Designation: D4243 – 23

Standard Test Method for Measurement of Average Viscometric Degree of Polymerization of New and Aged Electrical Papers and Boards[1]

This standard is issued under the fixed designation D4243; 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 (ε) 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-β-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 are obtained by other methods.

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* See Section 9.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D1711 Terminology Relating to Electrical Insulation

2.2 *Other Document:*

IEC 60450 Measurement of the Average Viscometric Degree of Polymerization of New and Aged Electrical Papers[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1711.

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 60450.

5. Significance and Use

5.1 This test method applies 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 X1 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. Use this test method with caution when it is applied to papers with mineral fillers.

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[1] This test method is under the jurisdiction of ASTM Committee D09 on Electrical and Electronic Insulating Materials and is the direct responsibility of Subcommittee D09.01 on Electrical Insulating Products.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

6. Interferences

6.1 Lignins, that are present in measurable amounts in most papers and boards, 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 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 or Flat-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 is suitable for use with a ground-neck flask.

NOTE 1—In lieu of a small-bore cock a capillary tube with constant nitrogen flow may be used. The use of constant nitrogen flow requires both an inlet and an outlet capillary to avoid pressure build-up in the flask.

7.1.3 *Glass Balls,* 4 mm 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 D445. The viscometer shall have a calibration constant, *C*, of from 0.00010 or 0.00013 St/s (10×10^{-9} to 13×10^{-9} m^2/s ^{2}).

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

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

7.2.3 *Constant-Temperature Water Bath,* regulated at 20 °C ± 0.1 °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 °C ± 2 °C.

7.3.3 *Desiccator.*

8. Reagents

8.1 *Cupriethylene-Diamine Solution:*

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

$$[Cu(NH_2 - CH_2 - CH_2 - NH_2)_2](OH)_2 \qquad (2)$$

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

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

8.1.2 Cupriethylene-diamine solution is available for purchase commercially at several different concentrations. At a concentration greater than 1 *M* it is suitable to 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 check the solution by:

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

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

8.1.3.2 Verify that there is no precipitate in the solution. Remove any precipitate 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. (**Warning—**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 mm^2 or 2 mm^2) with scissors, using gloves to avoid touching the paper.

NOTE 2—Optionally, to assist in the separation of fibers the specimen may be fluffed in a suitable blender or grinder. Care shall be taken to ensure any temperature increase caused by the fluffing process does not negatively affect the specimen.

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 mm^2 or 2 mm^2) with scissors, using gloves to avoid touching the paper.

NOTE 3—Optionally, to assist in the separation of fibers the specimen may be fluffed in a suitable blender or grinder. Care shall be taken to ensure any temperature increase caused by the fluffing process does not negatively affect the specimen.

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 between 20 mL and 25 mL, measured to the neared 0.1 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 ½ h to allow thorough impregnation with water.

10.1.2.7 Add the same volume (±0.1 mL) of 1 *M* solution of cupriethylene-diamine that was previously added of distilled water.

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 or initiate nitrogen flow.

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, increase 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 °C ± 0.1 °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 s 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 is 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 are used as a first approximation for temperatures between 20 °C and 27 °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 °C ± 2 °C until it reaches constant mass. This will normally require from 4 h 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 are suitable for use 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:

$$H = (M - M_0/M_0) \tag{5}$$

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:

$$c = (m \cdot 100/45) \cdot (1/1+H) \tag{6}$$

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

$$\eta_s = \frac{T_s - T_0}{T_0} \tag{7}$$

where:

T_0 = mean efflux time of the solvent, and
T_s = mean efflux time of the solution.

11.3 *Intrinsic Viscosity* [η]*:*

11.3.1 The intrinsic viscosity is calculated using Martin's formula from the specific viscosity η_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 η, use Table 1, which contains values of the product [η] · *c* as a function of [η_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:

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

where:

α = 1, and
K = 7.5×10^{-3}.

12. Validity of Results

12.1 Meet the following two conditions if the results are to be considered significant:

12.1.1 Both values $\overline{DP}_v$ 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 °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 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 was not 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 Volume of distilled water and CED solution.

13.1.7 Solution time.

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

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

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

13.1.11 Temperature at which the viscosity was measured.

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

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

14. Precision and Bias

14.1 *Precision:*

TABLE 1 Numerical Values of the Product [η] · C as a Function of [η_s] According to Martin's Formula (k = 0.14)[A]

	$\eta \cdot c$									
η_s	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									
η_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									

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