



# Standard Test Method for Measurement of Average Viscometric Degree of Polymerization of New and Aged Electrical Papers and Boards<sup>1</sup>

This standard is issued under the fixed designation D 4243; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method describes a standard procedure for determining the average viscometric degree of polymerization (abbreviated  $\overline{DP}_v$ ) of new or aged electrical papers. The determination is made by measuring the intrinsic viscosity of a solution of the paper in an appropriate solvent.

1.2 The degree of polymerization (or the degree of condensation) of a particular cellulose molecule is the number of anhydro- $\beta$ -glucose monomers,  $C_6H_{10}O_5$ , in the cellulose molecule. Within a sample of paper, not all the cellulose molecules have the same degree of polymerization so that the mean value measured by viscometric methods is not necessarily the same as that which may be obtained by other methods.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.* See Section 9

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)<sup>2</sup>

D 1711 Terminology Relating to Electrical Insulation<sup>3</sup>

### 2.2 Other Document:

IEC Publication 450 Measurement of the Average Viscometric Degree of Polymerization of New and Aged Electrical Papers<sup>4</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-9 on Electrical and Electronic Insulating Materials and is the direct responsibility of Subcommittee D09.19 on Dielectric Sheet and Roll Products.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 10.03.

<sup>3</sup> Annual Book of ASTM Standards, Vol 10.01.

<sup>4</sup> Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1711.

## 4. Summary of Test Method

4.1 This test method measures the specific viscosity of a solution of the paper in cupriethylene-diamine. From this measurement the intrinsic viscosity of the solution is deduced, and from this the degree of polymerization is easily calculated.

4.2 This test method follows very closely the procedures specified in IEC Publication 450.

## 5. Significance and Use

5.1 This test method may be applied to all papers made from unmodified cellulose, as used in transformer, cable, or capacitor manufacture. It applies to new or aged papers. For information, Appendix X2 shows an example of statistical distribution of  $\overline{DP}_v$  values for new papers intended for the insulation of transformers, together with information relative to cable and capacitor papers. Nevertheless, where evaluating the decomposition stage of aged papers, take care to use, as a reference, the  $\overline{DP}_v$  value of the new paper of the very same origin;  $\overline{DP}_v$  of new papers being a function, among other factors, of their specific gravity and of their manufacturing process.

5.2 This test method can also be used for the determination of the intrinsic viscosity of solutions of chemically modified papers, provided that these dissolve completely in the selection solvent. This test method should be used with caution when it is applied to papers with mineral fillers.

## 6. Interferences

6.1 Lignins, that are present in measurable amounts in most papers and boards, may have an effect on the test results, depending upon concentration and composition. For this reason, it is important in aging studies to use as a reference samples of the unaged paper as mentioned in 5.1.

6.2 Under some conditions of heat and atmosphere, cross linking of cellulose molecules may occur, resulting in erratic test values. This effect has been observed for capacitor tissue in

vacuum at temperatures as low as 110°C and for other papers aged in air at higher temperatures.

## 7. Apparatus

### 7.1 Apparatus for Solution:

7.1.1 *Round-Bottomed 50-mL Flask*, preferably with a short narrow neck, or a narrow-necked 50-mL Erlenmeyer flask.

7.1.2 *Rubber Stopper*, fitting the neck of the flask, through which passes a capillary tube fitted with a small-bore cock glass cock; or a ground stopper, fitted with a small-bore cock may be used with a ground-neck flask.

7.1.3 *Glass Balls*, 4 to 6-mm diameter, that shall not be able to enter the bore of the cock.

7.1.4 *Mechanical Stirrer*, to rotate the solution flask with a uniform circular motion with a horizontal axis between 20 and 40 r/min. The flask shall be mounted so that its axis is normal to the axis of rotation, and the radius of gyration shall not be greater than 200 mm.

### 7.2 Apparatus for Measurement of Viscosity:

7.2.1 *Apparatus for Measurement of Kinematic Viscosity*, as described in Test Method D 445. The viscometer shall have a calibration constant,  $C$ , of from 0.00010 or 0.00013 St/s ( $10 \times 10^{-9}$  to  $13 \times 10^{-9}$  m<sup>2</sup>/s<sup>2</sup>).

