



# Standard Test Method for Relative Resistance to Vented Water-Tree Growth in Solid Dielectric Insulating Materials<sup>1</sup>

This standard is issued under the fixed designation D 6097; 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 covers the relative resistance to vented water-tree growth in solid translucent thermoplastic or cross-linked electrical insulating materials. This test method is especially applicable to extruded polymeric insulation materials used in medium-voltage cables.

1.2 The values given in SI units are to be regarded as the standard.

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 and health practices and determine the applicability of regulatory limitation prior to use.* For specific hazard statements see 8.1.

1.4 There is no similar or equivalent IEC standard.

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 1898 Practice for Sampling of Plastics<sup>2</sup>

D 1928 Practice for Preparation of Compression-Molded Polyethylene Test Sheets and Test Specimens<sup>3</sup>

D 2275 Test Method for Voltage Endurance of Solid Electrical Insulating Materials Subjected to Partial Discharges (Corona) on the Surface<sup>4</sup>

D 3756 Test Method for Evaluation of Resistance to Electrical Breakdown by Treeing in Solid Dielectric Materials Using Diverging Fields<sup>5</sup>

## 3. Terminology

### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *water tree length (WTL),  $n$* —the maximum length of a stained tree-like micro-channel path in millimetres, measured from the tip of the conical defect in the direction of the conical axis.

3.1.2 *resistance to water-tree growth (RWTG)*— a dimensionless value which is  $L$  divided by the WTL.

3.1.3 *thickness of point-to-plane specimen ( $L$ ),  $n$* —the vertical distance in millimetres from the tip of the conical defect to the opposite surface of the solid dielectric material.

## 4. Summary of Test Method

4.1 Ten compression-molded disk specimens, each containing a conical-shaped defect, are subjected to an applied voltage of 5 kV at 1 kHz and  $23 \pm 2^\circ\text{C}$  in an aqueous conductive solution of 1.0 N NaCl for 30 days. This controlled conical defect is created by a sharp needle with an included angle of  $60^\circ$  and a tip radius of 3  $\mu\text{m}$ . The electrical stress at the defect tip is enhanced and can be estimated by the Mason's Hyperbolic point-to-plane stress enhancement equation.<sup>6</sup> This enhanced electrical stress initiates the formation of a vented water-tree grown from the defect tip. Each treed specimen is stained and sliced. The water-tree length and point-to-plane specimen thickness measured under microscope are used to calculate a ratio that is defined as the resistance to water-tree growth.

## 5. Significance and Use

5.1 This is a laboratory test designed to simulate the growth of vented water-trees in the solid dielectric insulating material initiated by a sharp protrusion at the insulating and conductive interface under a wet environment in a high electrical field. Water-treeing is the phenomenon which describes the appearance of tree-like growth in organic dielectrics under an ac field when exposed to moist environments. Two types of water-trees are formed. Bow tie trees (within the dielectric) and vented water-trees formed from conductive/insulating material interface into the insulating material. The water-trees referred to in this test method are the vented type. The insulating material is the solid dielectric organic material. The conductive material is the salt solution. This salt solution is used on both sides of the insulating material to simulate the same inner and outer

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<sup>2</sup> Discontinued; see 1997 Annual Book of ASTM Standards, Vol 08.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 08.01.

<sup>4</sup> Annual Book of ASTM Standards, Vol 10.01.

<sup>5</sup> Annual Book of ASTM Standards, Vol 10.02.

<sup>6</sup> The sole source of supply of the base, Dow Corning 3110RTV, the catalyst, Dow Corning RTV Catalyst S, and the sealant, Dow Corning Multipurpose Silicone Sealant 732, known to the committee at this time is Dow Corning, Inc., Midland, MI 48686. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

semiconductive shields saturated with moisture between the insulation layer used in a medium-voltage underground power cable.

5.2 This test method provides comparative data. The degree of correlation with the performance in service has not been established.

