

T 254 cm-10

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CAUTION:

This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Material Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this test method, the user should determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Cupriethylenediamine disperse viscosity of pulp (falling ball method)

1. Scope

1.1 This method describes a rapid procedure (1, 2, 3) for the measurement of the viscosity of 1% cellulose solutions using cupriethylenediamine saturated with cupric hydroxide $[\text{Cu}(\text{OH})_2]$ as the solvent. For convenience, this may be referred to as $\text{Cu}(\text{En})_2$ or CED.

1.2 This method is the preferred procedure for pulps containing more than 4% lignin; however, many high yield pulps, at levels above 50%, contain sufficient lignin (in excess of 10%) to interfere with the test. The falling ball method may be used over the entire range of bleached and unbleached pulps and is well suited to production control. However, the preferred procedure for pulps with less than 4% lignin, as defined by TAPPI T 222 "Acid-Insoluble Lignin in Wood and Pulp," is TAPPI T 230 "Viscosity of Pulp (Capillary Viscometer Method)," which should be used for referee purposes on these pulps.

1.3 This procedure covers preparation of cupriethylenediamine, dissolving the sample, viscometer calibration, and measurement of the viscosity of the sample solution.

2. Summary

In this method, the viscosity of a 1% cellulose solution is measured by dissolving a representative sample of air-dry pulp in 0.5M cupriethylenediamine solution at a controlled temperature. Viscosity is measured by timing the fall of an aluminum ball of known density through a given distance in a calibrated glass tube.

3. Significance

The purpose of measuring solution viscosity of a pulp is to determine the average cellulose molecular weight after processing and bleaching, hence, to measure the degree of degradation. In general, the lower the viscosity, the lower the physical strengths, such as burst and tear factor, breaking length, fold, and rupture energy.

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4. Apparatus

- 4.1 *Cylinder of nitrogen gas*, fitted with a reducing valve to give 14 to 21 kPa (2 to 3 psi) pressure, used to protect cupriethylenediamine from oxidation.
- 4.2 *Tube holder*, of a design similar to that shown in Fig. 1.
- 4.3 *Aluminum alloy balls*¹, 1.59 mm (0.0625 in.) in diameter with a sphericity of 0.051 mm (0.002 in.) made of 2017-T4 aluminum alloy.
- 4.4 *Dissolving bottles*, 50-mL (2.0-oz) flat medicine bottles with plastic screw cap and polyethylene gasket.
- 4.5 *Viscosity tubes*, glass, 300 mm long with an inside diameter of 10 ± 0.05 mm. These may be obtained from laboratory supply houses. They are to be etched with rings exactly 150 mm apart, each ring approximately equidistant from the end of the tube.
- 4.6 *Laboratory shaker*, variable speed, modified to accommodate one or more dissolving bottles in a horizontal position and securely held down.
- 4.7 *Stopwatch or timer*, with sweep hands calibrated to 0.1 s.

5. Reagents and materials

- 5.1 *Standard viscosity oil*¹, having a viscosity in the range of 200 mPa • s at 25°C. Table 1 lists several satisfactory viscosity standards.
- 5.2 *Sulfuric acid*, 4N H₂SO₄, reagent grade.

NOTE 1: Once opened, the standard viscosity oil does not have prolonged shelf life and should be replaced at regular intervals.

- 5.3 *Potassium iodide*, KI reagent grade crystals.
- 5.4 *Sodium thiosulfate*, 0.1N Na₂S₂O₃, reagent grade.
- 5.5 *Ammonium thiocyanate*, 20% NH₄CNS, reagent grade.
- 5.6 *Copper sulfate*, CuSO₄ • 5H₂O, reagent grade.
- 5.7 *Hydrochloric acid*, 0.1N HCl, reagent grade.
- 5.8 *Ammonium hydroxide*, NH₄OH, concentrated reagent grade.
- 5.9 *Sodium hydroxide*, 20% solution reagent grade.
- 5.10 *Ethylenediamine*, 70% technical grade.
- 5.11 *Cupriethylenediamine*, obtainable commercially¹ or prepared by the procedures given in the Appendix.

