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WORKING GROUP
CHAIR _____ N/A _____

SUBJECT
CATEGORY _____ Pulp Properties _____

RELATED
METHODS _____ See "Additional Information" _____

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)

Five-year review of Official Method T 230 om-2019

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.

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 ± 2°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

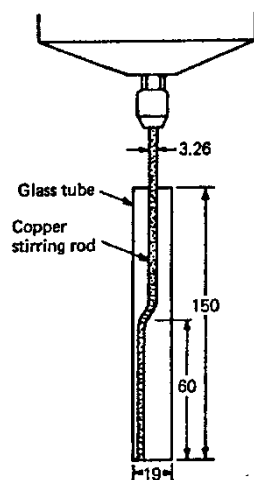
5.1 *Standard viscosity oil*¹, Table 2 lists the viscosity standards available and the approximate viscosity range for each oil.

5.2 *Cupriethylenediamine solution*, 1.0 ± 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

¹Names 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.

7. Sampling and test specimen

Obtain a 5–7-g sample of the pulp and reduce to the air-dried state as follows. Form slush pulp specimens into thin sheets on a Büchner funnel or sheet mold and air dry. Slurry hard, dry specimens of pulp in water by shaking or by mixing in a blender and make into thin sheets. Tear apart the air-dry sheets by hand into small pieces. Thick pulp sheets could also be shredded by means of a clean, coarse file. Do not cut or use a mechanical shredder because the viscosity is likely to be lowered as the result of this process of disintegration. Where possible, the sample used for the actual viscosity measurement should be air-dried and never dried above 60°C.

8. Procedure

8.1 Allow the sample to attain moisture equilibrium in a constant temperature-humidity atmosphere according to TAPPI T 402 “Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets and Related Products.” Determine the moisture content of duplicate 2-g portions of the sample according to TAPPI T 550 “Determination of Equilibrium Moisture in Pulp, Paper, and Paperboard for Chemical Analysis.” Use the average moisture content to calculate the specimen weight of conditioned pulp that must be weighed to give an amount of the sample that is equivalent to 0.1250 or 0.2500 ± 0.0005 g of oven-dry pulp depending on the dissolving technique to be used. *Duplicate specimens must be prepared.*

8.2 Closed bottle procedure.

8.2.1 Weigh into a dissolving bottle containing approximately eight 6-mm glass beads and an amount of air-dry pulp equivalent to 0.2500 g of moisture-free pulp. Add from a buret or pipet exactly 25.00 mL of distilled water, then cap the bottle and shake to disperse in the water. Allow the bottle to stand for about 2 min.

8.2.2 Add exactly 25.00 mL of the cupriethylenediamine solution. Purge with nitrogen for 1 min. Cap the bottle and shake until the fiber is completely dissolved. Usually, a period of 15 min is sufficient.

NOTE 1: Some difficult-to-dissolve pulps require preliminary shaker dispersion with only water or longer shaking time. It has been the experience of some workers that up to 90 min is required. The solution remaining after the viscometer has been filled can be filtered through a glass filter to verify that the pulp has been completely dissolved. The presence of gelatinous residue on the filter indicates that a repeat analysis employing more vigorous dissolving conditions is needed. A third technique for handling difficult-to-disperse pulps is to add $1/5$ the prescribed amount of cupriethylenediamine, agitate until the pulp appears well dispersed, then complete the addition of the cupriethylenediamine.

NOTE 2: The sample preparation procedures give a final solution concentration of 0.5% pulp in 0.5M cupriethylenediamine.

8.2.3 At the end of the shaking time, place the solution bottle on its side for 2 min to allow the solution to degas.

8.3 Copper rod stirring technique.

8.3.1 Weigh into a dissolving tube an amount of air-dry pulp equivalent to 0.1250 g of moisture-free pulp. Add from a buret or pipet exactly 12.50 mL of distilled water and stir gently with the motor-driven copper rod for about 30 s. (See Note 1 for additional information).

8.3.2 After purging the dissolving tube with nitrogen for 1 min, add exactly 12.50 mL of the cupriethylenediamine solution, and stir the mixture for 15 min with the stirrer rotating at approximately 400 rpm. To prevent chances of oxidation the open end of the tube should be continuously flooded with nitrogen.

