Designation: D6188 – 17

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 and health 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.

Current edition approved June 1, 2017. Published June 2017. Originally approved in 1997. Last previous edition approved in 2008 as D6188 – 97 (2008) which was withdrawn January 2017 and reinstated in June 2017. DOI: 10.1520/D6188-17.

[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

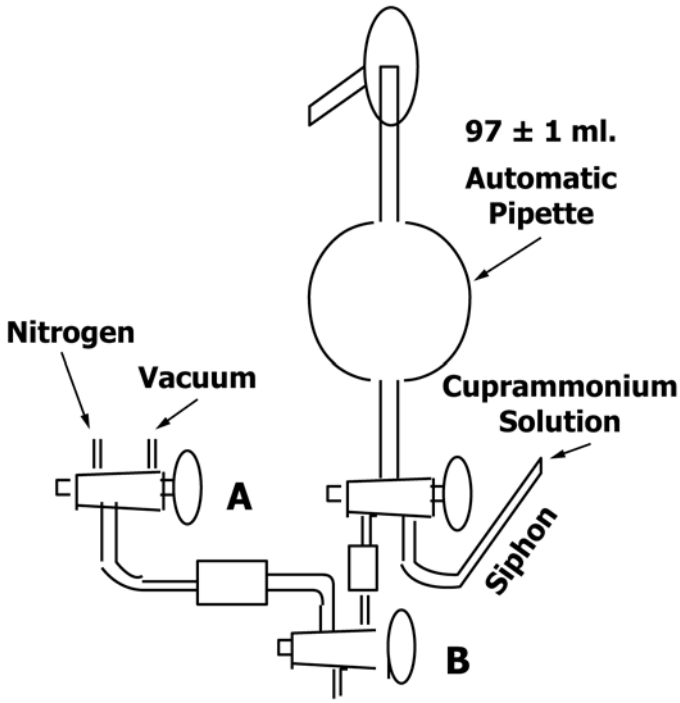


FIG. 1 Solution Filling System

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

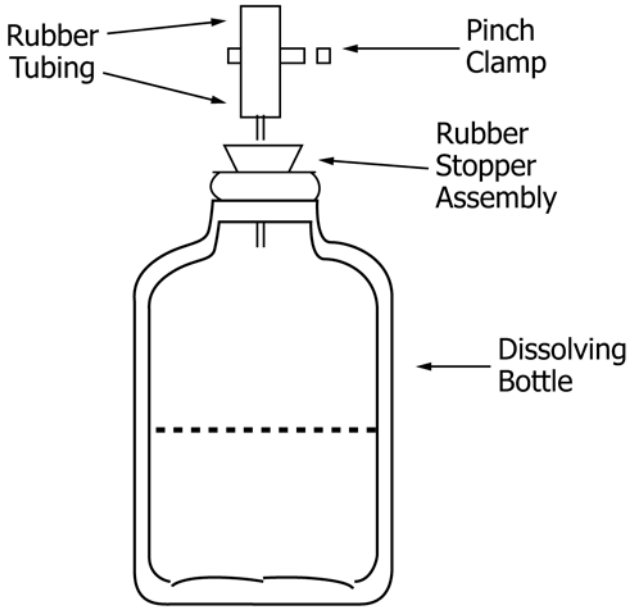


FIG. 2 Rubber Stopper Assembly In Bottle

time in cuprammonium-cellulose solution to equal 22 cP. (This will mean that a sample that has a bead fall time of 10 s will have a viscosity of 220 cP, and a sample that has a bead fall time of 50 s will have a viscosity of 1100 cP). Since the density of various shipments of glass beads will vary somewhat, the diameter of the beads will have to be varied to compensate for the variation in the density of the glass. Generally the beads will be about 3.3 mm in diameter, and they will weigh about 0.046 g.

7.14 *Moisture Balance*—Apparatus to determine the dry weight of cellulose.

7.15 *pH Meter.*

7.16 *Viscosity Tube Holder/Lighting Assembly*—This device holds the viscosity tube vertically with a bright light behind the tube so that the bead falling through the cuprammonium solution can be easily seen.

7.17 *Bead Centering Apparatus*—This consists of a small corrosion resistant funnel topped tube having an internal diameter just sufficient to permit the beads to fall through freely.

7.18 *Timer,* accuracy/precision: ±30 s over 1 h.

