



TAPPI/ANSI T 230 om-13

SUGGESTED METHOD – 1946
OFFICIAL STANDARD – 1976
OFFICIAL TEST METHOD – 1982
REVISED – 1989
REVISED – 1994
REVISED – 1999
REVISED – 2004
REVISED – 2008
REVISED – 2013
©2013 TAPPI

The information and data contained in this document were prepared by a technical committee of the Association. The committee and the Association assume no liability or responsibility in connection with the use of such information or data, including but not limited to any liability under patent, copyright, or trade secret laws. The user is responsible for determining that this document is the most recent edition published.

CAUTION:

This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. 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 method, the user must 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.

Viscosity of pulp (capillary viscometer method)

1. Scope

This method describes a procedure for determining the viscosity of 0.5% cellulose solutions, using 0.5M cupriethylenediamine (CED) as a solvent and a capillary viscometer. Measurements may be made on bleached cotton and wood pulps. Conventional kraft pulps with up to 4% lignin, as defined by TAPPI T 222 “Acid-Insoluble Lignin in Wood and Pulp” can also be analyzed. The applicability of this procedure to extended delignification pulps has not been determined.

2. Summary

2.1 This procedure describes the techniques for dissolving the pulp and measuring of the viscosity of the pulp solution. The procedure gives two alternative techniques for dissolving the pulp sample. This first technique involves mechanical shaking of the sample-solvent mixture in a closed bottle containing glass beads. The second technique involves stirring the sample and solvent in an open tube with a copper rod. A survey of users and an interlaboratory test program indicated an equal preference for the two techniques.

2.2 The second technique, which involves stirring the sample and solvent in an open tube, has the potential for oxidation of the cupriethylenediamine solution. If this occurs the method will give erroneous results. Caution should be taken to minimize the potential oxidation by continuous flooding of the open end of the dissolving tube with nitrogen.

3. Significance

3.1 The solution viscosity of a pulp gives an indication of the average degree of polymerization of the cellulose. Such a test therefore gives a relative indication of the degradation (decrease in cellulose molecular weight) resulting from the pulping and/or bleaching process.

3.2 Caution should be exercised in drawing conclusions about pulp strength properties strictly from viscosity results unless previous investigation has identified the relationship.

Approved by the Standard Specific Interest Group for this Test Method



Documents

Document Sharing Hub

4. Apparatus

4.1 General.

4.1.1 *Cylinder of nitrogen gas*, purity 99.998%, fitted with a pressure reducing valve to give 14 to 21 kPa (2 to 3 psi) pressure.

4.1.2 *Constant temperature bath*, capable of being maintained at $25.0 \pm 0.1^\circ\text{C}$ and equipped with clamps to support the viscometers in the thermostating fluid.

4.1.3 *Viscometer*, capillary type, size chosen according to expected viscosity range in compliance with the characteristics for oil calibrations shown in Table 1. In order to avoid correction for the kinetic energy effect, choose a viscometer with an efflux time of over 100 s, but less than 800 s. Viscometer calibration is given in Appendix A.

4.1.4 *Stopwatch or electric timer*, readable to 0.1 s.

4.1.5 *Buret*, 25 mL, automatic gravity fill, all openings sealed to air; for solvent.

4.1.6 *Buret or pipet*, 25 mL; for water.

4.1.7 *Büchner funnels*, for forming slush pulps into pads.

4.1.8 *Glass filter*, coarse, small diameter; and vacuum flask.

Table 1. Characteristics for oil calibrations

Size number	Viscosity range, mPa·s (cP)
50	0.8–3.2
100	3–11
150	7–27
200	19–76
300	48–190

4.1.9 *Vacuum*, source and tubing

4.1.10 *Drying oven*, $105 \pm 2^\circ\text{C}$

4.2 *Bottle technique.*

4.2.1 *Dissolving bottles*, 118-mL (4-oz) flat medicine bottles with plastic screw cap and polyethylene liner or rubber septa caps.

4.2.2 *Glass beads*, approximately 6 mm diameter.

4.2.3 *Mechanical shaker*, such as Burrell wrist action shaker.

4.3 *Copper stirring rod technique.*

4.3.1 *Dissolving tube*, round flat-bottom glass tube, 19 mm (3/4 in.) inside diameter and 150 mm (6 in.) high (see Fig. 1), such as A.O.C.S. color comparison tubes, Kimbel #45290, or equivalent.

4.3.2 *Copper stirring rod*, made from 3.2-mm (1/8-in.) diameter electrolytic copper rod (No. 8 B&S gauge) and shaped as shown in Fig. 1.

4.3.3 *Stirring motor*, capable of operation at 400 ± 25 rpm.

4.4 *Suction device*, such as a pipet bulb.

5. Reagents

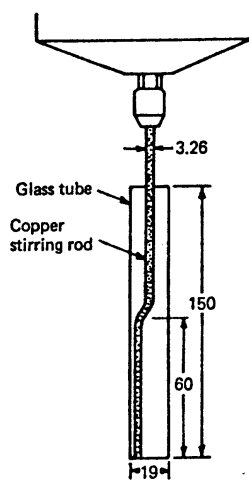
5.1 *Standard viscosity oil*¹, Table 2 lists the viscosity standards available and the approximate viscosity range for each oil.

5.2 *Cupriethylenediamine solution*, $1.0 \pm 0.02M$ in cupric ion and $2.0M$ in ethylenediamine. This solution can be purchased commercially¹ or prepared according to the procedure given in Appendix B. Cupriethylenediamine

¹ Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list in the set of TAPPI Test Methods, or may be available from the TAPPI Quality and Standards Department.

solutions must be stored under nitrogen at all times. *The concentrations of cupric ion and ethylenediamine should be checked as prescribed in Appendix B no less frequently than monthly.*

5.3 Acetone, reagent grade, if used for viscometer cleaning.



(Dimensions in mm)

Fig. 1. Dissolving tube.

Table 2. Standard viscosity oils.

Viscosity standard	Approximate viscosity, mPa·s (cP) at 25 °C*
S3	4.0
S6	8.9
S20	34
S60	120

*Refer to specifications in ASTM D-446.

6. Safety

6.1 Sulfuric acid cleaning solution is a strong acid. Cupriethylenediamine solution is a strong caustic. Both require care in handling. Avoid contact of the solution with skin, eyes, and clothing or breathing its vapor.

6.2 Do NOT bubble nitrogen into the liquid and always control pressure to 2 psi as back pressure can release corrosive CED out of the bottle.