7.2.2 This constant shall be determined by measuring the efflux-time  $T$  (seconds) of a liquid of known dynamic viscosity (Ns/m<sup>2</sup>) and density  $\rho$  (g/cm<sup>3</sup>). It is given by the formula:

$$C = \frac{\eta}{\rho \cdot T} \quad (1)$$

7.2.3 *Constant-Temperature Water Bath*, regulated at  $20 \pm 0.1^\circ\text{C}$ .

7.2.4 *Stopwatch*, with an accuracy of 0.1 s.

7.3 *Apparatus for Measurement of Water Content of Paper Sample:*

7.3.1 *Weighing Containers*, impermeable to water vapor, with airtight lids.

7.3.2 *Ventilated Drying Oven*, thermostatically controlled at  $105 \pm 2^\circ\text{C}$ .

7.3.3 *Desiccator*.

## 8. Reagents

### 8.1 Cupriethylene-Diamine Solution:

8.1.1 The formula ascribed to cupriethylene-diamine (CED) is:



This implies a molar ratio of 2 between the concentration of ethylene-diamine and the concentration of copper:

$$(C_{\text{ED}}/C_{\text{cu}}) = 2 \quad (3)$$

8.1.2 Cupriethylene-diamine solution may be purchased commercially at several different concentrations. At a concentration greater than 1  $M$  it may be kept for one year in the dark. It is diluted to 1  $M$  when required for use. Alternatively the CED solution can be made in the laboratory at its working strength of 1  $M$  by the methods described in Annex A1.

8.1.3 The 1  $M$  solution will keep only for a limited time. As often as necessary the solution should be checked by:

8.1.3.1 Using the method described in Annex A2 to verify that the ratio

$$C_{\text{ED}}/C_{\text{Cu}} = 2.0 \pm 0.1. \quad (4)$$

8.1.3.2 Verify that there is no precipitate in the solution. Any precipitate should be removed by filtering or by decanting.

## 9. Preparation of Specimens

### 9.1 Impregnated Papers:

9.1.1 Using a Soxhlet extractor, degrease the paper with hexane or, if necessary, with chloroform.

9.1.1.1 **Caution**—Chloroform is toxic, and hexane is flammable. Adequate precautions must be taken to avoid exposure to vapors and to prevent fire.

9.1.2 Allow the solvent to evaporate in air at room temperature.

9.1.3 Cut the sample into small pieces (1 or 2 mm<sup>2</sup>) with scissors, using gloves to avoid touching the paper.

9.1.4 Keep the sample in a controlled-humidity atmosphere until it reaches equilibrium water content before removing the material required for test purposes.

### 9.2 Nonimpregnated Papers:

9.2.1 Cut the sample into small pieces (1 or 2 mm<sup>2</sup>) with scissors, using gloves to avoid touching the paper.

9.2.2 Keep the sample in a controlled-humidity atmosphere until it reaches equilibrium water content before removing the material required for test purposes.

## 10. Procedure

### 10.1 Determination of Viscosity:

10.1.1 *Test Specimen*—Weigh to the nearest 0.1 mg an amount ( $m$ ) of paper, in equilibrium with the controlled atmosphere, of about:

10.1.1.1 125 mg when  $\overline{DP}_v$  lies between 100 and 300,

10.1.1.2 50 mg when  $\overline{DP}_v$  lies between 300 and 700, and

10.1.1.3 25 mg when  $\overline{DP}_v$  lies between 700 and 1500.

#### 10.1.2 Solution:

10.1.2.1 Put a few glass balls in the flask.

10.1.2.2 Add the weighed test specimen.

10.1.2.3 Carefully shake or stir by hand to ensure the small pieces of paper are separated and evenly distributed.

10.1.2.4 Add 22.5 mL of distilled water.

10.1.2.5 Again shake or stir by hand to disintegrate and wet all the paper completely.

10.1.2.6 Leave for ½h to allow thorough impregnation with water.

10.1.2.7 Add 22.5 mL of 1  $M$  solution of cupriethylene-diamine.

10.1.2.8 Add a number of glass balls so that, when the stopper with capillary tube, and so forth, is fitted, the liquid will entirely fill the flask and reach the bore of the cock. Do not allow any pieces of paper to enter the capillary, for such paper will not be dissolved.