5.3 The standard test conditions are designed to grow a sufficient water-tree length for most solid dielectric insulating materials of interest before electrical breakdown occurs. Materials with a very high resistance to water-tree growth may require a longer time under test conditions (such as 180 days) or higher voltage (such as 10 or 15 kV) in order to differentiate their performance. For materials with a very low resistance to water-tree growth, electrical breakdown may occur during the 30-day testing time. A shorter testing time (such as one or ten days) is recommended to prevent electrical breakdown during testing for those low water-tree resistant materials.

5.4 Other voltages, frequencies, temperatures, aqueous solutions, and defects can be used to evaluate specific materials for specific applications. Temperatures should not exceed the softening or melting point of the material or 10 to 15°C below the boiling point of the salt solution. Any nonstandard conditions should be reported along with the results.

5.5 Tree-growth rates generally increase with the test frequency. An acceleration factor due to frequency is given by  $(f/60)^k$  where  $f$  is the test frequency and  $k$  is between 0.6 and 0.7. The test frequency of 1 kHz is selected to accelerate the water-tree growth. However, the chemical nature of oxidized products from water-treeing may be different at different frequency ranges.

5.6 Two assumptions for this test method are: (1) all tested materials grow trees in the same power law kinetic manner and (2) the time under test conditions of 30 days is long enough to establish the difference in water-tree growth. If there is a doubt, at least three different testing times (such as 30, 90, and 180 days) should be used to verify their comparative performance and disclose their kinetic nature of water-tree growth. Of course, it is also assumed that all water-treed regions are oxidized regions that can be stained for optical observation. Different materials may also have different temperatures and times to stain the oxidized (treed) regions due to their different softening temperature.

## 6. Apparatus

6.1 *Power Supply*—A high-voltage supply with a sinusoidal voltage output of at least 5 kV at a frequency of 1 kHz and an output power of 3 kVA.

6.2 *Conical Needles*—Conical needles are made from steel or tungsten carbide. Their dimensions are  $14.5 \pm 0.5$  mm long,  $4 \pm 0.2$  mm in diameter, point radius of  $3 \pm 1$   $\mu$ m for the needle tip radius, and  $60 \pm 1^\circ$  point angle.

6.3 *Test Specimen Mold*—The test specimen mold is a three-layer metal mold. The top metal plate is flat. The center plate has at least ten holes to make ten test specimens for each material. Each hole has a 25.4-mm diameter and at least 31.75-mm spacing from center to center of each hole. The center plate also has the guide holes about 8 mm in diameter at two corners to mate with pins in the bottom plate section. The bottom plate section consists of two metal plates bolted together. The first bottom plate has the same number of holes