7.19 *Plastic Sieve,* with 0.250 mm openings, the same as US Alternate No. 70, Tyler 60 mesh sieve.

7.20 *Plastic Container,* 2500 to 3500 mL graduated polyethylene beaker with handle.

8. Reagents and Materials

8.1 *Cuprammonium Solution,* containing 20 ± 1 g/L of copper (expressed as copper) and 200 ± 2 g/L of ammonium hydroxide (expressed as ammonium).

8.2 *Water,* potable.

8.3 *Water,* reagent (in accordance with Specification D1193) with an electrical resistance of at least 1 000 000 Ω-cm. This water is used to determine the pH of the film left after acid washing.

9. Hazards

9.1 CuAm solution is corrosive, and thus harmful to the skin and eyes. Wear safety glasses or goggles while working with this solution. Gloves and laboratory coat or chemical apron are recommended.

10. Sampling

10.1 The sample for this test may consist of a wet pulp or a dry, finished product sample. Take a representative portion of the pulp sample that contains at least 10 and not more than 25 g of dry pulp.

10.2 If the sample is an in-process pulp sample, thoroughly wash out any process chemicals (acids, bases, or bleach), which may be present. Drain the excess water out of the wet pulp sample (see 10.3).

10.3 If any sample is above 40 % consistency, wet it with demineralized water. If the sample is below 20 % consistency, hand squeeze it or press it until the consistency is between 20 to 40 %.

10.4 Seven sample sizes are authorized by this test method. These sizes, in g, are 0.85, 1.00, 1.25, 1.50, 2.20, 2.40, and 3.50. Select the sample size which will be used for the test. Generally, select a sample size which will give a CuAm viscosity between 15 and 60 s. Never use a sample size that gives a CuAm viscosity of less than 10 or more than 100 s. (If a test result is not within these limits, a new sample size should be selected.)

10.5 In order to ensure that traces of the last sample passed through the picker will not contaminate the new sample, pick enough of the present sample that an amount equal to at least 1 g of dry pulp has passed through the picker. Discard all of this portion of the sample. Then, pick enough of the sample to carry out the viscosity test. Immediately after each sample has been passed through the picker, turn on the compressed air going into the picker for at least 2 s. (This is to blow out as much as possible of the sample before it has time to dry and stick to the surfaces of the picker.)

10.6 Dry the picked sample with air as follows:
10.6.1 Temperature does not exceed 120°C, and
10.6.2 Time does not exceed 20 min.

10.7 Weigh the sample in a manner that will consistently give weighed samples which contain dry pulp weights within the specifications of Table 4.

10.8 More accurate and precise results will be obtained if the pulp sample is conditioned to an equilibrium moisture content and the consistency of the sample determined by moisture balance. This modification provides better weight control, but is not suited for rapid turn-around process control.

11. Calibration and Standardization

11.1 Select control pulp samples for which a relatively large supply is available. Ensure that the cuprammonium viscosities

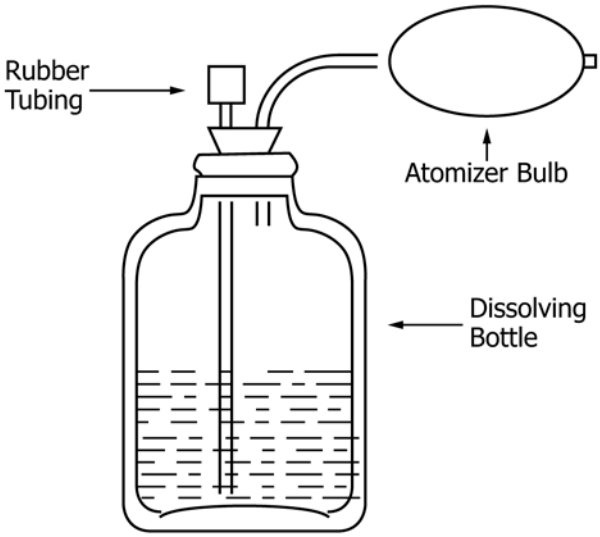


FIG. 3 Solution Transfer Assembly In Bottle

of these samples are sufficiently different so that different sample weights are required. Determine the cuprammonium viscosity of each sample using exactly the same procedure that would be used for testing unknown samples. Record the cuprammonium viscosity in a log book. Keep a separate control chart of the cuprammonium viscosity values (or logarithm of the values) for each of the samples. Measure these control samples at a convenient frequency.

