



Standard Test Methods for Vulcanized Fibre Used for Electrical Insulation¹

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1. Scope*

1.1 These test methods cover the procedures for testing vulcanized fibre sheets, tubes, and rods of such grades as can be used for electrical insulation.

1.2 The procedures appear in the following sections:

Procedure	Section
Arc resistance	23
Ash	6
Bond strength (ply adhesion)	16
Bursting strength	14
Compressive strength	12, 25, 30
Conditioning	4
Density	17, 26, 32
Dielectric strength	18, 27
Dimensional measurements	28, 33
Flammability	22
Flexural strength	13, 31
Silica	7
Tearing strength	11
Tensile strength	10, 24, 29
Thickness (sheets)	20
Resistance to impact	15
Rockwell hardness	19
Volatile matter	8
Water absorption	5
Zinc chloride	9

1.3 The values stated in inch-pound units are to be regarded as the standard. The SI values given in parentheses are for information only.

1.4 *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 determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in 7.3, 22.1, 22.2.

NOTE 1—The test methods described herein are similar technically to those described in IEC Publication 60667-2. Not all of the tests in this document are included in IEC 60667-2, and the procedures in the two publications are not completely identical; but it is expected that comparable results will be obtained from most of the procedures. Conduct comparative tests if necessary before directly comparing results of tests

¹ These test methods are under the jurisdiction of ASTM Committee D09 on Electrical and Electronic Insulating Materials and are the direct responsibility of Subcommittee D09.07 on Electrical Insulating Materials

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using the different procedures.

2. Referenced Documents

2.1 ASTM Standards:²

- D149 Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies
- D202 Test Methods for Sampling and Testing Untreated Paper Used for Electrical Insulation
- D229 Test Methods for Rigid Sheet and Plate Materials Used for Electrical Insulation
- D256 Test Methods for Determining the Izod Pendulum Impact Resistance of Plastics
- D348 Test Methods for Rigid Tubes Used for Electrical Insulation
- D349 Test Methods for Laminated Round Rods Used for Electrical Insulation
- D374 Test Methods for Thickness of Solid Electrical Insulation (Metric) D0374_D0374M
- D495 Test Method for High-Voltage, Low-Current, Dry Arc Resistance of Solid Electrical Insulation
- D570 Test Method for Water Absorption of Plastics
- D668 Test Methods of Measuring Dimensions of Rigid Rods and Tubes Used for Electrical Insulation
- D689 Test Method for Internal Tearing Resistance of Paper (Withdrawn 2009)³
- D695 Test Method for Compressive Properties of Rigid Plastics
- D710 Specification for Vulcanized Fibre Sheets, Rods, and Tubes Used for Electrical Insulation
- D785 Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials
- D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
- D828 Test Method for Tensile Properties of Paper and Paperboard Using Constant-Rate-of-Elongation Apparatus
- D952 Test Method for Bond or Cohesive Strength of Sheet Plastics and Electrical Insulating Materials

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 The last approved version of this historical standard is referenced on www.astm.org.

*A Summary of Changes section appears at the end of this standard

D1711 Terminology Relating to Electrical Insulation
D6054 Practice for Conditioning Electrical Insulating Materials for Testing (Withdrawn 2012)³

2.2 IEC Standard:

IEC 60667-2 Specification for Vulcanized Fibre for Electrical Purposes—Part 2: Methods of Test⁴

3. Terminology

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 In referring to the cutting of specimens and the application of the load, the following definitions of terms apply. These definitions of terms correspond to normal practice in the paper industry.

3.2.2 *vulcanized fibre, n*—a material made from chemically gelatinized cellulosic paper or board using zinc chloride as the gelatinizing agent.

3.2.2.1 *Discussion*—The zinc chloride is subsequently removed by leaching. The resulting product, after being dried and finished by calendaring, is a material of partially regenerated cellulose in which the fibrous structure is retained in varying degrees depending on the grade of paper used and on the processing conditions. Material up to about 3.2 mm in thickness is produced by bonding multiple layers of paper (or board) after chemical treatment. Vulcanized fibre thicker than 3.2 mm is typically produced by laminating multiple plies of vulcanized fibre together. Vulcanized fibre does not contain vulcanized rubber or sulfur as the name might imply. Thin vulcanized fibre has sometimes been termed “fish paper.” For more detail on vulcanized fibre refer to Specification **D710**.

3.2.3 *lengthwise (LW), adj*—the direction of the sheet which is strongest in flexure.