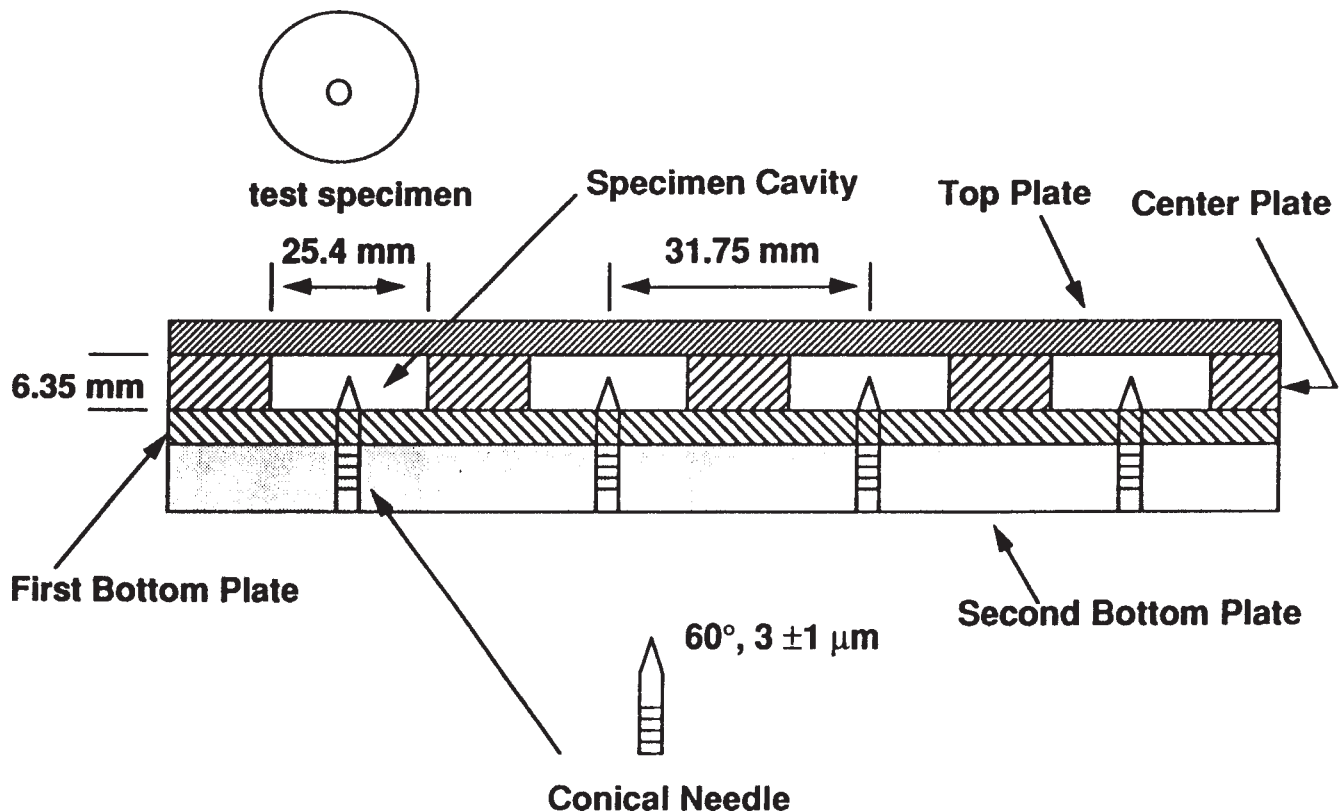


FIG. 1 Test Specimen Mold Cavity

as the center plate. Each hole has the inside diameter of 4 mm to accommodate needles. The second bottom plate has the holes with an inside diameter of 10 mm. The center points of all the holes in the bottom and center plates are matched and aligned. These holes at the second bottom plate are threaded to accommodate the needle support member. The needle support member is fabricated from threaded stainless steel rod drilled at one end to provide a snug fit for needles, and at the other end to accommodate an hexagonal head driver. Needles are threaded into the support member. The needle and needle support assembly is carefully screwed into the base until the needle point extends  $3.2 \pm 0.1$  mm above the surface. Fig. 1 is an example of the mold cavity.

**6.4 Specimen Holder**—The specimen holder, designed to hold at least ten specimens, is made from a solid block of clear polymethyl methacrylate (PMMA). The PMMA is used because of the ease of machining and its good electrical properties. The inside is machined to a depth of 50.8 mm with a 12.7-mm wall thickness. The outside bottom has the same number of holes with an inside diameter of 25.4 mm with a depth of 6.35 mm, drilled with a spacing of 38.1 mm from center to center of the holes. The inside bottom has the same holes with an inside diameter of 12.7 mm and a depth of 6.35 mm in line with the centers of the holes drilled at the outside bottom. Fig. 2 is an example of the specimen holder.

**6.5 Electrodes**—The electrode is made from a 1-m length of 24 AWG nickel-chromium wire or other suitable conductive, noncorrosive metal wire formed, on one end, into a closed loop about 50 mm smaller in diameter than the inside diameter of the specimen holder with the remainder bent perpendicular to

the loop so that it can be connected to the transformer to conduct the voltage into the electrolyte (the salt solution).

**6.6 Water Bath**—A circulating water bath; provided with heaters and temperature controls if tests are to be made at elevated temperatures.

