

# Methods of test for the evaluation of water treeing in insulating materials

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# National foreword

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**Méthodes d'essai pour l'évaluation de  
la formation des arborescences d'eau  
dans les matériaux isolants**

**Methods of test for the evaluation of  
water treeing in insulating materials**



Commission Electrotechnique Internationale  
International Electrotechnical Commission  
Международная Электротехническая Комиссия

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## INTERNATIONAL ELECTROTECHNICAL COMMISSION

METHODS OF TEST FOR THE EVALUATION OF WATER TREEING  
IN INSULATING MATERIALS

## FOREWORD

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Technical specifications are subject to review within three years of publication to decide whether they can be transformed into International Standards.

IEC 61956, which is a technical specification, has been prepared by subcommittee 15E: Methods of test, of IEC technical committee 15: Insulating materials.

The text of this technical specification is based on the following documents:

|               |                  |
|---------------|------------------|
| Enquiry draft | Report on voting |
| 15E/106/CDV   | 15E/123/RVC      |

Full information on the voting for the approval of this technical specification can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 3.

Annex A is for information only.

The committee has decided that this publication remains valid until 2005.

At this date, in accordance with the committee's decision, the publication will be

- reconfirmed;
- withdrawn;
- replaced by a revised edition, or
- amended.

## INTRODUCTION

Water treeing is one of the major causes of premature ageing and failure of extruded medium voltage cables which do not have water-impervious barriers. Besides technological efforts, it has been a challenge up to now to overcome such failures by improved insulating compounds, mainly on the basis of chemically crosslinked low density polyethylene (XLPE). Consequently, a need for suitable material test methods has arisen; primarily for a meaningful screening test to evaluate the water tree propensity of such materials, and thus reduce the need of cable testing to compare and preselect materials.

On the other hand, a material test generally cannot simulate all the influences of the manufacturing parameters on the extruded cable insulation system. Therefore, cable tests will still remain necessary for the final assessment of new insulating compounds.

The CIGRE Working Group "Solid insulating materials" considered the merits of existing material tests [3] to [8]\* concerning their practicability and reliability. The Task Force on "Water tree testing of materials" was set up to evaluate pre-selected test methods [6], [7], [8] based on different philosophies and to prepare a proposal to the IEC as a basis for a test method. Experts in the field of water treeing, coming from industrial and scientific laboratories worldwide, participated in this work [1], [2]. A final report of the Task Group is published in *Electra* [17]. A summary of this report is given in annex A.

Although the evaluated three test variants, which were improved and partly combined, are not yet ready for international standardization, they are made available by this technical specification to enable interested laboratories to gain experience with them.

Method I subjects easily producible plaque type test specimens to a divergent (variant A) or a uniform (variant B) a.c. electric stress while immersed in a wet environment. A newly developed test cell allows to evaluate either vented water trees (vt) in front of indentations (A) or bow tie water trees (btt) at imperfections (B) within the test specimen, or both types simultaneously.

Method II ages cup-shaped test specimens, made of insulating material sandwiched between two layers of semiconducting screen material, under uniform a.c. electric stress while exposed to a wet environment. In addition to microscopic inspection of water trees (vt and btt), this method enables an evaluation by electrical breakdown tests.

The two methods are mutually complementary as they permit the relative assessment of water treeing propensity to be determined for either the insulation alone or for the insulation/screen combination. In annex A, results of round robin tests achieved with these methods by the CIGRE Task Force are reported.

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\* Figures in square brackets refer to the bibliography.

## METHODS OF TEST FOR THE EVALUATION OF WATER TREEING IN INSULATING MATERIALS

### 1 General

#### 1.1 Scope

This technical specification describes methods of test for the evaluation of water treeing in polyethylene (PE) and crosslinked polyethylene (XLPE) compounds, as a means to assess their relative performance under alternating (a.c.) electric stress, in the presence of water. Two methods are described, method I covering the evaluation of insulating materials alone, and method II covering the evaluation of insulating sandwiches consisting of an insulating material in intimate contact with semiconducting screens.

#### 1.2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this technical specification. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this technical specification are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of IEC and ISO maintain registers of currently valid International Standards.

IEC 60243-1:1998, *Electrical strength of insulating materials – Test methods – Part 1: Tests at power frequencies*

IEC 61072:1991, *Methods of test for evaluating the resistance of insulating materials against the initiation of electrical trees*

#### 1.3 Definitions

Water treeing is a degradation process [11] as observed in low density polyethylene (LDPE) and XLPE under a.c. stress and moist conditions, which results in the formation of dielectrically weakened areas of so-called water trees.

