

# Fluidity of Dispersions of Cellulose from Bleached Cotton Cloth

Developed in 1954 by AATCC Committee RA34; reaffirmed 1961, 1968, 1972, 1975, 1979, 1984; editorially revised 1974, 1975, 1983, 1985, 1988, 1990, 2004; editorially revised and reaffirmed 1989, 2001; revised 1996.

## 1. Purpose and Scope

1.1 The test procedures in this test method are applicable to bleached, unfinished cotton cloth. The fluidity of a dispersion of bleached cellulose fibers in cupriethylene diamine or cuprammonium hydroxide solvent is a sensitive measure of the degradation of the cellulose resulting from the action of acids, alkalis, oxidizing or reducing agents. It is therefore useful in determining the completeness of the bleaching treatment to which the cloth has been subjected, as well as the effect of chemical treatments on the tensile strength of the cloth.

## 2. Principle

2.1 The length of chain of cellulose molecules is decreased when treated with acid, alkali, oxidizing or reducing agents, etc. The severity of the treatment determines the extent of the shortening of the cellulose chain and the fluidity, or ease of flow, of a dispersion of the cellulose in the solvents.

## 3. Terminology

3.1 **fluidity**, *n.*—of a cellulose solution, a measure of the ease of flow or motion of a solution, and therefore an indicator of the molecular weight of the cellulose.

3.2 **rhe**, *n.*—the unit of fluidity; the reciprocal of the unit of viscosity (the poise).

## 4. Safety Precautions

NOTE: These safety precautions are for information purposes only. The precautions are ancillary to the testing procedures and are not intended to be all inclusive. It is the user's responsibility to use safe and proper techniques in handling materials in this test method. Manufacturers MUST be consulted for specific details such as material safety data sheets and other manufacturer's recommendations. All OSHA standards and rules must also be consulted and followed.

4.1 Good laboratory practices should be followed. Wear safety glasses in all laboratory areas.

4.2 All chemicals should be handled with care. Use chemical goggles or face shield, impervious gloves and an impervious apron during dispensing and mixing. CAUTION: Always add acid to water.

4.3 An eyewash/safety shower should be located nearby and a self-contained breathing apparatus should be readily available for emergency use.

4.4 Exposure to chemicals used in this procedure must be controlled at or below levels set by governmental authorities (e.g., Occupational Safety and Health Administration's [OSHA] permissible exposure limits [PEL] as found in 29 CFR 1910.1000 of January 1, 1989). In addition, the American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Values (TLVs) comprised of time weighted averages (TLV-TWA), short term exposure limits (TLV-STEL) and ceiling limits (TLV-C) are recommended as a general guide for air contaminant exposure which should be met (16.1).

## 5. Materials and Reagents

- 5.1 Ammonium hydroxide ( $\text{NH}_4\text{OH}$ )
- 5.2 Cupriethylene diamine
- 5.3 Cuprammonium hydroxide
- 5.4 Nitric acid ( $\text{HNO}_3$ )
- 5.5 Glacial acetic acid ( $\text{CH}_3\text{COOH}$ )
- 5.6 Sodium hydroxide ( $\text{NaOH}$ )
- 5.7 Cupric hydroxide ( $\text{Cu}(\text{OH})_2$ )
- 5.8 Ethylene diamine ( $\text{H}_2\text{NCH}_2\text{CH}_2\text{NH}_2$ )
- 5.9 Sulfuric acid ( $\text{H}_2\text{SO}_4$ )
- 5.10 Nitrogen ( $\text{N}_2$ )
- 5.11 Bromine ( $\text{Br}_2$ )
- 5.12 Potassium iodide (KI)
- 5.13 Sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ )
- 5.14 Methyl Red indicator
- 5.15 Ostwald Cannon-Fiske viscometers (see 16.3)
- 5.16 Cuprammonium dissolving tubes (see 11.2)
- 5.17 Capillary viscometers (see 11.3)

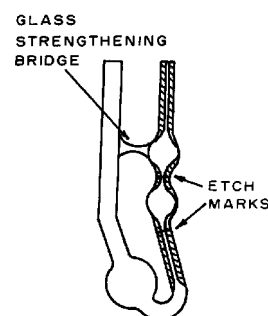


Fig. 1—Ostwald Cannon-Fenske cuen viscosimeter.