**NOTE 1**—Circulation of the solution in the bath even at room temperature is necessary to remove gas bubbles formed at the interface of the solution and the test specimens caused by electrolysis.

**6.7 Microscope**—A microscope equipped for 20 and 100 $\times$  magnification.

## 7. Reagents

**7.1 Salt**—Reagent-grade sodium chloride.

**7.2 Sealants**—The material used for sealing in this test method is a two-part silicone rubber sealant consisting of a base<sup>6</sup> and a catalyst<sup>6</sup>.

**7.3 Multipurpose Silicone Sealant**—One-part silicone<sup>6</sup> rubber sealant.

**7.4 Staining Dye**—The staining dye is a mixture of the methylene blue and sodium hydroxide.

**7.5 Deionized Water**, or distilled water.

## 8. Hazards

**8.1 Warning**—Lethal voltages are a potential hazard during the performance of this test method. 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.

**8.2** Solidly ground all electrically conductive parts that may be possible for a person to contact during the test. Provide

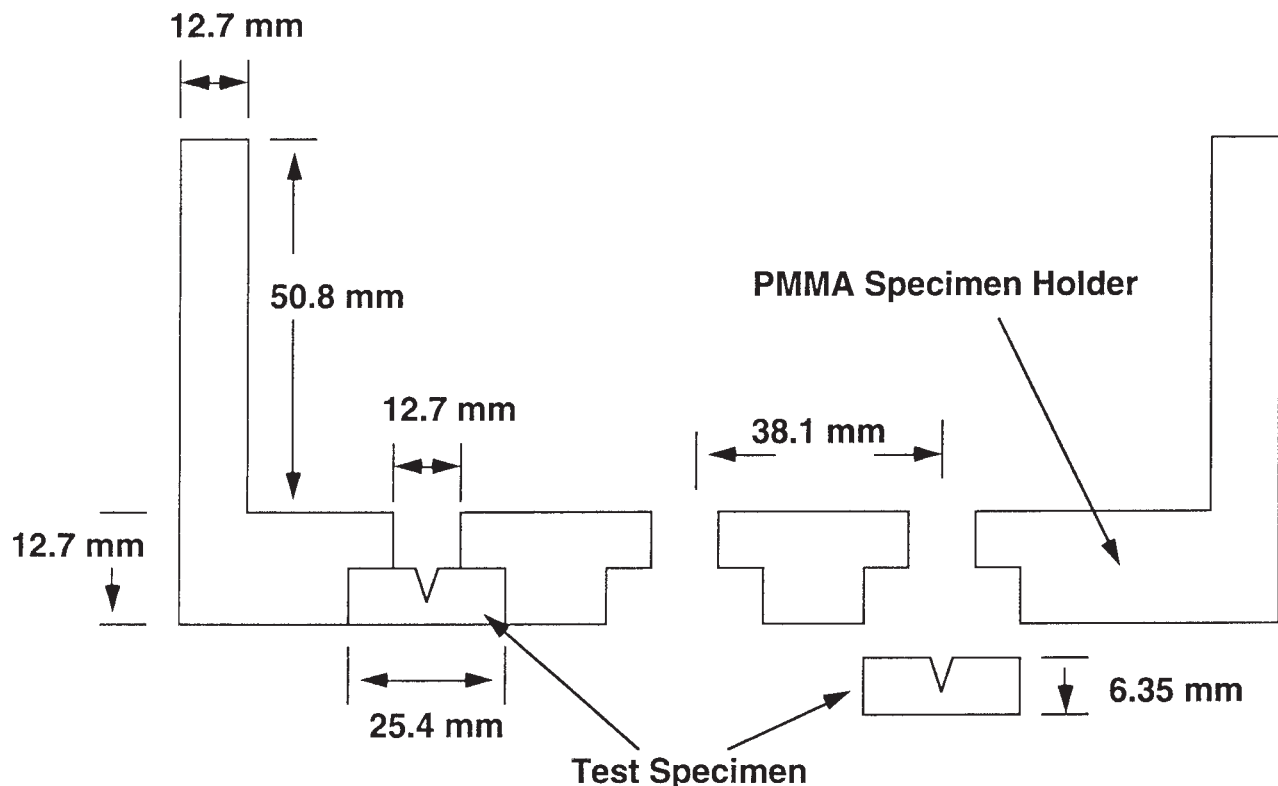


FIG. 2 PMMA Specimen Holder

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 high-voltage 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.