8.4 Viscosity measurement.

8.4.1 Fill the viscometer by immersing its small-diameter leg into the solution and drawing the liquid into the instrument by applying suction to the other end. Draw the liquid level to the second etch mark. Remove the tube from the solution, wipe the outside clean and return the instrument to a position within 1° of the vertical. Alternatively, use a precision pipette to dispense a charge volume of liquid as noted on the viscometer calibration certificate. Place the

viscometer in the constant temperature bath at $25.0 \pm 0.1^\circ\text{C}$ and allow at least 5 min for the vessel to reach the temperature and the sample to properly drain from the walls of the viscometer fill tube.

NOTE 3: The initial temperature of the solution will affect the time needed for the solution to reach the equilibrium temperature in the bath. Use a kinematic thermometer to verify that the time is adequate for the solution to reach the equilibrium temperature.

8.4.2 Draw the solution up into the measuring leg of the viscometer with a suction bulb, and then allow the solution to drain down to wet the inner surfaces of the viscometer. Determine the efflux time by drawing the liquid above the upper mark and measuring the time required for the meniscus to pass between the two marks. Repeat the measurement of efflux time; the results should check within $\pm 2.0\%$. Poor repeatability can result from incomplete dissolution of the pulp, inadequate temperature control, a dirty viscometer, or impurities in the nitrogen gas.

NOTE 4: The viscometer size is selected to give efflux times of over 100 s, but less than 800 s. In practice, efflux times greater than 350 s should be avoided as reproducible results will be difficult to obtain. To adjust the efflux time, change the size of the viscometer accordingly.

8.5 *Viscometer cleaning.* Drain the CED solution from the viscometer immediately after the viscosity determination is complete. Rinse the tube well with water to remove all traces of the CED solution. Clean each tube with a sulfuric acid based cleaning solution designed for use with laboratory glassware. Soak especially dirty tubes for 24 hours or more to remove all traces of contaminants. Drain all cleaning solution from the tube and rinse well with deionized or distilled water. Viscometer tubes may be dried in two ways, by using a forced air oven or by rinsing with acetone with air drying. With either drying method, traces of moisture or condensation should not be visible in tube after drying.

8.5.1 To dry a viscometer in a forced air oven, place the viscometer in an oven set at $105 \pm 2^\circ\text{C}$. Allow the viscometer tube(s) to remain in the air oven just until all traces of moisture are removed.

8.5.2 To dry a viscometer with acetone, rinse the tube well with reagent grade acetone, drain, and allow the tube to air dry.

NOTE 5: CED solution is highly alkaline and will etch the interior of the viscometer tubes over a period of time. This will change the calibration constant. For this reason, do not allow the CED solution to remain in the tube after the viscosity determination is complete. Drain all CED solution from the tube and rinse it with water to remove all traces of CED solution immediately after the determination is complete.

9. Calculation

Calculate the viscosity, V , of the pulp solution from the formula:

$$V = Ctd$$

where

V = viscosity of cupriethylenediamine solution at 25.0°C, mPa·s (cP)

C = viscometer constant found by calibration

t = average efflux time, s

d = density of the pulp solution, g/cm³ (= 1.052)

10. Report

Report the average result of two determinations in mPa·s to three significant figures, and identify it as the cupriethylenediamine (CED) viscosity of the 0.5% pulp solution by the capillary viscometer method.

11. Precision

11.1 The values of repeatability and reproducibility provided below have been calculated for test results, each of which is an average of two test determinations. The values are based on data obtained in an interlaboratory test program with 18 participants using six materials. Table 3 gives a statistical summary of the data. The footnote to Table 3 lists a description of the materials tested.

11.2 Repeatability (within a laboratory) = 4.0%. The range of average repeatability for all materials in the study was between 1.8 to 6.1% (0.109 to 2.63 mPa·s) with the % repeatability increasing as the grand mean increases.

11.3 Reproducibility (between laboratories) = 19%. The range of average reproducibility for all materials in the study was between 9.6 to 27.2% (0.58 to 11.0 mPa·s).

11.4 The repeatability and reproducibility are in accordance with the definitions of these terms in TAPPI T 1200 "Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility."

Table 3. Statistical summary of data.

<i>Statistical parameter</i>	Sample*					
	<i>A</i>	<i>B</i>	<i>C</i>	<i>D</i>	<i>E</i>	<i>F</i>
Grand mean, mPa·s	6.04	11.18	43.2	15.8	19.6	22.6
Repeatability, mPa·s	0.109	0.327	2.63	0.60	0.95	1.02
Repeatability, %	1.8	2.9	6.1	3.8	4.8	4.5
Reproducibility, mPa·s	0.58	1.38	11.0	3.19	3.65	6.15
Reproducibility, %	9.6	12.3	25.4	20.1	18.6	27.2

*A = sulfite - cellophane grade; B = prehydrolyzed kraft; C = cotton linters;
D = kraft bleached pine; E = kraft semi-bleached pine; F = sulfite paper grade.