12. Conditioning/Preparation

12.1 Conditioning of dry samples to an equilibrium moisture content will give more accurate and precise results.

12.2 It is usually necessary to treat bottles, stopper assemblies, and viscosity tubes with a solution of sulfuric acid to coagulate the CuAm-cellulose solution before this apparatus can be cleaned. (**Warning**—It is very important that this apparatus be thoroughly washed to remove all of the sulfuric acid before it is dried. If there is a small amount of sulfuric acid remaining on the surface of any apparatus when it dries, a thin film of sulfuric acid will remain on the surface of the apparatus.)

13. Procedure

13.1 Carry out the following operations in a room maintained at 25 ± 2°C (77 ± 3.6°F).

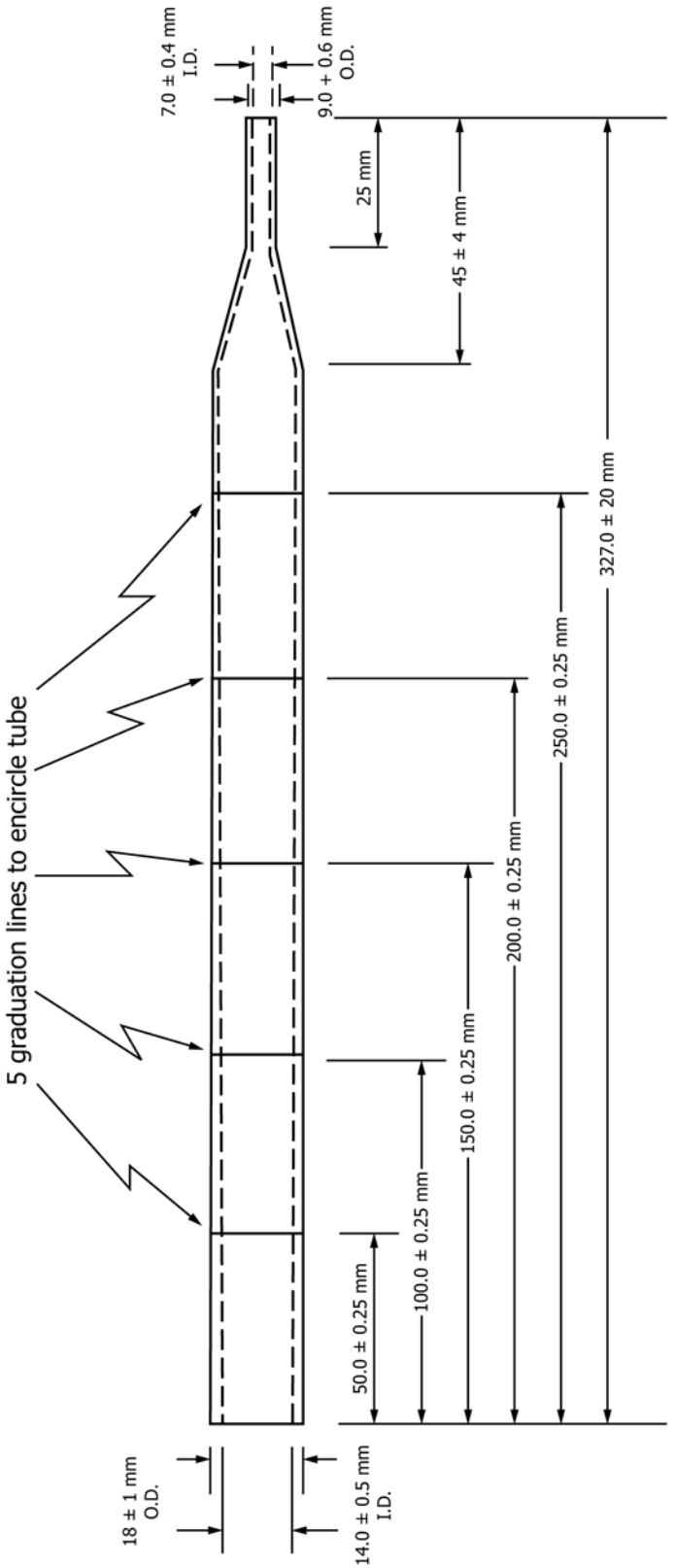
13.2 This test method defines clean, dry bottles; clean, dry stopper assemblies; and clean, dry viscosity tubes as ones that appear to be clean, do not have water or water droplets in or on them, and that will deliver water with a pH not lower than 5.0 or higher than 7.0 when their surfaces are washed with demineralized water.