NOTE 2—**Warning**—Water in the test tank is gradually evaporated. Keeping the water level constant is important to prevent an electrical hazard.

## 9. Sampling

9.1 Sample in accordance with Practice D 1898.

## 10. Test Specimen

10.1 *Geometry of Test Specimens*—The test specimen is a disk containing a conical defect at the center of one side. The disk has a diameter of 25.4 mm and a thickness of 6.35 mm. This conical defect has a diameter of 3.2 mm and height of 3.2 mm with an included angle of  $60^\circ$ . The radius of the cone tip is  $3 \pm 1 \mu\text{m}$ . Fig. 3 is the geometry of the test specimen.

10.2 *Preparation of Test Specimens*—Compression mold ten specimens for each solid dielectric material using the preparation method described in Practice D 1928. Use a pre-drilled polyethylene terephthalate sheet over needles to cover the metal surface of the bottom section of the test specimen mold to prevent cross contamination from the previous material residue. Apply a colorless mold release agent to all surfaces of the center section of the mold, to prevent cross contamination from the previous material residue. The mold release agent should not contain grease, wax, or silicone oil. Weigh a sufficient amount of each sample and fill the mold with the material. Cover the material with a polyethylene

terephthalate sheet under the top test specimen mold plate. Put the mold assembly together, and place the entire mold assembly in a hydraulic press and complete the molding cycle.

10.3 *Molding Conditions*—For thermoplastic polyethylene, the molding cycle is 5 min at a low pressure of 0.30 MPa, 2 min at a high pressure of 3 MPa at  $160 \pm 5^\circ\text{C}$ . For cross-linked polyethylene, the mold cycle is 5 min at  $125 \pm 5^\circ\text{C}$  at a low pressure of 0.30 MPa, 2 min at  $120 \pm 5^\circ\text{C}$  at a high pressure of 3 MPa, and 15 min at  $175 \pm 5^\circ\text{C}$  at the same high pressure. Cool the mold in the press at  $15^\circ\text{C}/\text{min}$  to ambient temperature. See Practice D 1928. Different materials may have different conditions for molding. For materials other than polyethylene, obtain molding conditions from the material supplier.

10.3.1 Remove the mold assembly from the press and take off the top plate. Slowly lift the center section of the mold, containing specimens, away from needles. Be careful not to drag material across needle tips. Remove the test specimens from the mold using a 25.4-mm diameter punch.

10.4 *Peroxide Cross-linked Materials*—Heat in a vacuum oven at a temperature of  $80 \pm 3^\circ\text{C}$  and an absolute pressure of 133 Pa (1 mm Hg) or less for 160 to 168 h. When releasing the vacuum do so with nitrogen instead of air. This step is necessary to remove by-products of the peroxide decomposition, some of which are known to be tree-retardants.

10.5 Do not use test specimens that have visible bubbles or cracks. It is also desirable that the flat surfaces on both sides of each disk are smooth. A dummy or trial sample could be used to measure and report the actual dimensions of the built-in protrusion. This step will take into account the difference in shrinkage response between different polymeric materials.

## 11. Procedure

11.1 *Sealant Preparation*—Prepare a mixture of silicone rubber encapsulant and its catalyst at a manufacturer's suggested ratio in a paper cup. Approximately 2.5 g of this mixture are needed for each specimen. Stir well. Place this sealant mixture in a vacuum desiccator and pump for 20 min to remove air from the sealant. Then pour the mixture into a small, wide-mouthed squeeze bottle that has a screw-on cap with a narrow opening at the top.