3.2.3.1 *Discussion*—This is also known as the *machine direction*. It is the lengthwise direction in which the paper is formed and travels on the paper machine, wire, or cylinder. Since making paper (which form the plies of vulcanized fibre) is a directional process, vulcanized fibre’s properties differ significantly between machine direction and cross direction. For some materials, including the raw materials used for the manufacture of materials considered herein, this direction may be designated as the warp direction.

3.2.4 *crosswise (CW), adj*—the direction of the sheet which is at 90° to the lengthwise direction, and which is normally the weakest direction in flexure.

3.2.4.1 *Discussion*—For some materials, including the raw materials used for manufacture of materials considered herein, this direction may be designated as the cross-machine direction or the weft direction. It is also known as *cross direction*.

3.2.5 *edgewise loading, v*—mechanical force applied in the plane of the original sheet or plate.

3.2.6 *flatwise loading, v*—mechanical force applied normal to the surfaces of the original sheet or plate.

4. Conditioning

4.1 Vulcanized fibre shall be conditioned for mechanical tests in accordance with Practice **D6054**, using Procedure A for conditioning material 0.045 in. (1.14 mm) and under in thickness, and Procedure B for conditioning material over 0.045 in. in thickness. In case of dispute in tests of materials over 0.045 in. in thickness, the material shall be exposed for 48 h at standard laboratory atmosphere prior to conditioning by Procedure B.

4.2 Vulcanized fibre shall be conditioned for electrical tests in accordance with Practice **D6054**, using Procedure A. In case of dispute, a referee test shall be used in which the time of exposure to standard laboratory atmosphere is increased to a minimum period of seven days for all thicknesses.

4.2.1 The following are the typical reasons to undertake conditioning of specimens: (1) for the purpose of bringing the material into equilibrium with normal or average room conditions of 23°C and 50% relative humidity, (2) to obtain reproducible results regardless of the previous history of exposure, or (3) to subject the material to various conditions of temperature or humidity in order to predict its service behavior.

4.2.2 It is possible that the conditioning of Procedure B prescribed in Practice **D6054** to obtain reproducible results will give physical values somewhat higher or somewhat lower than the values at equilibrium under normal conditions, depending upon the test. This procedure for conditioning is used because of the relatively short time required. To bring the material to an equilibrium condition in a controlled humidity would require a long period of time which might extend over many months, for example, for thicknesses over 25 mm. The exact length of time would depend upon such factors as thickness, grade, and previous history of the specimens, and it would be too long for ordinary commercial test purposes. It is probably possible to reduce the time of exposure for some very thin sizes of material conditioned in accordance with Procedure A, but sufficient data on the various thicknesses are not yet available to permit a decision to be made.

METHODS APPLICABLE TO SHEETS, TUBES, AND RODS

5. Water Absorption

5.1 *Significance and Use*—This test method is a guide for the proportion of water absorbed in vulcanized fibre sheets, tubes, and rods and to the effects thereof on certain electrical and mechanical properties. It also is useful in determining the uniformity of quality in these materials.

5.2 *Procedure*—Determine water absorption in accordance with Test Method **D570** on specimens dried in an oven for 1 h at 105 to 110°C prior to immersion in water.

6. Ash

6.1 *Significance and Use*—This test method provides a procedure for determining the amount of ash of a dried specimen, something potentially useful in determining the continuity of quality.

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

6.2 *Test Specimen*—The test specimen shall consist of 2 to 5 g of vulcanized fibre in the form of finely divided particles, such as millings or filings.

6.3 *Procedure*—Dry the test specimen for 2 h at 105 to 110°C and weigh. Then ignite the specimen to constant weight in a crucible and reweigh. Calculate the percentage of ash, based on the weight of the dried specimen.

6.4 *Report*—Report the following information:

6.4.1 Identification of the material, and

6.4.2 The percentage by weight of ash.

6.5 *Precision and Bias:*

6.5.1 This test method has been used for many years, but no information has been presented to ASTM upon which to base a precision statement. No activity has been planned to develop such information.

6.5.2 This test method has no bias because the value for percentage of ash is determined solely in terms of this test method itself.

7. Silica

7.1 *Significance and Use*—This test method is useful in determining the continuity of quality of vulcanized fibre sheets, tubes, and rods and in providing a means of evaluating changes in the leaching medium and the efficiency of the leaching process.

7.2 *Test Specimen*—The test specimen shall consist of approximately 3 g of vulcanized fibre in the form of finely divided particles, such as millings or filings.