12. Keywords

Pulp, Viscosity, Capillarity, Cuene, Cellulose

13. Additional information

13.1 Effective date of issue: To be assigned.

13.2 The 1999 revision includes a change in the requirements for check results for the viscosity measurement. Section 8.4.2 was changed from a “check within ± 0.25 ” to “a check within $\pm 2.0\%$.” This change permits the use of this method on extended lignification pulps which have a wider range of times. The results with other chemical pulps are not significantly affected.

13.3 The falling ball viscometer method, as described in Useful Method 247 “Falling Ball Measurement of Cupriethylenediamine Disperse Viscosity of Pulp,” may be applied as a control procedure, although this is a less reliable procedure. Four laboratories used the falling ball viscometer method in the interlaboratory program and the results showed a repeatability of 10% and a reproducibility of 38%.

13.4 Related methods: PAPTAC G.24, “Cupriethylenediamine Viscosity of Pulp,” Pulp and Paper Technical Association of Canada, Montreal, Canada; SCAN-C 15-16, “Viscosity of Cellulose in Cupriethylenediamine Solution

(CED),” Scandinavian Pulp, Paper and Board Testing Committee, Stockholm, Sweden; ISO Standard 5351/1; ASTM D 446-93, “Standard Specifications and Operating Instructions for Glass Capillary Kinematic Viscometer.”

13.5 The 1966 edition of this method included provisions for delignifying unbleached pulps as a means to extend the scope of the method. Chlorite delignification does attack cellulose and when used in conjunction with this procedure may result in lower viscosity measurements. If delignification is used, the results should be considered only as approximations. It has been noted that pulps above 35 to 40 kappa number should be delignified with a mild treatment of sodium chlorite or chlorine dioxide to obtain meaningful results. A mild treatment will minimize cellulose degradation.

13.6 It has been observed that oxygen pulping and oxygen or ozone bleaching of pulps produces viscosity/strength relationships different from traditionally bleached sulfite and kraft pulps.

13.7 Technical changes in this 2008 edition include updating safety precautions and adding a drying oven to 4.1.10. The change in the 2013 edition was a modification of 8.4.1 to include an alternative method to fill the viscometer, allowing the viscometers to be filled to the appropriate mark using a precision pipette.

Reference

Sihtola, H., Kyrklund, B., Laamanen, L., Palenius, I., “Comparison and Conversion of Viscosity and DP-Values Determined By Different Methods,” *Paperi ja Puu* **45**:225 (1963).

Appendix A. Calibration of viscometers

A.1 The calibration constant, C , for each viscometer is determined by the use of standard viscosity oils of known viscosity and density at $25.0 \pm 0.1^\circ\text{C}$. Depending on the viscometer size and viscosity range in Table 1, select the appropriate standard oil from Table 2.

A.2 Fill the viscometer by immersing its small diameter leg into the oil and drawing the liquid into the instrument by applying suction to the other end. Draw the liquid level to the second etch mark. Remove the tube from the solution, wipe the outside clean and return the instrument to a vertical position within 1° of vertical. Place the viscometer in the constant temperature bath at $25.0 \pm 0.1^\circ\text{C}$ and allow at least 5 min for the vessel to reach the temperature.

NOTE 6: The initial temperature of the solution will affect the time needed for the solution to reach the equilibrium temperature in the bath. Use a kinematic thermometer to verify that the time is adequate for the solution to reach the equilibrium temperature.

A.3 Draw the solution up into the measuring leg of the viscometer with a suction bulb, then allow the oil to drain back into the reservoir to wet the inner surface of the measuring vessel. Determine the efflux time by drawing the liquid above the upper mark and measuring the time required for the meniscus to pass between the reference lines.

Make duplicate measurements; these should check within ± 0.2 s. Failure to obtain the required repeatability is indicative of a dirty viscometer or inadequate temperature control.

A.4 The viscometer constant, C , is calculated from the formula:

$$C = V/t_d$$

where

V = viscosity of standard oil, mPa·s

d = density of standard oil, g/cm³ at 25°C

t = efflux time, s

Appendix B. Preparation of 1M cupriethylenediamine

B.1 Preparation of $\text{Cu}(\text{OH})_2$.

