BS 6469 : Section 4.2 : 1992 IEC 811-4-2 : 1990

# Insulating and sheathing materials of electric cables

Part 4. Methods of test specific to polyethylene and polypropylene compounds

Section 4.2 Elongation at break after pre-conditioning — Wrapping test after pre-conditioning — Wrapping test after thermal ageing in air — Measurement of mass increase — Long-term stability test — Test method for copper-catalysed oxidative degradation

(Implementation of IID 505.4.2 S1)



# Committees responsible for this **British Standard**

The preparation of this British Standard was entrusted by the Cables and Insulation Standards Policy Committee (CIL/-) to Technical Committee CIL/20, upon which the following bodies were represented:

Aluminium Federation

Association of Consulting Engineers

Association of Manufacturers of Domestic Electrical Appliances

British Approvals Service for Cables

British Cable Makers' Confederation

**British Plastics Federation** 

British Steel Industry

British Telecommunications plc

Department of the Environment (Property Services Agency)

Department of Trade and Industry (Consumer Safety Unit, CA Division)

**Electricity Association** 

Engineering Equipment and Materials Users' Association

Institution of Electrical Engineers

London Regional Transport

The following bodies were also represented in the drafting of the standard, through subcommittees and panels:

British Railways Board

British Rubber Manufacturers' Association Ltd.

ERA Technology Ltd.

GAMBICA (BEAMA Ltd.)

Institution of Incorporated Executive Engineers

London Underground Ltd.

Queen Mary and Westfield College

Telecommunications Cables Group of BCMC

This British Standard, having been prepared under the direction of the Cables and Insulation Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 15 August 1992

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# National foreword

This Section of BS 6469 has been prepared under the direction of the Cables and Insulation Standards Policy Committee. BS 6469: Parts 1 to 5 and Part 99 supersede BS 6469: 1990 which is withdrawn.

Parts 1 to 5 implement CENELEC HD 505: Parts 1 to 5. Part 99 describes test methods having national applicability only.

The International Electrotechnical Commission has completed its comprehensive update of the test methods previously given in IEC 538, IEC 538A and IEC 540, which are now largely brought together in IEC 811. Electrical tests from IEC 540 have been incorporated into IEC 885. The technical changes introduced during this update, and endorsed by CENELEC in HD 505, are now included in BS 6469.

BS 6469: Section 4.2 includes a number of test methods described in BS 6234. Other test methods from BS 6284, from BS 6746 and from BS 6899 have been incorporated in BS 6469: Part 99. The status of these test methods in BS 6234, BS 6746 and BS 6899 will be reviewed separately.

BS 6469 describes methods of test, but does not specify requirements for products or materials. These will be specified in the relevant cable standards or cable material standards.

This Section of BS 6469 implements CENELEC Harmonization Document HD 505.4.2 S1: 1992, which is identical with IEC 811-4-2: 1990.

Definitions of terms relating to electric cables are given in BS 4727 ; Part 2 ; Group 08.

It has been assumed in the preparation of this British Standard that the execution of its provisions will be entrusted to appropriately qualified and experienced people, for whose use it has been produced.

warning. The methods of test described in this British Standard do not necessarily detail all precautions necessary to meet the requirements of the Health and Safety at Work etc. Act 1974. Attention should be paid to any appropriate safety precautions and the tests should be carried out only by trained personnel.

Cross-references between the relevant clauses in BS 6469: 1990 and those in BS 6469: Parts 1 to 5 and Part 99 are given in table NB.1. Tests included in BS 6469: Sections 1.3 and 5.1 which were not given in BS 6469: 1990 are listed in table NC.1.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

# HARMONIZATION DOCUMENT DOCUMENT D' HARMONISATION HARMONISIERUNGSDOKUMENT

HD 505.4.2 S1

May 1992

UDC 621.315.6-036.742.2/.3:620.193.2

Descriptors: Electric cable, insulated cable, electrical insulation, outer sheath, polyethylene, polypropylene, test method, elongation at break, stability test, oxidation resistance, copper

### English version

# Common test methods for insulating and sheathing materials of electric cables

Part 4. Methods specific to polyethylene and polypropylene compounds Section 2 — Elongation at break after pre-conditioning — Wrapping test after pre-conditioning — Wrapping test after thermal ageing in air — Measurement of mass increase — Long-term stability test (Appendix A) — Test method for copper-catalysed oxidative degradation (Appendix B)

(IEC 811-4-2: 1990)

Méthodes d'essais communes pour les matériaux d'isolation et de gainage des cables électriques Quatrième partie: Méthodes spécifiques pour les mélanges polyéthylène et polypropylène Section deux — Allongement à la rupture après préconditionnement — Essai d'enroulement après préconditionnement — Essai d'enroulement après vieillissement thermique dans l'air — Mesure de l'augmentation de masse — Essai de stabilité à long terme (annexe A) — Méthode d'essai pour l'oxydation catalytique par le cuivre (annexe B) (CEI 811-4-2 : 1990)

Allgemeine Prüfungen für Isolier-und Mantelwerkstoffe für Kabel und isolierte Leitungen
Teil 4: Besondere Methoden für Polyäthylen und Polypropylen Compounds
Hauptabschnitt Zwei: Reißdehnung nach Konditionierung — Wickelprüfung nach Konditionierung — Wickelprüfung in Luft — Messung der Masseaufnahme — Langzeit-Stabilitätsprüfung (Anhang A)— Prüfungen für kathalytsiche Oxydation durch Kupfer (Anhang B) (IEC 811-4-2: 1990)

This Harmonization Document was approved by CENELEC on 1991-12-10. CENELEC members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for implementation of this Harmonization Document on a national level.

