Designation: D 3756 - 97 (Reapproved 2004)

# Standard Test Method for Evaluation of Resistance to Electrical Breakdown by Treeing in Solid Dielectric Materials Using Diverging Fields<sup>1</sup>

This standard is issued under the fixed designation D 3756; 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.

#### INTRODUCTION

When failure occurs in solid organic dielectrics that are subjected to very high, continuous, and nonuniform electrical gradients, it generally occurs by a mechanism called treeing. Materials of different molecular structures have different degrees of resistance to failure by treeing, and this resistance can sometimes be increased by the addition of other materials in low concentration.<sup>2</sup>

Trees that grow by a molecular degradation mechanism resulting from partial discharge (corona) are called electrical trees to distinguish them from water and electrochemical trees which are quite different.

This test method makes use of two opposing thin cylindrical electrodes, one sharpened to a point, the other with a hemispherical end. They are molded or inserted into blocks of the material to be tested. Because of the shape of the electrodes this is often called a needle test. This test provides a statistical estimate of electrical treeing initiation and propagation of solid dielectric materials in high, diverging electrical fields.

# 1. Scope

- 1.1 This test method covers the evaluation and comparison of the resistance of solid organic dielectric materials to the initiation or growth, or both, of tubular tree-like channels resulting from partial discharge (corona) and molecular decomposition that occur in the region of very high, diverging electric fields.<sup>3,4</sup>
- 1.2 This test method is primarily for use at a power frequency of 50 or 60 Hz.
- 1.3 The test may be carried out at room temperature or temperatures above or below room temperature. The temperature should not exceed the softening or melting point of the sample material.
- 1.4 This test method can be used for any solid material into which needles can be cast, molded, or inserted with heat after

molding. The resistance to tree initiation is measured by the double-needle characteristic voltage, which is only applicable

to non-opaque materials so that tree can be observed optically.

The resistance to tree initiation and growth is reported by the

double-needle voltage life, which is applicable to both opaque

1.5 The values stated in SI units are to be regarded as the

#### 2. Referenced Documents

bility of regulatory limitations prior to use.

and non-opaque materials.

- 2.1 ASTM Standards: <sup>5</sup>
- D 149 Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies
- D 1711 Terminology Relating to Electrical Insulation
- D 1928 Practice for Preparation of Compression-Molded Test Sheets and Test Specimens<sup>6</sup>

standard.

1.6 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 applica-

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D09 on Electrical and Electronic Insulating Materials and is the direct responsibility of Subcommittee D09.12 on Electrical Tests.

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<sup>&</sup>lt;sup>2</sup> Symposium on Engineering Dielectrics, ASTM STP 783, ASTM, 1982, and Symposium on Engineering Dielectrics, ASTM STP 926, ASTM, 1986.

<sup>&</sup>lt;sup>3</sup> W. D. Wilkens, Chapter 7, "Statistical Methods for the Evaluation of Electrical Insulating Systems," *Engineering Dielectrics, Vol IIB, Electrical Properties of Solid Insulating Materials, Measurement Techniques*, R. Bartnikas, Editor, *ASTM STP* 926, ASTM, Philadelphia, 1987.

<sup>&</sup>lt;sup>4</sup> R. M. Eichorn, Chapter 4, "Treeing in Solid Organic Dielectric Materials," *Engineering Dielectrics, Vol IIA, Electrical Properties of Solid Insulating Materials: Molecular Structure and Electrical Behavior*, R. Bartnikas and R. M. Eichorn, Editors, *ASTM STP 783*, ASTM Philadelphia, 1983.

<sup>&</sup>lt;sup>5</sup> 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.

<sup>&</sup>lt;sup>6</sup> Withdrawn.