## 6. Preparation of Sample

6.1 Use a representative sample of bleached and unfinished cloth weighing not less than 5 g.

6.2 Cut the sample by hand, with electric scissors, or by use of a Wiley cutting mill with a 20-mesh sieve and top delivery tube, so that short individual fibers about 1.5 mm (0.06 in.) long are obtained.

6.3 Run duplicate tests, using fibers from the one sample. Divide the cut sample into two portions of approximately equal weight and store in separate containers overnight in a desiccator containing conc. sulfuric acid. Samples conditioned in this manner will have a moisture content of 2%.

6.4 The purpose of dividing the sample into two portions is to reduce the time of exposure to the atmosphere during weighing. The weight of the specimen for the fluidity determination should be sufficient to give a 0.5% solution of the cotton.

## 7. Method No. 1—Dispersion in Cupriethylene Diamine Hydroxide (Cuen)

7.1 Ostwald Cannon-Fenske-type viscosimeters (Fig. 1) having the characteristics shown in Table I are suitable for determining fluidities in the range normally found for bleached cotton.

Table I—Viscosimeter Characteristics

| Size No. | Approx. Range of Efflux Time, s | Capillary Diameter, cm | Fluidity Range Rhes |
|----------|---------------------------------|------------------------|---------------------|
| 200      | 100 to 700                      | 0.097 to 0.103         | 1.43 to 10.0        |
| 300      | 100 to 700                      | 0.120 to 0.130         | 0.80 to 4.0         |
| 400      | 100 to 700                      | 0.180 to 0.190         | 0.1175 to 0.83      |

## 8. Preparation of Solutions

8.1 Prepare 0.167*M* cupriethylene diamine solution by diluting 167 mL of the 1.0*M* solution with freshly boiled and cooled water to 1 L in a volumetric flask, observing all precautions to exclude air.

8.2 Store the reagents at all times under oxygen-free nitrogen to minimize decomposition.

8.3 Measure the 1.0*M* and 0.167*M* solutions from burettes of known accuracy. The burettes may be attached to the reagent storage bottles by means of a side arm sealed into the lower part of the burette and filled by application of nitrogen pressure on the storage bottle.

## 9. Procedure

9.1 Weigh 0.4-0.45 g of the sample, prepared as previously described to the nearest 0.1 mg. Remove the samples from the desiccator one at a time, and make the weighings as quickly as possible to prevent additional moisture pickup from the air.

9.2 Transfer the fibers carefully from the weighing dish or watch glass to an approximately 120 mL (4 oz.) wide-mouth amber glass bottle.

9.2.1 To the specimen in the solution bottle, add *X* mL of 0.167*M* cupriethylene diamine solution, where *X* is calculated as follows:

$$X = 120 \times \text{weight of specimen} \times 0.98$$

9.3 Sweep out the air in the bottle above the solution by applying a brisk stream of purified nitrogen over the surface of the solution for 20 s, and quickly screw the cap of the bottle in place.

9.4 Shake the bottle by means of an automatic shaker at intermediate speed for 2 h.

9.5 Remove the bottle from the shaker and add to the solution *Y* mL of 1.0*M* cuen, where *Y* is calculated as follows:

$$Y = 80 \times \text{weight of specimen} \times 0.98$$

9.6 Repeat the nitrogen treatment and bottle-shaking as described, with the exception that the shaking time should be 3 h.

9.7 Permit the bottle to stand for 30 min after removing from the shaker and before loading the viscosimeter. The viscosimeter is loaded by inverting it with the capillary side arm submerged in the cellulose dispersion and applying suction to the other arm of the instrument. Draw the dispersion into both the bulbs on the capillary arm to the etched mark. Rotate the pipette to its normal vertical position, and place the viscosimeter in a constant-temperature water bath at  $25 \pm 1^\circ\text{C}$  ( $77 \pm 2^\circ\text{F}$ ). The liquid will drain into the lower reservoir and will reach temperature equilibrium with the bath (ap-

prox. 5 min).

9.8 Determine the efflux time by drawing the liquid above the mark between the two bulbs and measuring the time for the meniscus to pass from the mark between the two bulbs to the mark below the lower bulb. Take the average of two or more observations.