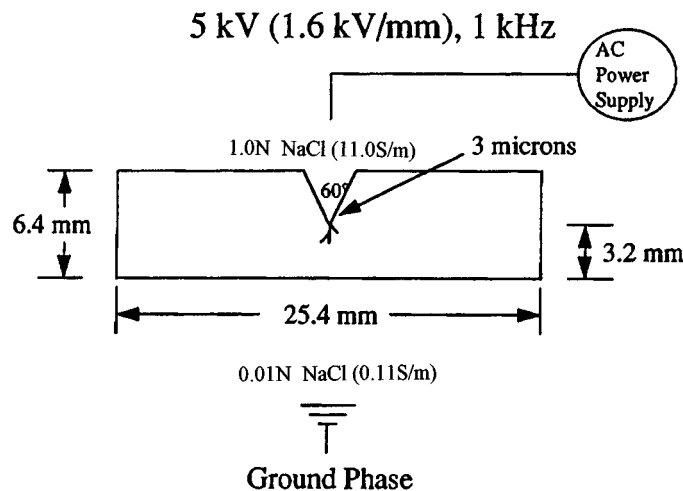


FIG. 3 Water-Tree Growth Test Specimen



**11.2 Mounting Test Specimens in the Specimen Holder—**Wipe the inside and outside surfaces of the specimen holder with isopropyl alcohol only (some solvents can cause PMMA to crack). Squeeze about 2.5 g of the sealant into the inner lip of the holder, and lightly press each specimen facing down into each hole. After all specimens are in place, let the specimen holder stand overnight so that the sealant can cure. After overnight cure, carefully remove the excess sealant from the bottom of the sample holder. Squeeze a sufficient amount of multipurpose sealant around the edges of the underside of each test specimen. This sealant should overlap the test specimen and the test specimen holder, completely covering the interface to ensure water tightness. Remove any sealant that is more than 3 mm from the outer edge of the test specimen.

**11.3 Conditioning—**After all test specimens are tightly mounted in the sample holder, store the holder for at least 24 h at 23°C and  $50 \pm 5$  % relative humidity before testing.

**11.4 Electrolyte Solution Preparation—**Prepare the electrolyte solution by dissolving  $2.92 \pm 0.1$  and  $292.3 \pm 0.1$  g of NaCl respectively in 5 L of deionized or distilled water at 20°C for electrode solutions of 0.01 and 1.0 N NaCl. The conductivities of 0.01 and 1.0 N NaCl solutions at 20°C are 0.11 and 7.78 S/m, respectively. The 1.0 N solution is used on the high-voltage side with the defect to accelerate water-tree growth, while the 0.01 N solution is used to provide sufficient conductivity for efficient grounding of the low-voltage side of the specimen.

**11.5 Placing Specimen Holder in a Water Bath—**Rinse the surfaces of specimens with deionized or distilled water and blot up excess. Place the test specimen holder in the water bath and fill the bath with the electrolyte solution until the level is  $13 \pm 3$  mm above the bottom of the specimen holder. The solution level and electrolyte concentration shall remain constant by refilling with deionized or distilled water. A water-level controller is recommended to maintain the constant level of electrolyte in the water bath to compensate for water evaporation. Use a rubber squeeze bulb attached to a short length of flexible tubing to remove all air bubbles from the underside of the sample holder. Use a small rubber squeeze bulb connected to a short length (10 mm) of narrow flex tubing, by a rigid glass tube about 150 mm long to carefully remove all traces of air from inside the test area of each specimen by forcing the electrolyte into the needle depression. This ensures the proper wetting of the specimen surface.