10.1.2.9 Close the cock.

**10.1.2.10** Allow the specimen to dissolve with mechanical stirring as described in 7.1.4. The preferred solution time is 2 h; however, when the specimen is not satisfactorily dissolved, the solution time may be increased to a maximum of 18 h. For a specimen of an aged paper, there is little risk of further measurable degradation due to this increase of solution time.

10.1.3 *Measurement of Viscosity:*

10.1.3.1 After the period of stirring and before filling the viscometer, allow the solution to stand for 1 h at the temperature of measurement,  $20 \pm 0.1^\circ\text{C}$ .

10.1.3.2 Take the required volume of solution and transfer it to the viscometer reservoir. Assemble the viscometer and place the assembly in the thermostatic bath.

10.1.3.3 Wait 15 min before beginning the measurements. Then raise the liquid level above the upper mark of the viscometer either by means of air pressure or slight vacuum. Measure the efflux time between the upper and lower marks. Make three successive measurements. The difference between the maximum and minimum time of flow shall be less than 0.5 s. If this repeatability is not achieved, clean the viscometer as described in 10.1.4. Take a second specimen of the solution that has been kept at the measurement temperature and make a further series of three measurements. If again the difference is not less than 0.5 s, take a new specimen of paper and prepare a fresh solution.

10.1.3.4 Under the same conditions, measure the flow time of the solvent. This shall be between 90 and 110 s for the viscometer constant  $C$  stated in 6.2 ( $0.00010 \leq C \leq 0.00013$ ).

#### 10.1.4 *Cleaning the Viscometer:*

10.1.4.1 By aspiration, fill the tube with chromic-acid mixture and immediately empty it again.

10.1.4.2 Rinse thoroughly with distilled water.

10.1.4.3 Rinse twice with acetone or alcohol.

10.1.4.4 Dry by means of compressed gas from a cylinder or dust-and-oil-free compressed air.

10.1.5 Make all tests in duplicate, making up two separate solutions from separately weighed specimens.

#### 10.2 *Determination of Water Content of Paper:*

##### 10.2.1 *Test Specimen:*

10.2.1.1 Weigh to the nearest milligram approximately 2 g of paper in equilibrium with the controlled-humidity atmosphere, and in a closed weighing container that has been dried and tared.

10.2.1.2 If the paper sample is too small for 2 g to be reserved for determining its water content, use a smaller specimen. However, if the amount of paper is only sufficient for the viscometric measurements, its water content may be deduced from the moisture equilibrium curves and the relative humidity of the conditioning atmosphere with which the paper was in equilibrium. If equilibrium curves are not available for the particular paper tested, the following average values may be used as a first approximation for temperatures between 20 and  $27^\circ\text{C}$ .

Relative Humidity of Air, %	Water Content of Air, %
50	6
65	8

##### 10.2.2 *Measurement of Water Content:*

10.2.2.1 After weighing, open the container and place it, with the weighed test specimen and its cap, in the oven and heat at  $105 \pm 2^\circ\text{C}$  until it reaches constant mass. This will normally require from 4 to 18 h.

10.2.2.2 After drying, replace the cap on the container and allow it to cool in a desiccator for about 45 min. After cooling, equalize the air pressures inside and outside the container by raising and lowering the cap quickly. Weigh the container with the specimen.

10.2.3 Alternative procedures may be used to determine the water content of the paper sample provided that it can be verified that the procedure used will give results that differ from the results used in 10.2.2 by no more than 0.5 %.

## 11. Calculation

### 11.1 *Concentration, $c$ , of Dry Paper in the Solution:*

11.1.1 The water content  $H$  of the paper, as a proportion of the dry weight, is as follows:

$$H = (M - M_0/M_0) \quad (5)$$

where:

$M$  = mass of the test sample used for determining the water content before drying, g and

$M_0$  = mass of this sample after drying, g.