Table 1. Standard viscosity oil

Viscosity standard*	Approximate viscosity at 25°C, mPa • s	To replace NBS viscosity standards
S-3	3.3	D
S-6	7.7	H, I
S-20	30	J, K
S-60	100	L, SB
S-200	430	M
S-600	1400	N, SF

*Standard identification number for general purpose viscosity standards from Cannon Instrument Company delegated by NIST to provide U. S. national measurement standards for liquid viscosity measurement.

¹ Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list, available as part of the CD or printed set of Standards, or on the TAPPI website general Standards page.

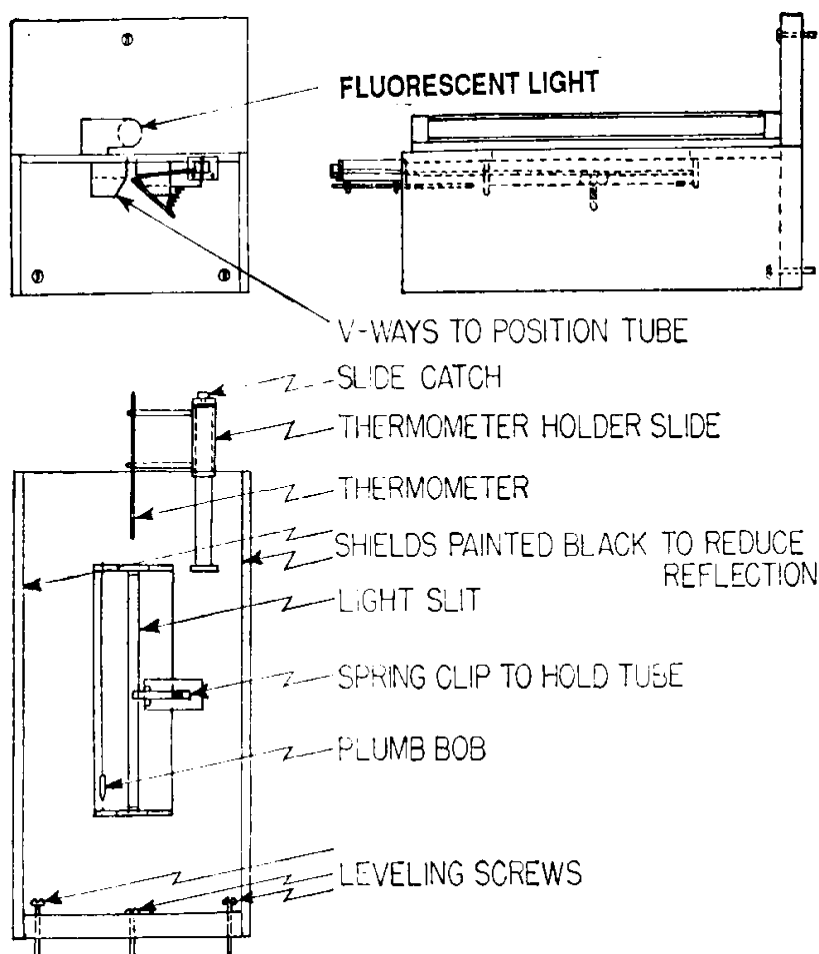


Fig. 1. Tube holder.

6. Safety

Cupriethylenediamine solution is a strong caustic. Avoid contact with eyes, skin, and clothing. Avoid breathing the vapor. Spills on the skin or splashes in the eye must be thoroughly flushed with cold running water. Seek medical attention at once, particularly for exposure to the eyes.

7. Sampling and test specimens

7.1 Obtain a 5 to 7 g sample of the pulp and reduce to the air-dry state as follows. Form slush samples into thin sheets on a Büchner funnel and air dry. Slurry hard, dry samples in water by shaking or stirring and make into thin sheets. Tear apart the air-dry sheets by hand into small pieces. The use of a mechanical shredder, such as a Wiley mill, is not recommended as the viscosity is likely to be lowered as a result of this process of disintegration.

7.2 Many pulps, particularly those having a lignin content above 4%, are difficult to redisperse (8.3) after air-drying from a water suspension. With these pulps, the following alternative procedure is recommended.