B.1.1 Dissolve 250 g of reagent grade copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in approximately 2000 mL of hot distilled water. Heat to boiling and add reagent grade NH_4OH slowly with vigorous agitation until the solution is faintly alkaline to pH (about 115 mL of NH_4OH are required). Let the precipitate settle, and wash by decantation with 1000-mL portions of distilled water, four times with hot water and twice with cold. Then add sufficient cold water to make the volume of the slurry 1500 mL; cool below 20°C (preferably below 10°C), and add slowly 850 mL of cold 20% NaOH solution with vigorous stirring. Wash the precipitated $\text{Cu}(\text{OH})_2$ with distilled water by decantation until the washings are colorless to phenolphthalein indicator and give no precipitation of sulfate upon addition of BaCl_2 solution.

B.1.2 Cupric hydroxide purchased from chemical reagent suppliers may be used provided it is free from ammonia, chlorides, sulfates, and nitrates, and retains its blue-green color when heated 1 h at 100°C. In this case, 97.5 ± 0.5 g of cupric hydroxide shall be used beginning at step B.2.1. Cupric hydroxide of good quality has a blue-green color. If the sample is not pure, and particularly if it contains ammonia, it will gradually decompose and turn brown due to formation of the oxide. Such a discolored product is not satisfactory for the preparation of the reagent. If the hydroxide contains chlorides, sulfates, or nitrates, the determination of ethylenediamine will give too low results.

B.2 Preparation of cupriethylenediamine (CED) solution.

B.2.1 Transfer the washed $\text{Cu}(\text{OH})_2$ slurries, using sufficient water to make a total volume of 500 mL, to a 1000-mL reagent bottle (see Fig. 2). The bottle is equipped with a rubber stopper carrying two glass tubes, one of which (A) is straight and extends to within approximately 50 mm of the bottom of the bottle, and the other having two side tubes (B and C) at right angles and extending just through the rubber stopper. One of the side tubes (B or C) is connected to a suction source and the other to the nitrogen supply.

B.2.2 Clamp down the rubber stopper, exhaust the air with a laboratory water aspirator and refill the bottle with nitrogen at 14 kPa (2 psi) pressure three separate times. The rubber tubes and pinch clamps attached to the glass

tubes of the solution bottle, as shown in Fig. 2, are used for this purpose. Draw a partial vacuum on the bottle and add 160 mL of 70% ethylenediamine, taking care that no air enters the bottle. This is accomplished by inserting a funnel in the rubber tube attached to the longer glass tube of the solution bottle and opening the pinch clamp just enough to allow the ethylenediamine to be drawn into the bottle. Since considerable heat is evolved at this point, it is desirable to keep cold water running over the bottle during the initial phase of the reaction. After the addition of the ethylenediamine, the gas over the liquid should be alternately evacuated and flushed three times with nitrogen at 14 kPa (2 psi) pressure.

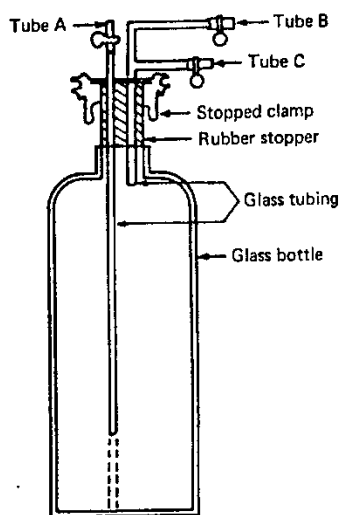


Fig. 2. Solution or stock bottle Cu(En)_2 , viscosity solvent.

B.2.3 Shake the contents of the bottle several times during the course of an hour, and then let stand 12 to 16 h. A clear supernatant liquor will usually be obtained, but if desired, the solution may be filtered through a fritted-glass Büchner funnel, using suction, and again stored under nitrogen.

B.3 *Standardization of Cu(En)_2 solution.*

NOTE 7: Visual observation during standardization of cupriethylenediamine (for cupric ion).