- D 2275 Test Method for Voltage Endurance of Solid Electrical Insulating Materials Subjected to Partial Discharges (Corona) on the Surface
- 2.2 Other Document:

ANSI/IEEE 930-1987 IEEE Guide for the Statistical Analysis of Electrical Insulation Voltage Endurance Data<sup>7</sup>

#### 3. Terminology

- 3.1 Definitions:
- 3.1.1 partial discharge, n—refer to D 1711.
- 3.2 Definition of Terms Specific to This Standard:
- 3.2.1 characteristic voltage or DNCV (double-needle characteristic voltage), n—that voltage which, when applied for 1 h between the ends of two thin cylindrical electrodes (one sharpened to a point, the other with a hemispherical end) in a group of replicate specimens, produces observable dielectric damage at the point of the sharp electrode in half of the specimens.
- 3.2.2 median voltage life  $(t_{50})$ , n—the time, determined from a Weibull plot, when 50 % failure occurs from a group of 10 identical specimens subjected to the same voltage stress.

## 4. Summary of Method

4.1 In this test method, specimens are prepared and needles inserted to serve as electrodes. Voltage is applied to the needles and continued for 1 h in the double-needle characteristic voltage test or until electrical breakdown occurs in the double-needle voltage life test. Results are expressed as the voltage at which half of the specimens show dielectric damage in 1 h, or the median time to failure of a group of specimens subjected to a given continuous voltage, at a selected or predetermined temperature.

# 5. Significance and Use

- 5.1 This is a laboratory test designed to simulate the effects of (I) the presence of rough interfaces between conductor or semiconductive screen and primary insulation in an insulation system, (2) the presence of foreign particles (contaminants) in an insulation system, and (3) the presence of small voids or cavities within the insulation.
- 5.2 This test method provides comparative data. The degree of correlation with actual performance in service has not been established.

## 6. Apparatus

- 6.1 *Power Supply*—A high voltage supply having a sinusoidal voltage output at a power frequency equipped with continuous voltage control and an adjustable protective automatic circuit-breaking device that operates at a controllable current level. See Test Method D 149.
- 6.2 *Current*—Sensitive Individual Specimen Disconnect—When ten specimens are tested to failure for the voltage life test, use a disconnect circuit for each.
- 6.3 *Electrodes*—The critical electrode is a round steel rod, 1 mm in diameter, sharpened at one end to a controlled radius of  $3 \pm 1 \mu m$  and an included angle of  $30 \pm 1 degrees$ .
- <sup>7</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

- 6.4 *Needle-Grinding Lathe*, for preparation of the sharpened electrodes to a controlled-point sharpness and included angle. A typical lathe and grinder combination are shown in Fig. 1.
- 6.5 *Specimen Molding Chase*, for single-step preparation of compression molded specimens containing needles. A typical chase is shown in Fig. 2.
- 6.6 *Needle-Insertion Jig*—A jig is required for slow, controlled insertion of electrodes. Fig. 3 shows a specimen insertion jig.
- 6.7 *Test Chamber*—Any temperature-controlled test chamber or enclosure, which can hold at least ten specimens and maintain uniform temperature, is appropriate for this test.

#### 7. Sampling

7.1 Sample so that the specimens tested will represent the entire lot.

#### 8. Test Specimens

- 8.1 Test specimens are approximately 25-mm² blocks, 6 mm thick, containing two needle-like electrodes as shown in Fig. 4. Prepare by compression molding, extrusion, or cutting from finished pieces. The tips of the electrodes are separated by 12.0  $\pm$  0.5 mm for characteristic voltage determination and 6.5  $\pm$  0.5 mm for voltage life determination. The number of test specimens is at least 24 for characteristic voltage determination (i.e. six specimens for each of at least four different testing voltages) and 10 for voltage life determination.
- 8.2 Preparation of Thermoplastic and Crosslinked Specimens—Compression-mold plaques, 6-mm thick, of the sample material, in a steam or electrically heated hydraulic press equipped for cold-water cooling of the platens. Use a positive pressure mold, which may be of either the picture-frame type or the milled-cavity type. Use parting sheets of cellophane, polyester film, or aluminum foil between the mold surfaces and the resin. The choice of parting sheet depends somewhat on the molding temperature, although aluminum sheets, washed with alcohol and thoroughly dried, are preferred. The size of the mold is not critical,  $200 \times 200$  mm is

