

Test Method for Fluidity of Dispersions of Cellulose from Bleached Cotton Cloth

Developed in 1954 by AATCC Committee RA34; reaffirmed 1961, 1968, 1972, 1975, 1979, 1984, 2012, 2016; editorially revised 1974, 1975, 1983, 1985, 1988, 1990, 2004, 2008, 2010, 2019; editorially revised and reaffirmed 1989, 2001; revised 1996, 2007.

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 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. (Fluidity of dispersions in cuprammonium hydroxide has been dropped from this test method; see 11.1 for details.)

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 manufacturers recommenda-

tions. 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; see web site: www.osha.gov for latest version). 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 (see 11.2).

5. Materials and Reagents

5.1 Cupriethylene diamine, 1.0 M solution.

5.2 Nitrogen (N_2)

5.3 Ostwald Cannon-Fiske viscometers (see Fig. 1 and 11.3).

6. Preparation of Sample

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

6.2 Cut the sample by use of a 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

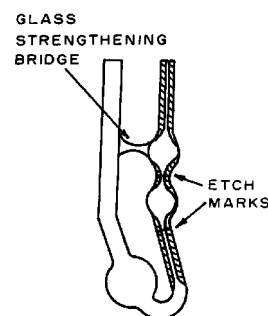


Fig. 1—Ostwald Cannon-Fenske viscometer.

weighing. The weight of the specimen for the fluidity determination should be sufficient to give a 0.5% solution of the cotton.

7. Preparation of Solutions

7.1 Prepare 0.167 M cupriethylene diamine solution by diluting 161 mL of the 1.0 M solution with freshly boiled and cooled water to 1.0 L in a volumetric flask, observing all precautions to exclude air.

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

7.3 Measure the 1.0 M and 0.161 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,

8. Procedure

8.1 Ostwald Cannon-Fenske-type viscometers (see Fig. 1) having the characteristics shown in Table I are suitable for determining fluidities in the range normally found for bleached cotton. It is recommended that the efflux time (see 8.9) exceed 100 s in order to ensure accuracy.

8.2 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.

8.3 Transfer the fibers carefully from the weighing dish or watch glass to an approximately 120 mL (4 oz.) widemouth amber glass bottle.

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

8.3.1 To the specimen in the solution bottle, add X mL of 0.167 M cupriethylenediamine solution, where X is calculated as follows:

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

8.4 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.

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

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

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

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

8.8 Permit the bottle to stand for 30 min after removing from the shaker and before loading the viscometer. The viscometer should be flushed with nitrogen gas prior to being loaded with the dispersion. The viscometer 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 viscometer 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 (approx. 5 min).

8.9 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.

9. Calculations

9.1 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 in seconds.

d = density of cuen solution, 1.052.

10. Precision and Bias

10.1 Precision Estimate.

10.1.1 *Single Laboratory Study.* Estimates of Components of Variance for technicians (VO) and residual error (V), and their associated Critical Differences, CDm (multiple technicians in lab) and CDo (single technician in lab) were determined in a single laboratory in 2005. Three technicians performed duplicate tests on materials, according to 6.3 at three treatment levels of no bleach (NB), standard bleach (SB), and over bleached (OB), using the Cuen Method.

10.1.2 *95% Precision Estimates from Individual Technician and Treatment Values.* The results shown in Table II are the average of two determinations by a single technician. Based on these averages, the components of variance (Vo) and residual error (V) for each technician were computed. As can be seen even in this limited study, there was an apparent difference in the results reported by Technician 3, for

each of the treatments. This is a very limited study, and caution is advised in the application of these estimates to specific testing scenarios (see Table II).

With three technicians involved in this limited study, it was possible to assess the effect of multiple technicians in test result comparisons by looking across technicians in treatments and calculating VO (technician) and V (residual) components of variance for each treatment. The above summary shows rather large values for CDm (multiple O in lab), due to the level differences among technicians. This is a very limited study, and caution is advised in application of these estimates to specific testing scenarios. All of the above considerations were based on a 95% confidence level.

10.1.3 *95% Precision Estimates from Analysis of the Whole Data Set.* Using Analysis of Variance, Components of Variance for technicians and residual were computed and are shown in Table III. As a result of the difference in results reported by Technician 3, the values generated for CDm (multiple operators in a single lab) were also high. These estimates pertain only to *within laboratory* precision. A much broader study, with multiple laboratory participation, must be made to yield reliable *between laboratory* estimates (see Table III).

Table II—Data Summary for Technicians and Treatments

		TMT NB	TMT SB	TMT OB	Technician Summaries
Tech 01	Av(2)	0.564	0.811	4.033	V 0.0006
	V	0.0013	0.0004	0.0002	CDo 0.05
	CDso	0.07	0.04	0.03	
Tech 02	Av(2)	0.609	0.885	4.172	V 0.00014
	V	0.0003	0.0001	0.00004	CDo 0.02
	CDso	0.03	0.02	0.01	
Tech 03	Av(2)	0.397	0.745	3.625	V 0.0107
	V	0.0022	0.0238	0.0060	CDo 0.20
	CDso	0.09	0.31	0.15	
		Treatment Summaries			AV-Rhes Units DC-Rhes Units V-Rhes squared
		TMT1	TMT2	TMT3	
3 Tech (O=3)	VO	0.0118	0	0.0795	
	V	0.0012	0.0088	0.0021	
Single O Multiple O	CDo	0.07	0.19	0.09	
	CDm	0.31	0.19	0.79	

CD Equations: $CDo = 2.8 (V/2)^{0.5}$ $CDm = 2.8 (V/2 + VO)^{0.5}$

Table III—Components of Variance and 95% Precision Estimates

		Components of Variance	
Source		COV	Unit Var
O.L		VO	0.0307
Res		V	0.0038
		95% Precision Estimates	
Single O in Lab		CDo	0.12
Multiple O in Lab		CDm	0.51
		$CDso = 2.8 (V/2)^{0.5}$ $CDm = 2.8 (V/2 + VO)^{0.5}$	