7.3 *Procedure*—Dry the test specimen for 1 h at 105 to 110°C. Transfer the dried specimen to a crucible and slowly ignite it to constant weight. Wet the ash with distilled water and transfer to a heat-resistant glass beaker. Add about 75 mL of HCl (sp gr 1.19) and cover the beaker with a watchglass. Evaporate the contents of the beaker to dryness. To the residue slowly add 10 mL of HCl (sp gr 1.19) followed by 75 mL of distilled water. Filter the mixture through ashless filter paper and wash with cold water, then with warm water, until the filtrate is free of chlorides. Ignite the filter paper to constant weight in a weighed platinum crucible. Then add 4 mL of HF (48 to 60 %) and apply heat until all white fumes are driven off. Cool and weigh the crucible. The difference between this weight and the previous one indicates silicon present as silica. The results shall be expressed as a percentage of the weight of the dried specimen. (**Warning**—Both hydrochloric acid (HCl) and hydrofluoric acid (HF) are corrosive and toxic. Take care to avoid spillage and contact with the skin. Evaporate solutions of these acids in a well-ventilated fume hood.)

7.4 *Report*—Report the following information:

7.4.1 Identification of the material, and

7.4.2 The percentage by weight of silica.

7.5 *Precision and Bias:*

7.5.1 This test method has been used for many years, but no information has been presented to ASTM upon which to base a precision statement. No activity has been planned to develop such information.

7.5.2 This test method has no bias because the value for percentage of silica is determined solely in terms of this test method itself.

8. Volatile Matter

8.1 *Significance and Use*—The presence of high levels of volatile matter has the potential to be detrimental to the use of vulcanized fibre sheets, tubes, and rods in some electrical applications. It is possible that data on volatile content will be helpful to determine the suitability for a particular application and to determine the continuity of quality.

8.2 *Purpose*—This test method is intended for the rapid determination of the amount of moisture and other volatile matter in vulcanized fibre of all grades and thicknesses.

8.3 *Test Specimens*—Prepare the test specimen which consists of the minimum number of pieces of fibre required to give a total weight of at least 1 g, as follows:

8.3.1 *Sheets*—For sheets less than $\frac{1}{16}$ in. (1.59 mm) in thickness, the pieces for the test specimen shall be 75 by 25 mm by the thickness of the sheet; for sheets $\frac{1}{16}$ in. and over in thickness, the pieces for the test specimen shall be 75 by 3 mm by the thickness of the sheet. The pieces shall be band-sawed or sheared from the sample so as to produce smooth edges free of cracks. The sawed faces shall be sanded or filed to remove any protruding sections that might be broken off during the test. The sample shall be sawed slowly so that the fibre is not heated appreciably. The thickness of the specimens shall be measured to the nearest 0.001 in. (0.025 mm) in the direction perpendicular to the natural faces of the original sample.

8.3.2 *Tubes*—For tubes less than $\frac{1}{16}$ in. (1.59 mm) in wall thickness, the pieces for the test specimen shall be 25-mm lengths. For tubes $\frac{1}{16}$ in. and over in wall thickness, the pieces for the test specimen shall be 3-mm lengths slowly cut with a band saw.

8.3.3 *Rods*—For rods less than $\frac{3}{16}$ in. (4.76 mm) in diameter, the pieces for the test specimen shall consist of continuous lengths. For rods $\frac{3}{16}$ in. and over in diameter, the pieces for the test specimen shall be 3-mm lengths slowly cut with a band saw.

8.4 *Procedure*—Test three specimens, each consisting of one or more pieces of fibre as required, individually. Weigh each specimen to the nearest 1 mg, and place it in a mechanical convection oven maintained at a temperature of $135 \pm 2^\circ\text{C}$ and heated for the period prescribed in the following table. Specimens less than $\frac{1}{32}$ in. (0.80 mm) in thickness shall be weighed (but not heated) in a weighing bottle.

	Size	Heating Period, h
Sheets	Under $\frac{1}{16}$ in. (1.59 mm), in thickness	2
	$\frac{1}{16}$ and over in thickness	4
Tubes	Under $\frac{1}{16}$ in. in wall thickness	2
	$\frac{1}{16}$ in. and over in wall thickness	4
Rods	Under $\frac{3}{16}$ in. (4.76 mm) in diameter	2
	$\frac{3}{16}$ in. and over in diameter	4

8.4.1 Remove the specimens from the oven, cool in a desiccator, and weigh to the nearest 1 mg. The difference between the original weight and the final weight of the specimens shall be considered as the volatile matter content.