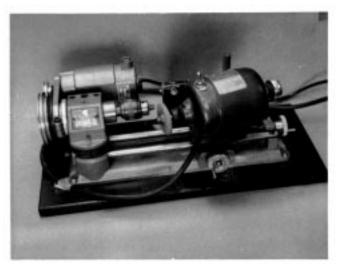


FIG. 1 Machine Setup for Needle Sharpening

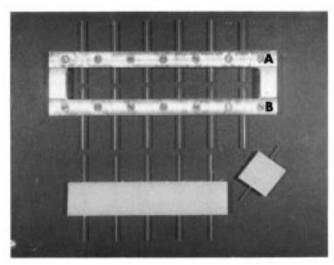


FIG. 2 Chase for Preparation of Specimens Containing Electrodes



FIG. 3 Needle Insertion Jig

recommended. For peroxide-crosslinkable materials, the typical compression-molding conditions should follow the material manufacturers recommendation of temperature, time and pressure. The by-products of peroxide decomposition should be removed before testing by use of a vacuum oven at elevated temperatures (80°C for 7 days for XLPE using dicumyl peroxide).

8.2.1 After molding, cut the plaque into 25-mm square blocks with square and smooth edges. Store the squares under standard laboratory conditions, 23°C and 50 % relative humidity, and protect them from dirt and atmospheric contamination until used.

8.3 *Insertion of Needles*—Insert the needle electrodes into the specimen blocks slowly and carefully to avoid orientation strains, formation of cavities, and damage to the sharp points. Use a jig, such as the one shown in Fig. 3, to ensure that every needle will be inserted under identical conditions.

8.3.1 Make the insertions as follows: Place twelve specimen blocks in the slots provided for them and lightly clamp into

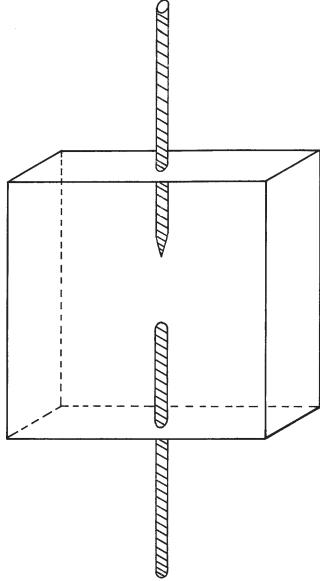


FIG. 4 Finished Specimen

place. Inspect the twelve sharpened needles, after cleaning with methyl ethyl ketone; then carefully place them into the needle slots on one side so their tips just touch the specimens. Use the individual adjusting screws for positioning the needles. Slip shims into the needle slots above the needles, and use a cover plate to close the top of the needle slots. Secure this cover with small C-clamps at each end. The purpose of the shims and cover is to prevent the needle from cocking, and to force it to enter straight into the specimen. Place twelve electrodes with hemispheric ends in the slots on the opposite sides of the specimens in the same manner.

8.3.2 When the specimens and needles are mounted and the needles are adjusted into proper position, place the whole jig into a circulating air oven at 105°C, for low-density polyethylene or crosslinked materials, for a 1-h preheating period. For other partially crystalline polymeric materials use a temperature approximately 10°C below the thermodynamic first-order transition point. After preheating, simultaneously advance the

needles 1.30 mm by making one full turn of the large lead screw. Repeat at 5-min intervals. Make sufficient turns to accomplish the insertion. Five turns are normally required for the characteristic voltage test and seven for the voltage life test. An electrode gap of  $12.0\pm0.5$  mm is commonly used for characteristic voltage determination and  $6.5\pm0.5$  mm is preferred for voltage life tests. Use constant gaps for comparison of materials by this test.