1. Perform the proper dilutions with the CED solution. Prepare and standardize 0.1N thiosulfate solution.
2. Pipet 25 mL of the diluted CED into a 250-mL Erlenmeyer flask. Add 3 grams KI and swirl to dissolve. Add 50 mL 4N sulfuric acid. The solution immediately takes on a dark brown color that quickly changes to a dark greenish-brown color. The color is the result of a precipitate formed in the reaction.
3. To perform the titration, it is necessary to constantly stir the mixture. This is achieved with best results via a magnetic stirrer.
4. The green color remains for about 70-75% of the titration. For example, if an end point of 25.0 mL is expected, the green color should remain until about 17-19 mL of the titrant is consumed. At this point, the mixture takes on a very creamy coffee color, and the effect of adding more of the titrant is like adding more cream to the coffee.

5. After addition of about 3-4 more mL, a mauve color will appear with each drop and quickly fade. The mixture remains a very light creamy color. At this point, the starch should be added. If the starch is not added, the mixture will become completely mauve, and it is extremely difficult to see the end point.
6. Addition of the starch results in a creamy purple color. The purple color should fade with the addition of more titrant. Only about 0.3-0.5 mL of titrant should be needed after the addition of the starch. This should be the point right before the end point.
7. Add 10 mL of 20% ammonium thiocyanate. The mixture becomes purple again. The titration should be carried out until the mixture fades to a mauve color and finally becomes white at the end point. However, the end point may appear as a salmon pink color with the white color of the precipitate developing only upon standing for a few minutes.

B.3.1 *Cupric ion concentration (Y).*

B.3.1.1 Pipet out a 25 mL sample of CED from a jar in the cold room and dilute to 250 mL in a volumetric flask. (Note: Save sample for Step B.3.2.)

B.3.1.2 Pipet out a 25 mL aliquot to a 250 mL flask.

B.3.1.3 Add 3 grams of KI (30 mL of 10% KI solution).

B.3.1.4 Add 50 mL of 4N H₂SO₄ to acidify.

B.3.1.5 Titrate with 0.1N thiosulfate near to the starch end point (add starch indicator at a proper time).

B.3.1.6 Just before the starch end point, add 10 mL of 20% ammonium thiocyanate solution to intensify the end point. (Note: 20% solution preparation: Dissolve 10 g of thiocyanate crystal to 50 mL of distilled H₂O.)

B.3.1.7 Continue to titrate to the starch end point.

B.3.1.8 Calculate cupric ion concentration (Y): $Y = 0.04$ (mL of 0.1N thiosulfate in mL consumed).

B.3.2 *Ethylenediamine concentration (X).*

B.3.2.1 Pipet out 25 mL of CED solution prepared in Step A.1 above to a 250 mL beaker.

B.3.2.2 Add 75 mL of distilled water.

B.3.2.3 Titrate to pH 3.0 using a pH meter with standard 1.0N H₂SO₄ while stirring.

B.3.2.4 Calculate ethylenediamine concentration (X): $X = 0.2$ (volume of 1N H₂SO₄ in mL consumed) – Y (from B.3.1.8).

B.3.3 *Ratio ethylenediamine/cupric ion.*

Calculate the ethylenediamine and the cupric ion concentration and their ratio as follows:

$$X = [N_1a - 2N_2b] / [(2 \cdot V_1 \cdot V_3) / V_2]$$

$$Y = [N_2b] / [(V_1 \cdot V_3) / V_2]$$

$$R = X/Y$$

where

a = volume of sulfuric acid solution consumed, mL

b = volume of sodium thiosulfate solution consumed, mL

V_1 = volume of sample solution used for initial dilution, mL

V_2	=	volume of sample dilution, mL
V_3	=	volume of diluted solution used for titration, mL
N_1	=	normality of sulfuric acid solution
N_2	=	normality of sodium thiosulfate solution
X	=	mole ethylenediamine per 1000 mL of solution
Y	=	mole cupric ion per 1000 mL of solution
R	=	ratio ethylenediamine/cupric ion

B.3.4 *Dilution.* The ratio R must be 2.00 ± 0.04 and the cupric ion concentration $1.00 \pm 0.02M$. If the ratio exceeds 2.00:1, add fresh $\text{Cu}(\text{OH})_2$ and repeat the agitation and standardization of the solution as given above. If the ratio falls below 1.92:1, start with fresh $\text{Cu}(\text{OH})_2$ and increase the volume of ethylenediamine accordingly.

B.3.5 The solution should be stable for several months if stored under nitrogen. There is usually evidence of a small deposit of cuprous oxide after the solutions have been stored for varying periods of time. Usually this has a negligible influence on the copper and the solutions need not be discarded if the copper analysis checks within the required limits.

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department.