11.1.2 Knowing the mass,  $m$ , in grams of the test specimen and its water content,  $H$ , the concentration  $c$  (g/100 mL), of dry paper in the solution is given by:

$$c = (m \cdot 100/45) \cdot (1/1 + H) \quad (6)$$

11.2 *Specific Viscosity,  $\eta_s$* —The densities of the solvent and of the solution are practically identical. Thus, the specific viscosity  $\eta_s$  as defined in 4.2 is given by:

$$\eta_s = \frac{T_s - T_0}{T_0} \quad (7)$$

where:

$T_0$  = mean efflux time of the solvent, and

$T_s$  = mean efflux time of the solution.

### 11.3 *Intrinsic Viscosity $[\eta]$ :*

11.3.1 The intrinsic viscosity is calculated using Martin's formula from the specific viscosity  $\eta_s$  and the concentration  $c$  (g/100 mL), of dry paper.

11.3.2 As Martin's formula does not readily lend itself to calculation of  $\eta$ , use Table 1, which contains values of the product  $[\eta] \cdot c$  as a function of  $[\eta_s]$ .

11.3.3 In Table 1,  $k = 0.14$ , which value has been found experimentally for the operating conditions described.

11.4 *Degree of Polymerization  $\overline{DP}_v$* —The average viscometric degree of polymerization  $\overline{DP}_v$  is given by the equation:

$$\overline{DP}_v^\alpha = [\eta]/K \quad (8)$$

where:

$\alpha = 1$ , and

$K = 7.5 \times 10^{-3}$ .

## 12. Validity of Results

12.1 Two conditions should be met if the results are to be considered significant:

12.1.1 Both values  $\overline{DP}_v$  should agree within 2.5 % of their mean value.

12.1.2 After measuring the viscosity, filter the solution through a fine-sintered glass filter; wash the filter and any deposit with hydrochloric acid (diluted with one part water to three parts acid), then with distilled water; dry in an oven of about  $110^\circ\text{C}$ , allow to cool in a desiccator, and weigh. The mass of any insoluble residue shall be less than 5 % of the initial weight of the specimen.

12.2 If one or both of these conditions are not met, a further series of tests shall be made. If, again, one or both requirements

**TABLE 1 Numerical Values of the Product  $[\eta] \cdot C$  as a Function of  $[\eta_s]$  According to Martin's Formula ( $k = 0.14$ )<sup>A</sup>**

$\eta_s$	$\eta \cdot C$									
	0.00	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
0.0		0.010	0.020	0.030	0.039	0.049	0.059	0.068	0.078	0.087
0.1	0.097	0.106	0.116	0.125	0.134	0.143	0.152	0.161	0.170	0.179
0.2	0.188	0.197	0.206	0.215	0.223	0.232	0.241	0.249	0.258	0.266
0.3	0.275	0.283	0.291	0.300	0.308	0.316	0.324	0.332	0.340	0.349
0.4	0.357	0.365	0.372	0.380	0.388	0.396	0.404	0.412	0.419	0.427
0.5	0.435	0.442	0.450	0.457	0.465	0.472	0.480	0.487	0.495	0.502
0.6	0.509	0.516	0.524	0.531	0.538	0.545	0.552	0.559	0.566	0.574
0.7	0.581	0.588	0.594	0.601	0.608	0.615	0.622	0.629	0.636	0.642
0.8	0.649	0.656	0.662	0.669	0.676	0.682	0.689	0.695	0.702	0.708
0.9	0.715	0.721	0.728	0.734	0.740	0.747	0.753	0.759	0.766	0.772
1	0.778									

  

$\eta_s$	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
1	0.78	0.84	0.90	0.96	1.01	1.06	1.12	1.17	1.22	1.26
2	1.31	1.36	1.40	1.44	1.49	1.53	1.57	1.61	1.65	1.68
3	1.72	1.76	1.79	1.83	1.86	1.90	1.93	1.96	2.00	2.03
4	2.06	2.09	2.12	2.15	2.18	2.21	2.24	2.26	2.29	2.32
5	2.35	2.37	2.40	2.43	2.45	2.48	2.50	2.53	2.55	2.57
6	2.60	2.62	2.64	2.67	2.69	2.71	2.73	2.76	2.78	2.80
7	2.82	2.84	2.86	2.88	2.90	2.92	2.94	2.96	2.98	3.00
8	3.02	3.04	3.06	3.08	3.10	3.11	3.13	3.15	3.17	3.19
9	3.20	3.22	3.24	3.26	3.27	3.29	3.31	3.32	3.34	3.36
10	3.37									

<sup>A</sup> If the conditions imposed in Section 10 are complied with,  $\eta_s$  shall remain below 1. The values of  $\eta \cdot c$  when  $\eta_s > 1$  are given for information only.

are not complied with, the values obtained are considered to be results, but the test report shall state that the above validity requirements have not been met.