Water trees are hydrophilic dendritic, tree-like features (specifically, they appear initially to be chains of water-filled cavities which later become bushes of microscopic channels with hydrophilic surfaces), which grow typically under wet and electrical operating conditions and may reach lengths of the order of 1 mm within several years. Two different types of water trees can be distinguished:

- a) Bow tie tree resembles a bow tie and consists of divergent straight branches radiating in opposite directions from a central point. The bow tie trees, contained within the bulk of the insulation, are generally lined up in the direction of the electric field.
- b) Vented tree resembles a tree whose trunk is vented to the surface of the insulation or the insulation/screen interface. The branches are generally oriented in the direction of the electric field, away from the insulation surface or the interface.

## 2 Test method I (plaque test)

### 2.1 Principle

The plaque tests (variant A and variant B) are intended to assess the development of water trees in low density polyethylene (LDPE) and crosslinked polyethylene (XLPE) based insulating materials.

Both test variants make use of plaque-shaped test specimens and of the same test cell. Both tests are screening tests to differentiate and to preselect insulating compounds with regard to water treeing.

The test with stress concentrations (variant A) is primarily intended to assess the development of vented trees from the tips of protrusions of the screen or intrusions in the insulation at the insulation/screen interface, simulated by needle-shaped and water-filled indentations. A plaque with a number of identical indentations is exposed simultaneously to water and to an electric field, producing electric field stress concentration at those points, within a test cell according to 2.3.1.

By means of this test, bow tie treeing can be additionally assessed within the bulk of the test specimen, away from the stress concentration areas.

The test with uniform field stress (variant B) is intended to assess the development of bow tie trees, when a plaque is exposed simultaneously to water and to a uniform electric field within a test cell described in 2.3.1.

### 2.2 Test specimen

Test specimens in the form of disc-shaped slabs with a diameter of  $(35 \pm 1)$  mm can be achieved by punching them out from plaques of one of the following thicknesses:  $(4,0 \pm 0,1)$  mm;  $(3,0 \pm 0,1)$  mm;  $(2,0 \pm 0,1)$  mm. Comparison between materials should be made at equal thicknesses only.

For vented tree testing, plaques may be manufactured from pellets by pressmolding. For bow tie tree testing, it is recommended to homogenize the compound by extrusion in order to avoid concentration of additives and impurities on the surface of the pellets. In every case extreme care should be taken to prevent contamination of the materials as well as of the resulting plaques and slabs.

In the case of XLPE based compounds (using dicumyl-peroxide) the plaques are preformed within the press frame at circa 130 °C. They are heated up to 180 °C, maintained for 30 min and cooled down, all under pressure, to circa 70 °C. The plaques taken out of the frame are annealed for 72 h at  $(90 \pm 2)$  °C to evaporate the volatile by-products.

In the case of LDPE based materials (without crosslinking agent) the plaques are also preformed at circa 130 °C within the press frame, heated up to circa 200 °C and then cooled down, both under pressure, to circa 70 °C.

Experience shows that when making plaques from XLPE or PE based compounds, a press force of at least 5 N/mm<sup>2</sup> of plaque gives satisfactory results.

## 2.3 Test apparatus

### 2.3.1 Test cell

The test cell [10], shown in figure 1, comprises:

- a cup, made of high density polyethylene (HDPE), with a circular opening at its bottom;
- a ground electrode assembly including a HDPE base support;
- six nylon screws to compress the test specimen between the cup and the ground electrode;
- a transparent plastic cover through which the high voltage lead is installed. This lead shall be made of a noble metal, such as palladium, platinum or other.

A dimensional drawing of the test cell is shown in figure 2 (different constructions and materials can be used, as long as the principle of the test is preserved; care should be taken in the choice of materials to avoid leaching of soluble contaminants from the material into the test solution).

### 2.3.2 Electrodes

The cup of the test cell is intended to contain a NaCl solution (see 2.3.3) which will constitute the high voltage electrode. The lead to be submerged in the NaCl solution should be made of a noble metal such as platinum or palladium.

The ground electrode is a circular brass electrode having a flat upper surface, 20 mm to 25 mm in diameter, with rounded edges according to figure 1.

### 2.3.3 Test liquid

The NaCl solution (1,8 mmol/litre) is obtained by dissolving 0,1 g of NaCl in 1 l of distilled and deionized water.