**11.6 Electrical Test Conditions—**Place the nickel-chromium wire electrode assembly into the center of the sample holder. Make sure that the loop is totally immersed in the electrolyte, and place the PMMA cover on the sample holder to reduce water evaporation. This wire electrode transfers the voltage into the electrolyte inside the PMMA specimen holder. The electrolyte in the water bath is the ground phase. The water bath should be connected to the ground. Otherwise, place a copper plate in the water bath and connect to the ground. Connect the wire electrode to a 1-kHz power supply and inspect all connections including the ground. Set the frequency at 1 kHz, and raise the test voltage from 0 to 5 kV. Maintain this test condition for 30 days. At the end of the test period, lower the voltage to zero, turn off the power supply, and

disconnect and remove the electrode. Take the test specimen holder from the bath, pour out the electrolyte solution, and remove the test specimens from the specimen holder.

**11.7 Staining Treed Specimens—**Stain the aged test specimens by the following procedure immediately after they are removed from the sample holder. Prepare Parts A and B of the staining solution in separate flasks. Part A consists of 2.5 g of methylene blue in 1000 mL of distilled water. Part B is 4.0 g of sodium hydroxide (NaOH) in 100 mL of distilled water. Mix nine parts A to one part B in a beaker. Stir this mixture using a magnetic stirring rod and heat it to its boiling point (about 100°C) on a combination magnetic stirrer/hot plate. For materials with melting points lower than the boiling point of the sodium hydroxide/methylene blue solution, the water temperature should be adjusted to about 10°C below the melting point. Identify test specimens by notching or scribing, and put them into the boiling solution. Adjust controls to maintain a constant boiling and stirring, and let test specimens remain in the boiling staining solution for at least 2 h. At a lower temperature (such as 60°C), staining time shall be increased to at least 6 h. After staining, remove the test specimens from the solution and rinse first with cold water then twice with acetone (use in a well-ventilated area) to remove any staining residue from the surfaces.

**11.8 Slicing and Slide Preparation—**(See Fig. 4.) Separate the stained test specimens in accordance with the material identification. Use either a sharp knife or other appropriate sharp tool to trim each test specimen to a rectangle approximately 6.35 mm with a thickness of 0.25 to 0.65 mm containing the full needle depression. Under an illuminated magnifier and using a sharp single-edge razor blade or microtome, slice inward toward the center of the depression. Examine each thin slice under an illuminated microscope of sufficient magnification to clearly show the needle depression and water tree. The object is to obtain a thin slice at the exact center of the depression which contains the needle tip and tree growth. Trim slices and place them on a 25 by 76-mm frosted end glass microscope slide on which has been written any pertinent information such as the material identification, test date, and so forth. Prepare all ten specimens in a like manner and maintain in a numerical order. Spread a small amount of a histological mounting medium over a 25-mm square area in the center of the slide. Immerse slices in a histological mounting medium in proper numerical order and arrange the slices neatly. Place a 25-mm square cover glass over the slices and press down slightly to expel the excess histological mounting medium and any trapped air. Using a very fine wire, dipped frequently in toluene (use in a well-ventilated area), arrange slices neatly under a cover glass. Carefully remove excess mounting medium from the slide surface with a toluene-saturated cotton-wool swab. When all slides have been prepared, allow the histological mounting medium to cure overnight.

**11.9 Tree Measurement—**Place each prepared slide under an illuminated profile projector and set at 100× magnification. If the projector is not available, all measurements may be made on a standard microscope equipped with a calibrated reticle. Uncertainty as to the exact location of the tip of the defect is