### 13. Report

13.1 The test report shall include the following information:

13.1.1 *Information Regarding Test Sample:*

13.1.1.1 *Origin*—Report whether the sample is new or aged (taken from service) with a statement, if required, of the exact location from which the sample was removed.

13.1.1.2 *Condition*—State whether the sample is impregnated or not impregnated and the nature of impregnation.

13.1.2 Extraction solvent.

13.1.3 Water content of the paper as determined by the method in 10.2. (State if necessary, that this determination could not be made through lack of a sufficient sample).

13.1.4 Characteristics of the CED solution, origin, and ratio  $C_{ED}/C_{Cu}$ .

13.1.5 Weight of specimens.

13.1.6 Solution time.

13.1.7 Mean efflux times of the solvent and solution through the viscometer tube.

13.1.8 Individual values of  $\overline{DP}_v$  in the two tests and the mean value  $\overline{DP}_v$ .

13.1.9 Difference between these two values as a percentage of the mean  $\overline{DP}_v$ .

13.1.10 Temperature at which the viscosity was measured.

13.1.11 Amount of insoluble residue, as a percentage of the specimen mass.

13.1.12 Whether or not the requirements of Section 12 have been met.

### 14. Precision and Bias

14.1 *Precision:*

14.1.1 Within-laboratory results of tests on specimens from the same sample should agree within 2.5 %, as stated in 12.1.1.

14.1.2 Two between-laboratory tests, with one of three laboratories participating in both tests, and the other two laboratories in one test each, gave the following results:

14.1.2.1 Testing nine samples of new pressboard with  $\overline{DP}_v$  values between 1040 and 1120, the average absolute difference between laboratories was 25, and the maximum difference was 71. The difference between grand averages for the two laboratories was 5.

14.1.2.2 Testing six samples of kraft paper which had been thermally aged for different periods of time, and which had  $\overline{DP}_v$  values between 560 and 970, the average absolute difference between laboratories was 6, and the maximum difference was 14. The difference between grand averages for the two laboratories was 2.

14.1.3 In both sets of tests reported in 14.1.2, each of the laboratories made only a single determination on each sample.

14.2 *Bias*—No statement of bias can be made due to the unavailability of the standard sample.

### 15. Keywords

15.1 aging; board; cellulose; cupriethylene-diamine; degree of polymerization; paper; pressboard; viscometry

**(Mandatory Information)**
**A1. PREPARATION OF THE CUPRIETHYLENE-DIAMINE SOLUTION**
**A1.1 Reagents**

A1.1.1 *Crystallized Copper Sulfate* ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ), analytical grade.

A1.1.2 *Ammonium Hydroxide Solution*, ACS reagent grade.

A1.1.3 *Sodium Hydroxide Solution* ( $\text{NaOH}$ ), 8 %.

A1.1.4 *Barium Chloride Solution*—Dissolve 7 g  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  in 1 L of distilled water.

A1.1.5 *Acetone* ( $\text{CH}_3\text{COCH}_3$ ).

A1.1.6 *Anhydrous Sodium Sulfate* ( $\text{Na}_2\text{SO}_4$ ).

A1.1.7 *Hydrochloric Acid* ( $\text{HCl}$ ), 1 *M*.

A1.1.8 *Potassium Iodide Solution* ( $\text{KI}$ ), 10 %.

A1.1.9 *Sodium Thiosulfate* ( $\text{Na}_2\text{S}_2\text{O}_3$ )—Standard 0.05 *M* solution.

A1.1.10 *Indicator*—0.2 % solution of soluble starch, prepared fresh daily.