- 8.3.3 When the insertions are complete, leave the jigs untouched for at least 30 min in the oven for stress relief or annealing. Remove the jigs from the oven and cool naturally to room temperature. Examine each specimen with a microscope, if possible, to ensure that the needle point was not damaged during insertion.
- 8.4 Preparation of Specimens with Molded-In Electrodes—Condition as specified in Section 9.
- 8.4.1 The molding is  $150 \times 25 \times 6$  mm thick and contains six pointed and six hemispherical electrodes. Cut six square specimens from this block. See Fig. 2.
- 8.4.2 A compression mold that can be used for preparation of the six specimen plaques consists of a chase, in two layers, grooved to clamp the electrodes during molding. It is surrounded laterally by a steel backup plate to prevent distortion of the chase. Slits cut in the backup plate accommodate the ends of the needles. The top and bottom are polished stainless steel plates.
- 8.4.3 To mount the needles, remove the top clamp plates A and B. Lay the hemispherical electrodes carefully in place in the grooves so the ends project approximately 11 mm into the hollow section. Replace plate A and tighten the screws. Follow the same procedure for the sharp electrodes under plate B, except do not tighten the screws. Position the sharp electrodes under a microscope with a built-in optical scale. Carefully manipulate each sharp electrode so that the specified spacing between the opposing electrodes is obtained. Then tighten the screws in plate B. Check to see that the spacing has been maintained. Care must be exercised throughout in order not to bend the needle points.
- 8.4.4 Place one of the  $200 \times 250$ -mm polished plates on the lower platen of the molding press. Cover this with a sheet of polyester film or aluminum foil, at least 0.1-mm thick, to prevent sticking of the resin in the polished plate. Lay the chase centrally on this polyester sheet or aluminum foil. Evenly distribute approximately 30 g of the molding material (for example, polyethylene) to be tested within the cavity in the mold. Then lay a second sheet of polyester film or foil and the second polished plate over the mold.
- 8.4.5 Mold the specimen in accordance with Practice D 1928.
- 8.4.6 Remove the specimens by removing the screws in both plates A and B.
- 8.4.7 Cut the 150-mm block into six 25-mm<sup>2</sup> individual specimens.
- 8.4.8 Check each specimen under a microscope with an optical scale and use only if the electrode spacing is found to be correct. If any contamination or voids are found within the test region, reject the specimen.

#### 9. Conditioning

9.1 After specimen preparation is complete, store all specimens for approximately 24 h at 23°C and 50 % relative humidity before testing.

## 10. Procedure

10.1 Warning—Lethal voltages are a potential hazard during the performance of this test. It is essential that the test apparatus and all associated equipment that may be electrically connected to it be properly designed and installed for safe operation. Solidly ground all electrically conductive parts which are possible for a person to contact during the test. Provide means for use at the completion of any test to ground any parts which were at high voltage during the test or have the potential for acquiring an induced charge during the test or retaining a charge even after disconnection of the voltage source. Thoroughly instruct all operators as to the correct procedures for performing tests safely. When making highvoltage tests, particularly in compressed gas, oil, water or aqueous solution, it is possible for the energy released at breakdown to be sufficient to result in fire, explosion, or rupture of the test chamber. Design test equipment, test chambers, and test specimens so as to minimize the possibility of such occurrences and to eliminate the possibility of personal injury. If the potential for fire exists, have fire suppression equipment available.

10.2 Characteristic Voltage Test:

10.2.1 Electrical Stressing of Specimens—Prepare 24 specimens of one sample for double-needle characteristic voltage determination. Carefully wash all lateral surfaces of the test blocks with alcohol. Place six of the specimens in a suitable specimen holder designed to support the blocks in a vertical position spaced at least 50 mm between centers. The test chamber must be large enough to accommodate the specimen holder and specimens with sufficient clearance to prevent flash-over to any parts of the chamber. All access doors to the chamber must be electrically interlocked. After electrical connections have been made to the specimens, close the door of the chamber and apply the voltage. Raise the voltage at 500 V/s and without interruption to the test level, and maintain it constantly for 1 h. After this period, reduce the voltage to zero, turn off the power supply, and remove the specimens for inspection.