This kind of water is prepared by passing distilled water, made in a non-glass apparatus, through a mixed bed deionizing column and storing it in an airtight polyethylene container. The water shall be tested immediately prior to use for pH ( $7,0 \pm 0,1$ ) and conductivity ( $\sigma \leq 100 \mu\text{S/m}$ ).

### 2.3.4 Needle holder – plunger assembly

The needle holder – plunger assembly is shown in figure 3. It is used specially for the test with stress concentration to perform simultaneously eight needle tip indentations, two rows in parallel, four needles in each row, in the middle of the upper surface of the test specimen. The eight needle tips, protruding ( $0,5 \pm 0,05$ ) mm from the needle holder surface are fixed each by two set screws to ensure their correct position.

The needles according to IEC 61072 shall be made of stainless steel and have a tip radius of ( $4 \pm 1$ )  $\mu\text{m}$  and an included angle of  $30^\circ$ , the preferred overall diameter is 0,7 mm to 1,0 mm <sup>1)</sup>.

Prior to use, the needles shall be cleaned and dried, taking care not to damage the needle tip. Each needle shall be inspected for tip radius and shape after cleaning, before and after being fixed in the holder. Extreme care shall be exercised to ensure that the needle tips are free from corrosion products.

<sup>1)</sup> Such needles may be obtained by suppliers as Ogura Juwel Industry Co. Ltd. 7-12 Omori Kita 5, Chome, Ota-ku, Tokyo 143, Japan.

Needle holder and plunger are assembled as follows (see figure 3): first the needle holder is installed on the mounting plate using the needle holder mounting screws. Next, the assembled needle holder is connected to the plunger by means of the connecting screw. After having tightened the screw, the plunger is lifted up in order to line up the upper surface of the needle holder mounting plate with the bottom surface of the cell-top guide. Any gap between the mounting plate and the cell-top guide can be eliminated by adjusting the tightness of the screw. A detailed drawing of the needle holder is shown in figure 4.

## **2.4 Test procedure (ageing test)**

### **2.4.1 Test procedure with uniform field stress (variant B)**

The circular test specimen is fitted in the test cell. The six nylon screws are tightened evenly and gradually by working successively on diametrically opposite screws without using excessive torque.

The cup of the test cell is filled to three-quarters with the test liquid (see 2.3.3) and capped with the cover that contains the high voltage lead bushing. The lower part of the test cell, including the test specimen, is immersed in silicone oil to suppress surface discharges. The high voltage lead is connected with the a.c. voltage source (48 Hz to 62 Hz), the ground electrode to ground. The resulting average electric field stress should be 5 kV r.m.s./mm, for example 20 kV r.m.s. in case of a slab 4 mm thick.

The preferred ageing conditions are 240 h at room temperature. Other field stresses, ageing times and temperatures are possible.

### **2.4.2 Test procedure with stress concentration (variant A)**

The cup of the test cell is prepared as in 2.4.1. To make the indentations into the slab, the test cell (without cover), the needle holder-plunger assembly, 10 ml of the NaCl solution and a 1 000 g weight are placed into an oven preheated to 60 °C. After 15 min, the NaCl solution is poured into the cup, the plunger assembly is placed in the recessed area of the cell after pulling the plunger to its final position. Now the plunger is caused to slide down gently until the needle tips rest on the surface of the test specimen. The weight is carefully placed on top of the plunger handle and the oven is closed. After 1 h, the weight is removed, and the cell with the plunger is taken out of the oven. Cooling to room temperature needs approximately 30 min. After removing the plunger assembly, the cup of the test cell is filled to three-quarters with NaCl solution. The needles are immediately cleaned in distilled water, blown dry and stored in a dry area to prevent corrosion.

The test cell is covered, connected to high voltage and to ground and the test specimen is aged as described in 2.4.1.

## **2.5 Examination after ageing**

### **2.5.1 Microscopic inspection regarding bow tie trees (variant B)**

At the completion of voltage ageing the test specimen is subjected to an examination for water trees. The test specimen is microtomed perpendicular to the water aged surface into a number of slices, 20 mm long and 0,1 mm to 0,2 mm thick; 20 evenly distributed slices are examined. All slices shall be dyed, using procedures described in [13]. The dyed slices are examined with an optical microscope at  $\times 100$ . The maximum length of the observed bow tie trees in each slice is measured. Their density is determined, taking into account only those larger than 50  $\mu\text{m}$  (both wings included). As the number of trees observed in a material is high, only 1/5 of the total area of each slice is examined. The average density of trees exceeding 50  $\mu\text{m}$  in length for all slices examined is calculated and noted in the test report.