A1.1.11 *Commercial Ethylene-Diamine Solution* ( $\text{NH}_2\text{CH}_2\text{CH}_2\text{NH}_2$ ), (about 70 %).

A1.1.12 *Sulfuric Acid* ( $\text{H}_2\text{SO}_4$ ), 0.5 *M*.

**A1.2 Preparation of the Copper Hydroxide**

A1.2.1 Dissolve 200 g of crystallized copper sulfate in 1 L of boiling distilled water. Allow to cool to a temperature of about 45°C. Add ammonium hydroxide slowly until a blue-violet color appears. Do not add more ammonium hydroxide or the precipitate will dissolve in the surplus of reagent. About 100 mL of ammonium hydroxide will be required. Wash the greenish precipitate of basic copper sulfate with distilled cold water by decantation, until the washings are colorless.

A1.2.2 Add 640 mL of the sodium hydroxide solution drop by drop to the moist precipitate of basic copper sulfate. Stir carefully. The temperature shall not exceed 20°C and should preferably be kept below 10°C. Allow to stand for 10 min. Wash the copper hydroxide precipitate with distilled water. Stop washing when some drops of barium chloride solution no longer produce a precipitate of  $\text{BaSO}_4$  in the washings. Shake the moist hydroxide with 1000 mL of acetone that has been dehydrated by passing through dry sodium sulfate. Filter through a Büchner funnel, and wash again on the filter with 100 mL of acetone. Dry either in the air or in a vacuum, at room temperature.

A1.2.3 Blue copper hydroxide prepared this way is free from black copper oxide and agrees with the theoretical formula  $\text{Cu}(\text{OH})_2$ . It shall dissolve in hydrochloric acid, concentrated ammonia, or ethylene-diamine without leaving any insoluble residue, and it shall be free from either sulfate or sodium ions. It shall be kept away from light and in a brown glass bottle with a ground stopper.

A1.2.4 *Determination of the Copper Content:*

A1.2.4.1 In a 200-mL volumetric flask, dissolve 2 g of copper hydroxide in 50 mL of 1 *M* hydrochloric acid. Fill up to the mark with the same acid. Pipet out 25 mL from this solution, transfer into a titration flask, and add 25 mL of the 10 % potassium iodide solution.

A1.2.4.2 Titrate with sodium thiosulfate, using the starch solution as the indicator (1 to 2 mL of the starch solution is added when the titration is nearing the end).

A1.2.4.3 The copper content, expressed in grams per 100 g of hydroxide, is

$$n \times 0.04 \times 63.5 \quad (\text{A1.1})$$

where:

$n$  = number of millilitres of 0.05 *M* sodium thiosulfate solution used for the titration.

**A1.3 Preparation of the Ethylene-Diamine Solution**

A1.3.1 Ethylene-diamine  $\text{NH}_2\text{-CH}_2\text{-CH}_2\text{NH}_2$  is sold commercially as a 70 % solution (see A1.1.11). It is necessary, to ensure the stability of the cupriethylene-diamine solution that will be used to dissolve the paper, to start from a pure reagent. The commercial solution can be purified by distillation at atmospheric, or better, at a slightly reduced pressure. If, with time, the solution turns yellow, it should be redistilled.

A1.3.2 *Determination of the Ethylene-Diamine Content:*

A1.3.2.1 Take 25 mL of ethylene-diamine, transfer to a 250-mL volumetric flask, and fill up to the mark with distilled water. Pipet out 20 mL from this solution and titrate with 0.5 *M* sulfuric acid solution, using methyl orange as the indicator (see A1.1.12). The ethylene-diamine content, in g/100 mL of the original solution, is:

$$n \times 1.5 \quad (\text{A1.2})$$

where:

$n$  = number of millilitres of 0.5 *M*  $\text{H}_2\text{SO}_4$  used. Keep the ethylene-diamine solution away from light, in a brown glass bottle with a ground stopper.