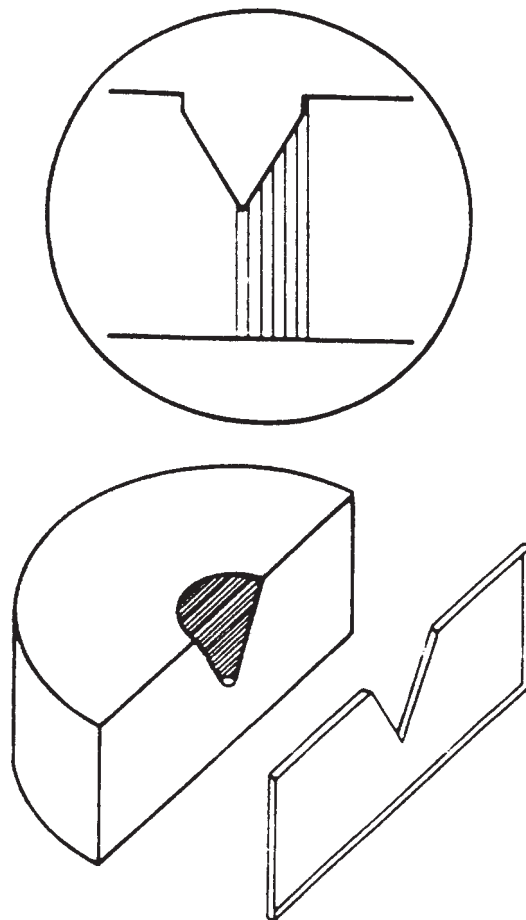


FIG. 4 Sectioning of Stained Test Specimen

sometimes a problem with such a measurement. Place a  $100\times$  transparency (1 cm = 0.1 mm) of the needle profile, that has been superimposed on a calibrated grid, over the lighted screen of the projector. Adjust the specimen slide so that the projected image of the needle depression coincides with the profile on the transparency. Measure the longest tree length along the axis of the needle tip and record the measurement of the water-tree length in millimetres. Fig. 5 is typical of water-tree growth in polyethylene. Change the magnification to  $20\times$  (1 cm = 0.5 mm), and measure the distance from the projected needle tip to the opposite surface of the test specimen as the point-to-plane test specimen thickness. Record this measurement as thickness in millimetres.

## 12. Calculation

12.1 Measure the water-tree length and point-to-plane specimen thickness of each test specimen, and calculate their average and standard deviation for each material.

12.2 Calculate the resistance to water-tree growth, RWTG, by dividing the average point-to-plane specimen thickness by the average water-tree length.

## 13. Report

13.1 Report the following information:

13.1.1 Description and identification of the test material (such as thermoplastic or cross-linked),

13.1.2 Number of test specimens for data reporting and date of test,

13.1.3 Conditions prior to testing (particularly any vacuumed at  $80^{\circ}\text{C}$ , seven days),

13.1.4 Test conditions (bath temperature, frequency, electrolyte on high voltage and ground side),

13.1.5 Radius of needle tip included angle,

13.1.6 Aging time,

13.1.7 Staining temperature and time.

13.1.8 For each test specimen:

13.1.8.1 Measured water-tree length,

13.1.8.2 Measured point-to-plane test specimen thickness, and

13.1.8.3 Description of tree shapes (vented, bent, split, and so forth).

13.1.9 For each material:

13.1.9.1 Average water-tree length and its standard deviation,

13.1.9.2 Average point-to-plane test specimen thickness and its standard deviation, and

13.1.10 Resistance to water-tree growth RWTG (30 days, 5 kV, 1 kHz).

## 14. Precision and Bias

14.1 *Precision:*



FIG. 5 Typical Water-Tree Growth of Polyethylene

14.1.1 The precision of tree length and point-to-plane distance measurement is at least 0.01 mm. The tree length measurement has the percent coefficient of variance of less than 15 %. The point-to-plane specimen thickness measurement has the percent coefficient of variance of less than 5 %. As a result, the resistance to water-tree growth, RWTG, has a percent coefficient of variance within 15 %.

14.1.2 The tip radius of the needle used to create a controlled specimen defect is extremely sharp and can vary from 2 to 4  $\mu\text{m}$  (inspection of needles for damage should be made and damaged needles removed). This possible wide variation

in the tip radius does not affect the tree growth data. It may only affect the tree initiation time that is not measurable in this test method. However, the tree initiation time may not be negligible for the materials with a very high resistance to water-tree growth.

14.2 *Bias*—This test method has no bias as the values reported are determined solely in terms of this test method.

## 15. Keywords

15.1 electrical breakdown; solid dielectrics; treeing; water-treeing

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