8.5 *Calculation*—Calculate the percentage of volatile matter content of the specimen as follows:

$$\text{Volatile matter, \%} = [(W_1 - W_2)/W_2] \times 100 \quad (1)$$

where:

W_1 = original weight of specimen, and

W_2 = final weight of specimen.

8.6 *Report*—Report the following information:

8.6.1 Color and dimensions of the specimen, and

8.6.2 Percentage of volatile matter.

8.7 *Precision and Bias*:

8.7.1 This test method has been used for many years, but no information has been presented to ASTM upon which to base a precision statement. No activity has been planned to develop such information.

8.7.2 This test method has no bias because the value for percentage of volatile matter is determined solely in terms of this test method itself.

9. Zinc Chloride

9.1 *Significance and Use*—The amount of zinc chloride is indicative of the efficiency of the leaching process used in the manufacture of vulcanized fibre sheets, tubes, and rods.

9.2 *Test Specimen*—The test specimen shall consist of approximately 3 g of vulcanized fibre in the form of finely divided particles, such as millings or filings.

9.3 *Procedure*—Dry the test specimen for 1 h at 105 to 110°C. Place the dried specimen in a Soxhlet, Wiley-Richardson, or similar type of extractor and extract with distilled water for 3 h. The rate of extraction with the Wiley-Richardson size of extractor shall be sufficient to cause the tube containing the specimen to be filled by condensation and emptied by the siphon at least six times per hour. When the Soxhlet or similar size extractor is used, the tube shall be filled and emptied at a rate of at least three cycles per hour. After this extract has cooled, add 0.5 mL of K_2CrO_4 solution (10 %) and titrate the extract with standard $AgNO_3$ solution. The end point is indicated by the appearance of a permanent red color. Make a blank test on an equal volume of distilled water and K_2CrO_4 solution and subtract the result from that of the specimen. Calculate the net result expressed as $ZnCl_2$.

9.4 *Report*—Report the following information:

9.4.1 Identification of the material, and

9.4.2 Percentage by weight of $ZnCl_2$.

9.5 *Precision and Bias*:

9.5.1 This test method has been used for many years, but no information has been presented to ASTM upon which to base a precision statement. No activity has been planned to develop such information.

9.5.2 This test method has no bias because the value for percentage of zinc chloride is determined solely in terms of this test method itself.

METHODS APPLICABLE TO SHEETS

NOTE 2—In addition to the test methods for sheets covered in the following Sections 10 – 23, inclusive, the methods described in Sections 5 – 9 are also applicable to sheet materials.

10. Tensile Strength

10.1 *Significance and Use*—Tensile strength is of importance as a measurement of uniformity and quality of the material. It also serves to indicate its ability to withstand stress in application and service.

10.2 For sheets 0.030 in. (0.762 mm) and under in thickness, determine the tensile strength in accordance with the procedure described in Test Method D828.

10.3 For sheets over 0.030 in. (0.762 mm) in thickness, determine the tensile strength in accordance with the procedure described in Test Methods D229.

11. Tearing Strength

11.1 *Significance and Use*—Resistance to tearing is an important property for measuring suitability for certain application processes and as a means of determining the continuity of quality.

11.2 Determine the tearing strength of sheets 0.030 in. (0.762 mm) and under in thickness in accordance with the procedure described in Test Method D689.

12. Compressive Strength

12.1 *Significance and Use*—Compressive test data provide information useful in research and development, engineering design, determining the suitability of the material for specific load requirements in service, and in determining the continuity of quality.

12.2 Determine the compressive strength on four specimens using the procedure described in Test Method D695.

13. Flexural Strength

13.1 *Significance and Use*—Flexural strength data are useful for determining the continuity of quality and it is possible that they will provide important guidance in the design and construction of electrical equipment.

13.2 Determine the flexural strength of sheets $\frac{1}{16}$ in. (1.59 mm) and over in thickness in accordance with the procedure described in Test Methods D229.

14. Bursting Strength

14.1 *Significance and Use*—Bursting strength data are useful as a means of determining the continuity of quality and as an index of resistance to puncture under stresses found in construction and assembly of electrical equipment.

14.2 Determine the bursting strength of sheets 0.060 in. (1.52 mm) and under in thickness in accordance with the procedure described in Test Methods D202.

15. Resistance to Impact

15.1 *Significance and Use*—Data on impact resistance are useful in evaluating the suitability of vulcanized fibre sheets in applications where shock stresses are present in the construction, assembly, or service use of equipment in which it is used. It also serves as a guide for determining the continuity of quality.