13.3 In carrying out this part of this test, note that CuAm solution is present in glass tubes and rubber or plastic hose under a slight pressure. (**Warning**—CuAm solution is harmful to the skin, and eyes (see 9.1)).

13.4 Place the weighed sample of pulp in a clean, dry bottle (see 7.5), and add 25 ± 10 clean, dry steel shot to the bottle. Tightly insert a clean dry rubber stopper assembly containing a short glass tube with a short rubber hose connected to the top of the glass tube. It may be desirable to dampen the wall of the stopper with water so that it can be inserted tightly, but do not have any liquid water on or in the rubber stopper assembly. Connect the end of the rubber tubing to the filling assembly illustrated in Fig. 1, and apply a vacuum of at least 660 mm Hg for at least 3 s. to the bottle containing the sample. Then, without loosing the vacuum in the bottle, add 97 ± 1 mL of cuprammonium solution to the bottle. Be sure that there is no CuAm solution in the filling system below the stopcock on the automatic pipet before the addition is begun, and be sure that all of the CuAm solution in the filling system is added to the bottle and that none remains in any part of the filling system. However, do not let air into the bottle after all of the CuAm solution has entered the bottle. (Never apply vacuum to the bottle after the addition of the CuAm solution is begun, since this may result in the loss of several millilitres of the measured 97 mL of CuAm solution that was supposed to have been added to the bottle.)

13.5 Place a hose clamp on the rubber hose on the stopper assembly to prevent air from entering the bottle (see Fig. 2), and disconnect the hose on the stopper assembly from the filling assembly. Place the bottle in the shaker so that the bottle is in a horizontal position, and shake for 25 ± 3 min. Stop the shaker and remove the bottle. Observe the bottle to see if there is any indication of undissolved material in the solution. If there is any indication of undissolved material, discard solution, and report that the sample did not dissolve. If there is no indication of undissolved material, point the open end of the rubber hose on the stopper assembly away from you and remove the hose clamp. (Sometimes, some of the cuprammonium/ cellulose solution is sprayed out of the rubber hose when the hose clamp is removed.) Remove the stopper assembly from the bottle.

13.6 Insert the transfer assembly into the bottle of cuprammonium-cellulose solution (see Fig. 3). Place a clean, dry viscosity tube in an exactly vertical position in the viscosity



NOTE 1—Fabricate from borosilicate glass conforming to type 1, class A or class B specifications in accordance with Specification E438.

FIG. 4 Custom Made Viscosity Tube

TABLE 4 Limits on Sample Sizes

Sample Size, g	Range, g
0.85	0.850 ± 0.008
1.00	1.000 ± 0.010
1.25	1.250 ± 0.012
1.50	1.500 ± 0.015
2.20	2.200 ± 0.022
2.40	2.400 ± 0.024
3.50	3.500 ± 0.035

tube holder. Connect the top of the longest tube of the transfer assembly to the rubber hose on the bottom of the viscosity tube. Using a very low air flow rate through a rubber hose connected to the shortest tube of the transfer assembly, blow the cuprammonium-cellulose solution into the viscosity tube, filling the tube to a level which is at least 2.5 cm. above the top mark on the viscosity tube. Then, clamp the hose on the bottom of the viscosity tube with a hose clamp and disconnect the bottom end of the hose from the filling assembly.

13.7 When carrying out the viscosity measurement, the solution level must be at least 2.5 cm above the top mark on the viscosity tube so that the glass bead will have passed through enough solution to slow down before it gets to the first mark used for measuring the viscosity. Position the bead centering apparatus over the viscosity tube. Drop one of the special glass beads into the center of the viscosity tube. The time in seconds required for the bead to fall the 20 cm distance between the top mark on the viscosity tube and the bottom mark on the tube is the uncorrected CuAm viscosity for the sample size used. It is permissible to measure the time in seconds for the bead to fall between two of the marks on the viscosity tube (5 cm apart) and then multiplying the measured time by four to get the time that would have been required for the bead to fall 20 cm as long as the measured time is at least 10 s. Never measure the bead fall time for a shorter distance than 5 cm. (In a similar manner, a 10 cm distance may be used.)

