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.

[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.

Current edition approved May 1, 2023. Published May 2023. Originally approved in 1983. Last previous edition approved in 2016 as D4243 – 16. DOI: 10.1520/D4243-23.

[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:

$$C = \frac{\eta}{\rho \cdot T} \tag{1}$$

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:

$$[Cu(NH_2 - CH_2 - CH_2 - NH_2)_2](OH)_2 \tag{2}$$

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

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

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

$$C_{ED}/C_{Cu} = 2.0 \pm 0.1. \tag{4}$$

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