10.2.2 Inspection of Specimens—Inspect for the presence of trees at the tip of the sharp electrode with a 100× optical microscope. If necessary, the specimen may be sectioned to decrease thickness and improve visibility. A drop of microscope immersion oil<sup>8</sup> on the top and bottom of the specimen facilitates the recognition of internal structure. Any change in appearance at the needle tip is considered to be a tree. This includes a short hollow void, a trunk or a tree, and even dots. The number of specimens in which trees have formed is expressed as "fraction of specimens treed." Do not reuse the specimen.

<sup>&</sup>lt;sup>8</sup> Suitable oil of high optical quality and refractive index that is close to the organic plastics of Cargille's nondrying immersion oil for microscopy is supplied by Fisher Scientific Co., 203 Fisher Building, Pittsburgh, PA 15219.

10.2.3 Repeat 10.2.1 and 10.2.2. Use four test voltages; at least one test voltage with some treed specimens, but less than half the specimens treed; and at least one test voltage with treed specimens more than half, but not all of the specimens treed. The suggested starting voltage is 10 kV, particularly for crosslinked polyethylene. The next voltage can be higher or lower, depending on the fraction of specimens treed at the previous voltage.

10.3 Divergent-Field Voltage Life Test—Proceed as outlined in 10.2.1, preparing only 10 specimens. Apply the voltage as in 10.2.1 and maintain until failure (complete breakdown) occurs for all 10 specimens. Of course, a lower applied voltage gives a longer voltage life time. Determine a test voltage by experimentation so that breakdown of all 10 specimens occurs in a reasonable time (usually less than 30 days). The typical applied voltage range for crosslinked polyethylene is 10 to 20 kV

10.3.1 Record the applied voltage and the amount of time for each specimen to fail.

# 11. Presentation of Results

11.1 *Voltage Life Test*—When all 10 specimens fail, the percentage failures from the first to the tenth failure are 6.7 %, 16.3 %, 25.9 %, 35.6 %, 45.2 %, 54.8 %, 64.4 %, 74.1 %, 83.7 %, and 93.3 %, respectively, in a Weibull probability plot. Plot the results of the percent Weibull failure versus the failure time of each specimen in a Weibull probability plot. The data should define a reasonably straight line (Weibull line). When the slope of the line is chosen by rotating a line counterclockwise from the horizontal, about the 50 % region, and stopping at the slope where half of the data points in the first quadrant lie to the right of the line, and in the third quadrant, and half lie to its left, the intersection of the line at the 50 % Weibull failure defines the voltage life ( $t_{50}$ ). Of course, the voltage life at 10 %, 63.2 %, or 90 % Weibull failure can be determined in the same way.

# 12. Report

- 12.1 Report the following information:
- 12.1.1 Material tested,
- 12.1.2 Number of specimens tested,
- 12.1.3 Conditioning prior to testing,
- 12.1.4 Test conditions,
- 12.1.5 Specimen geometry,
- 12.1.5.1 Electrode tip radius including angle, and
- 12.1.5.2 Electrode separation distance.
- 12.1.6 For the Characteristic Voltage Test:
- 12.1.6.1 Measured characteristic voltage, and
- 12.1.6.2 Temperature.
- 12.1.7 For the Voltage Life Test:
- 12.1.7.1 Voltage life, expressed at  $t_{50}$ ,
- 12.1.7.2 Applied voltage, and
- 12.1.7.3 Temperature.

