Designation: D 6188 – 97 (Reapproved 2003)

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

This standard is issued under the fixed designation D 6188; 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.*

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[3]

D 1695 Terminology of Cellulose and Cellulose Derivatives[4]

E 438 Specification for Glasses in Laboratory Apparatus[5]

## 3. Terminology

3.1 This standard terminology of cellulose and cellulose derivatives, see Terminology D 1695.

## 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.)

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

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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] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 6.03.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

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).

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).

**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 |

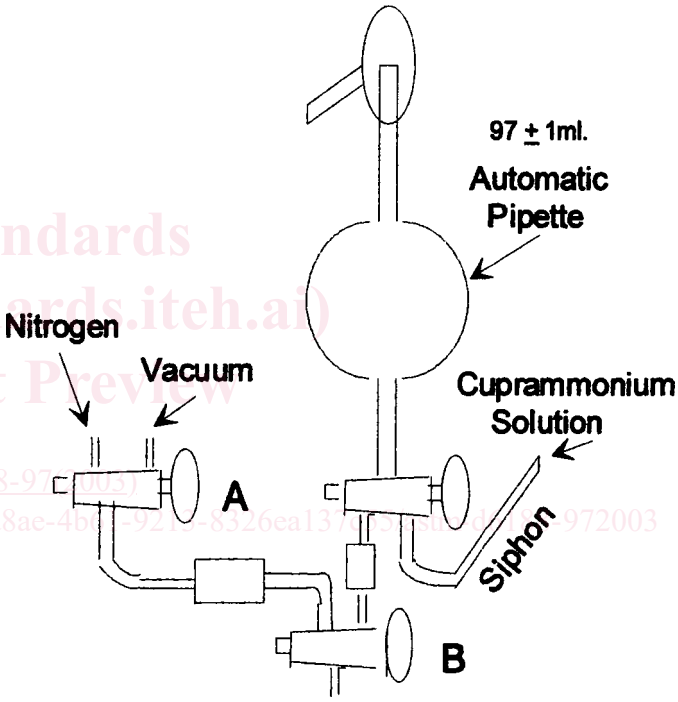


**FIG. 1 Solution Filling System**

7.12 *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 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*.

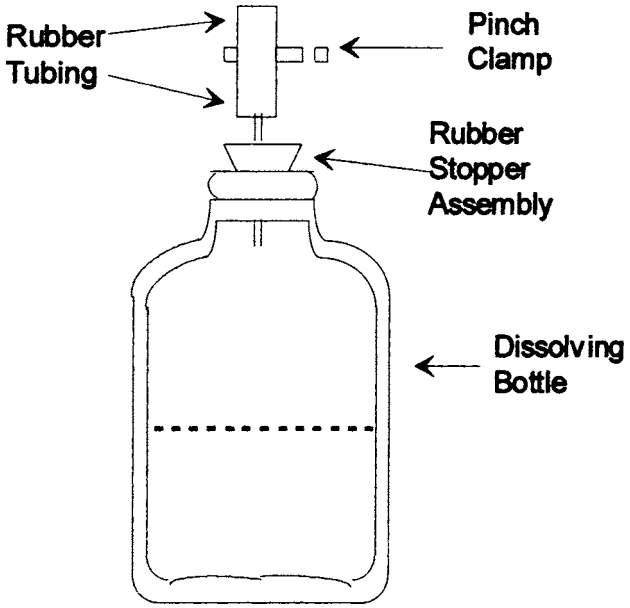


FIG. 2 Rubber Stopper Assembly In Bottle

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 D 1193) 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 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