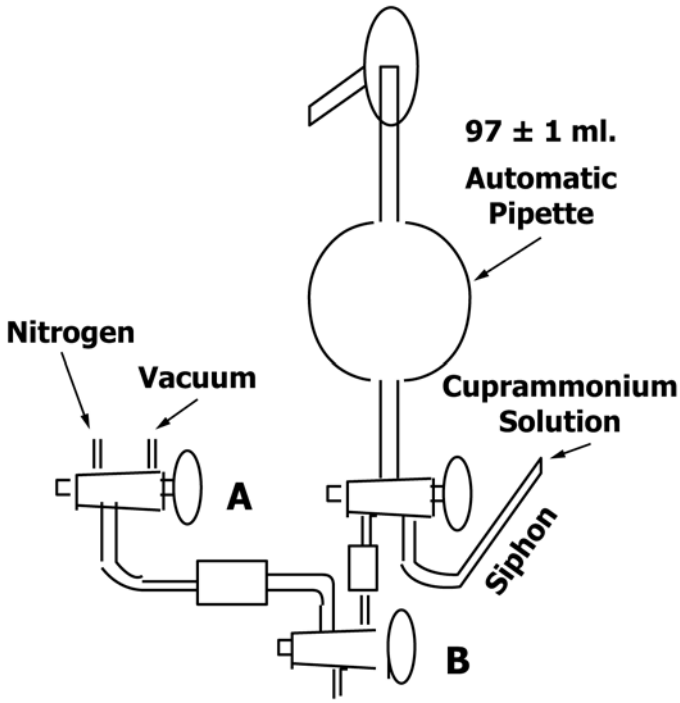

FIG. 1 Solution Filling System

7.8 *Rubber Stopper Assembly,* (see Fig. 2).

7.9 *Vacuum Source,* capable of pulling a vacuum of 686 mm Hg.

7.10 *Shaker,* capable of shaking bottles of cuprammonium solution containing pulp. The shaker is to hold the bottles in a horizontal position, and its design and operation should be such that in-process pulps will be completely dissolved after 20 min of shaking.

7.11 *Transfer Assembly,* for transferring the cuprammonium-cellulose solution from the bottle to the viscosity tube (see Fig. 3).

7.12 *Viscosity Tube,* specially made (see Fig. 4).

7.13 *Glass Viscosity Beads,* for ACS cuprammonium viscosity determination. These beads are to be ground to a diameter that causes the viscosity of each second of bead fall