## 13. Precision and Bias

- 13.1 The characteristic voltage parameter should be capable of estimation up to  $\pm 500$  V or approximately 5 %. Differences in voltage life,  $t_{50}$ , are estimated significant at the  $2\sigma$  level.
- 13.2 The divergent-field voltage life test is not a direct measurement, but only a statistical estimation. Implicit in the statistic are the assumptions that (1) all the pointed electrodes are equally sharp within reasonable limits, (2) all insertions are accomplished without leaving residual strains or orientation about the needles in the specimens, (3) the surroundings of all the needle tips are the same, which really means that the specimens are homogeneous, and (4) any components in the formulation of the specimens that might exude to the surface can be completely washed away and therefore not produce erroneous results due to the Faraday cage effect.

## 14. Keywords

14.1 needles; partial discharge; resistance to electrical breakdown; treeing; voltage life

#### ANNEX

(Mandatory Information)

## A1. PREPARATION OF NEEDLE ELECTRODES

A1.1 Use standard sewing needles called "No. 7 Sharps" which have a shank diameter of approximately 0.7 mm, length 37.5 mm, and a tip diameter of approximately 0.025 mm. Clip off the eye with diagonal cutters and insert the needle into the chuck of a lathe so that only approximately 5 mm protrudes. It is easier and quicker to do this precision grinding with a watchmaker's lathe spindle and a precision steel collet in place of the standard chuck. Sharpen the point to a cone shape with an included angle of 30° by grinding against a rotating wheel mounted on a tool post grinder. The wheel shall be a very fine grained and very hard stone. "Hard Arkansas, Fine Grain" oilstone grinding wheels made by the Norton Co. are the only wheels used for this purpose. Wheels shall measure 50 mm in

diameter, 6 mm thick and have a 6-mm diameter arbor hole.<sup>9</sup> It is prudent to purchase these in multiples, possibly six at a time, because they are a special item and the delivery time is quite long.

A1.2 Accomplish the grinding by rotating the needle and grinding wheel in opposite directions at rates of 3500 and 10 000 rpm, respectively. Wet the wheel with a few drops of kerosine before starting and observe the sharpening process continuously with a  $10\times$  stereo microscope positioned directly

<sup>&</sup>lt;sup>9</sup> Available from Abrasives Distributors, 26 W. Passaic St., Rochell Park, NJ 07662.

above the needle, illuminated by a small microscope spot lamp. When the needle point is sufficiently sharp, remove and temporarily store it, point up and untouched, until several others have been prepared. The group of needles may number up to 30, which is approximately the maximum number of needles that can be sharpened before the surface of the wheel must be redressed. Watch the sharpening carefully, because that is the best way to determine when the needle does not sharpen as expected and dressing is necessary. Sometimes this may be after only a few needles. Use a diamond tool mounted horizontally and at a 75° angle with respect to the axis of the lathe for dressing the wheel. Dressing is done dry.

A1.3 After a group of needles has been prepared, examine each needle with a  $100 \times$  microscope to be sure the points are smooth, conical, and sharp. Those which appear satisfactory, should be examined further with a microprojector. Mounted on the stage, an image of the point is projected at  $220 \times$  upon a white card that bears the outline of a  $30^{\circ}$  plane angle

terminated at the apex by a circular arc 1.0 mm in diameter. This dimension corresponds to a point diameter of 0.00018 in., the maximum permitted. Needles that have an appropriately sharp point, the proper angle, and are smooth and burr-free are acceptable. Dip the tips in light machine oil and store, points up and untouched, in a desiccator prior to future use.

- A1.4 As an alternative to preparing electrodes from sewing needles, drill blanks may be used. A hemisphere is ground on one electrode, a sharp point on the other.
- A1.5 An additional approach is to purchase electrodes. Electrodes made from steel rods 1 mm in diameter with a 3  $\mu$ m-radius point at the end of a 30° included angle are available. Blunt electrodes made from the same 1-mm rods have 0.5 mm radius ends. <sup>10</sup>

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<sup>&</sup>lt;sup>10</sup> Available from Ogura Jewel Industry Co., Ltd., 7-12 Omori Kita 5 Chome, Ota-ku, Tokyo, 143, Japan.