13.8 As soon as the uncorrected cuprammonium viscosity of the sample is determined, record the temperature of the cuprammonium-cellulose solution remaining in the bottle to the nearest °C. For finished product samples, if the solution temperature is below 20°C or above 30°C, do not use this value. Retest the sample when the solution temperature is under control.

14. Calculation

14.1 Correct the viscosity values for all sample weights to the value that would have been obtained if the cuprammonium-cellulose solution had been 25°C using the following equation:

$$CV = UV \times CF \quad (1)$$

where:

CV = corrected CuAm viscosity in seconds,
UV = uncorrected viscosity in seconds required for a standard glass bead to fall 20 cm in the CuAm-cellulose solution at the actual solution temperature, and
CF = temperature correction factor from 6.4. (It is recommended that solution temperature be maintained within ±5°C of the target temperature.).

14.2 If reporting the uncorrected CuAm viscosity, also report the sample weight, because the method is actually seven different tests–one test for each of the 7 sample weights used.

14.3 Convert the "as is" CuAm viscosity value to the ACS CuAm viscosity value by substituting the appropriate factors from Table 5 into the following equation:

$$\text{ACS seconds} = \text{anti}\log_{10}\left[A \quad (\log_{10}\text{"as is" viscosity}) + B\ \right] \quad (2)$$

where:

ACS seconds = the 2.5 g viscosity in s. (see Footnote[2]),
A = the conversion factor listed in Table 5 for the test size used,
B = the conversion factor listed in Table 5 for the test size used, and
"as is" viscosity = the corrected viscosity value from 14.1.

14.4 Factors for converting to "2.5 g ACS seconds cuprammonium viscosities" are in Table 5.

14.5 For example, if a sample is found to have a 40 s. "as is" viscosity using the 2.2 g test, the value is reported as 68.9 ACS s ($10^{\wedge}$(1.0191 × log(40) +0.2056). For 60 s at 0.85 g, a 15 365 s ACS viscosity would be reported. For 50 s at 3.5 g, an ACS viscosity of 11.8 s. would be reported.

14.6 For manual operation, the equations in 14.5 can be used to prepare printed "look-up" tables at values ranging from 10 to 100 s for each test. For computerized applications, these equations may be built into a spreadsheet or database program.

15. Report

15.1 Report the following information:

15.1.1 The "as is" viscosity value in seconds and the test weight used. (This data may be used for process control and preparing control charts.)

15.1.2 The ACS viscosity calculated from the "as is" value.

15.2 *Conversion to Degree of Polymerization:* The following equation[4] can be used to convert the ACS viscosity measured by this test method to a weight average degree of polymerization as follows:

$$DP_W = 47.485 \times \text{In}^2\,\text{CuAm} + 243.745 \times \text{In CuAm} + 422.04 \quad (3)$$

where:

DP_W = weight average degree of polymerization, and
CuAm = ACS viscosity from 15.1.2.

[4] Morton, J. H., *The Chemistry and Processing of Wood and Plant Fibrous Materials*, Kennedy, J. F., Phillips, G. O., Williams. P. A. eds., Chapter 15, Woodhead Publishing Ltd., Cambridge, Eng., 1996.

TABLE 5 Factors for Converting "As Is" Seconds to "ACS Viscosity"

Test	*A*-factor	*B*-factor
0.85	1.2845	1.9025
1.00	1.2952	1.4646
1.25	1.1355	1.1501
1.50	1.0991	0.8549
2.20	1.0191	0.2056
2.40	0.9948	0.0694
3.50	0.8490	-0.3690

16. Precision and Bias

16.1 *Precision*—Interlaboratory data has not been obtained. Control charting of the 1.0 g test indicates that the 3σ upper and lower control limits are ±27 s at 100 s for the "as is" viscosity. In general, precision improves at lower viscosity, that is, the precision improves as the test weight increases or as the "as is" time decreases at a given weight.

16.2 *Bias*—No justifiable statement can be made on the bias of the procedure for measuring viscosity because no suitable reference material exists.

17. Keywords

17.1 ball fall viscosity; cellulose; cuprammonium; DP; molecular weight; process control; viscosity