## 10. Calculations

10.1 The fluidity in rhes, *F*, is calculated as follows:

$$F = 100/ctd$$

where:

*c* = instrument constant determined from a liquid of known viscosity.

*t* = time of discharge of the liquid within the ring interval.

*d* = density of cuen solution, 1.052.

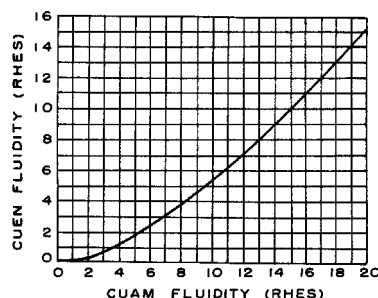
## 11. Method No. 2—Dispersion in Cuprammonium Hydroxide (Cuam)

11.1 Use viscosimeters of the glass pipette type with the dimensions shown in Fig. 2.

11.2 Dissolving tube.

11.2.1 The dimensions of the tube used for dissolving cellulose in cuprammonium solution are shown in Fig. 3. It is recommended that the tubes be made of heavy walled tubing to withstand the dropping force of the stainless steel balls. The volume of the tube is approximately 35 mL but this volume should be measured accurately by weighing the empty stoppered tube containing the stainless steel balls and then weighing it again filled with distilled water. Two 0.63 cm (0.25 in.) diameter stainless steel balls are used in each dissolving tube. These tubes can be purchased from the Ace Glass Co., Vineland NJ (Cat. No. B-1980).

| CLOTH             | FLUIDITY— RHES |             |
|-------------------|----------------|-------------|
|                   | CUEN           | CUAM        |
| GREIGE            | 0.2 - 0.4      | 1.0 - 2.0   |
| MILDLY TREATED    | 0.3 - 0.7      | 1.5 - 2.5   |
| AVERAGED BLEACHED | 0.9 - 3.3      | 3.0 - 7.0   |
| OVERBLEACHED      | 5.5 - 15.3     | 10.0 - 20.0 |



Evaluation—Cuam and cuen fluidities.

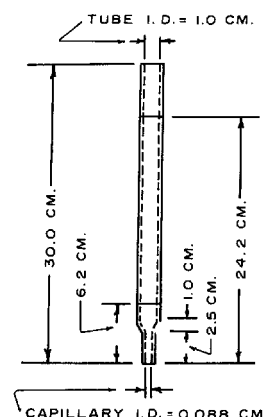


Fig. 2—Cuprammonia viscosimeter tube.

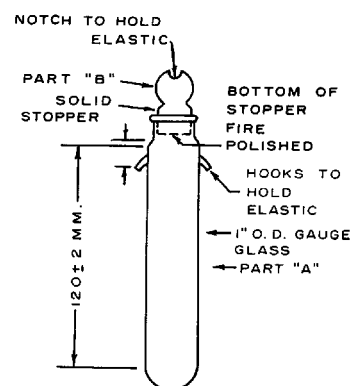


Fig. 3—Dissolving tube for cuam solutions.

## 12. Preparation of Solution

12.1 The cuprammonium solution should contain per liter  $15 \pm 0.25$  g of copper,  $200 \pm 10$  g of ammonia, and less than 0.5 g of nitrites and nitrates calculated as nitrous acid. Prepare the cuprammonium solution by passing a stream of air through a solution of ammonium hydroxide (sp gr 0.88-0.90) at room temperature, and then bubbling it into approximately 1.5 L of a solution of ammonium hydroxide (sp gr 0.88-0.90) at  $0 \pm 1^\circ\text{C}$  ( $32 \pm 2^\circ\text{F}$ ) in which is dissolved 1.5 g of cane sugar, and to which has been added 60 g of powdered copper. Stir the solution constantly and continue the stream of air until the solution contains a little more than 17 g of copper per liter as determined by the following test: Stop the stirring and the flow of air and allow the undissolved copper to settle for a few minutes. Transfer 5 mL of the dispersion by means of a pipette to a Nessler tube and dilute to 50 mL with water. To a second Nessler tube containing 10 mL of ammonium hydroxide (sp gr 0.90), add 5