**A1.4 Preparation of the Cupriethylene-Diamine Solution**

A1.4.1 Tests are carried out with 1 *M* cupriethylene-diamine solution (217.50 g CED/L). Weigh an amount of copper hydroxide precipitate corresponding to  $63.5 \pm 0.5$  g copper. Transfer to a 1-L volumetric flask. Moisten with about 50 mL of distilled water. Using a burette, introduce into the flask a volume of ethylene-diamine solution containing  $120 \pm 0.5$  g anhydrous ethylene-diamine. Keep very cool in order to maintain the temperature below 10°C. Allow the solution to stand for 1 h at room temperature (shake from time to time). Fill up to the mark with distilled water and shake. Allow to settle for 24 h, and filter through a fine-sintered glass filter.

## A2. CHECKING THE RATIO $C_{ED}/C_{Cu}$ OF THE CUPRIETHYLENE-DIAMINE SOLUTION

### A2.1 Reagents

A2.1.1 Potassium Iodide Solution (KI), 10 %.

A2.1.2 Sodium Thiosulfate ( $Na_2S_2O_3$ )—Standard 0.1 M solution.

A2.1.3 Sulfuric Acid ( $H_2SO_4$ ), 0.5 M.

A2.1.4 Sulfuric Acid ( $H_2SO_4$ ), 2 M.

### A2.2 Copper Content:

A2.2.1 Dilute 25 mL of the 1 M cupriethylene-diamine solution to 250 mL, with distilled water, in a 250-mL volumetric flask. Pipet out 25 mL, transfer to a titration flask, add 50 mL of the 10 % potassium iodide solution (see A2.1.1). Acidify with 100 mL of 2 M sulfuric acid (see A2.1.4). Titrate with the sodium thiosulfate solution (see A2.1.2). The copper molarity of the solution is:

$$p = n \cdot 0.04 \quad (A2.1)$$

where:

$n$  = sodium thiosulfate solution used, mL.

### A2.3 Ethylene-Diamine Content

A2.3.1 Take 20 mL of the diluted solution and titrate with 0.5 M sulfuric acid until the methyl orange used as the indicator shows a faint pink coloration (see A2.1.3).

A2.3.2 Part of the acid used reacts with copper hydroxide.

A2.3.3 The ethylene-diamine molarity of the solution is given by:

$$(q - 4p) \cdot 0.25 \quad (A2.2)$$

where:

$q$  = number of millilitres of 0.5 M  $H_2SO_4$  used, and

$p$  = copper-hydroxide molarity as determined previously.

A2.3.4 If the symbols  $C_{Cu}$  and  $C_{ED}$  are used for the copper- and ethylene-diamine molarities, then the ratio  $C_{ED}/C_{Cu}$  should be  $2 \pm 0.1$ .

## APPENDIX

### (Nonmandatory Information)

#### X1. VISCOSITY CALCULATIONS

X1.1 The specific viscosity  $\eta_s$  is:

$$\eta_s = \frac{\text{viscosity of paper solution} - \text{viscosity of solvent}}{\text{viscosity of solvent}} \quad (X1.1)$$

X1.2 The intrinsic viscosity  $[\eta]$  is:

$$\lim_{c \rightarrow 0} \frac{\eta_s}{c} \quad (X1.2)$$

where:

$c$  = concentration of the solution, g/100 mL.

X1.3 The average viscometric degree of polymerization  $\overline{DP}_v$  (the ratio of the mean molecular mass indicated viscometrically to the molecular mass of the monomeric unit) is related to the intrinsic viscosity by the equation:

$$[\eta] = K \cdot \overline{DP}_v^\alpha \quad (X1.3)$$

where:

$K$  and  $\alpha$  = characteristic coefficients of the polymer-solvent system (paper, cupriethylene-diamine) and of the monomer.

X1.4 The intrinsic viscosity  $[\eta]$  is calculated from the specific viscosity  $\eta_s$  and the concentration  $c$  by Martin's empirical formula:

$$\eta_s = [\eta] \cdot c \cdot 10^{k[\eta]c} \quad (X1.4)$$

NOTE X1.1—Solutions of cellulose are non-Newtonian fluids (their viscosity decreases as the flow velocity increases, sometimes known as solutions varies only slightly with the gradient of the velocity modulus, it is necessary to comply strictly with the conditions stated in this method to ensure maximum accuracy (the gradient of the velocity modulus shall not exceed  $500 \text{ s}^{-1}$ ).

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