Up-to-date lists and bibliographical references concerning national implementation may be obtained on application to the Central Secretariat or to any CENELEC member.

This Harmonization Document exists in three official versions (English, French, German).

CENELEC members are the national electrotechnical committees of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

## CENELEC

European Committee for Electrotechnical Standardization Comité Européen de Normalisation Electrotechnique Europäisches Komitce für Elektrotechnische Normung

Central Secretariat: rue de Stassart 35, B-1050 Brussels

BS 6469 : Section 4.2 : 1992

### **Foreword**

The CENELEC questionnaire procedure, performed for finding out whether or not the International Standard IEC 811-4-2: 1990 could be accepted without textual changes, has shown that no CENELEC common modifications were necessary for the acceptance as Harmonization Document.

The reference document was submitted to the CENELEC members for formal vote and was approved by CENELEC as HD 505.5.1 S1 on 10 December 1991.

The following dates were fixed:

- latest date of announcement of the HD at national level (doa) 1992-06-01
- latest date of publication of a harmonized national standard (dop) 1992-12-01
- latest date of
   withdrawal of
   conflicting national
   standards (dow) 1992-12-01

For products which have complied with the relevant national standard before 1992-12-01, as shown by the manufacturer or by a certification body, this previous standard may continue to apply for production until 1997-12-01.

Annexes designated "normative" are part of the body of the standard. In this standard, annex ZA is normative.

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BS 6469: Section 4.2: 1992

# COMMON TEST METHODS FOR INSULATING AND SHEATHING MATERIALS OF ELECTRIC CABLES

Part 4: Methods specific to polyethylene and polypropylene compounds
Section Two - Elongation at break after pre-conditioning Wrapping test after pre-conditioning Wrapping test after thermal ageing in air Measurement of mass increase - Long-term stability test (Appendix A) Test method for copper-catalysed oxidative degradation (Appendix B)

### Scope

This standard specifies the test methods for testing polymeric insulating and sheathing materials of electric cables for power distribution and telecommunications including cables used on ships.

This Section Two of Part 4 gives the methods for measurement of elongation at break after pre-conditioning, for wrapping test after pre-conditioning, for wrapping test after thermal ageing in air, for measurement of mass increase, for long-term stability test and for measurement of copper catalysed oxidative degradation, which apply to polyolefin insulations.

### 2. Test values

Full test conditions (such as temperatures, durations, etc.) and full test requirements are not specified in this standard; it is intended that they should be specified by the standard dealing with the relevant type of cable.

Any test requirements which are given in this standard may be modified by the relevant cable standard to suit the needs of a particular type of cable.

### 3. Applicability

Conditioning values and testing parameters are specified for the most common types of insulating and sheathing compounds and of cables, wires and cords.

### 4. Definitions

For the purpose of these tests, a distinction shall be made between low-density, medium-density and high-density PE:

Low-density polyethylene  $\leq 0.925 \text{ g/cm}^3$ Medium-density polyethylene  $> 0.925 \leq 0.940 \text{ g/cm}^3$  at 23 °C High-density polyethylene  $> 0.940 \text{ g/cm}^3$ 

Note. - These densities refer to unfilled resins as determined by the method specified in Clause 8 of Publication 811-1-3.

### 5. Type tests and other tests

The test methods described in this standard are primarily intended to be used for type tests. In certain tests where there are essential differences between the conditions for type tests and those for more frequent tests, such as routine tests, these differences are indicated.

Note. - For multicore cables and cords, not more than three cores (of different colours, if any) shall be tested unless otherwise specified in the relevant cable standard.

### 6. Pre-conditioning

All the tests shall be carried out not less than 16 h after the extrusion or vulcanization (or cross-linking), if any, of the insulating or sheathing compounds.

### 7. Median value

When several test results have been obtained and ordered in an increasing or decreasing succession, the median value is the middle value if the number of available values is odd, and the mean of the two middle values if the number is even.

### 8. Elongation at break after pre-conditioning

### 8.1 General

This test is intended for polyolefin insulations with a wall thickness of less than 0.8 mm of filled cables.

### 8.2 Pre-conditioning procedure

A sample of complete cable of sufficient length shall be preconditioned in air (i.e. suspended in an oven). The temperature of the air shall be maintained continuously at a temperature and duration as follows:

- 7 x 24 h at 60 °C for filling compound having a nominal drop-point above 50 °C and up to and including 70 °C.
- 7 x 24 h at 70 °C for filling compound having a nominal drop-point above 70 °C.

After pre-conditioning, the cable sample shall be left at ambient temperature for at least 16 h without being exposed to direct sunlight. Then the sheath shall be removed and the cores shall be cleaned by suitable means.

### 8.3 Apparatus

A tensile strength testing machine with grips either of a self-tightening type or of a non self-tightening type for tubular test pieces.

### 8.4 Sampling and preparation of test pieces

The ageing behaviour shall be tested on at least two test pieces.

A tube not less than 100 mm long is obtained from a wire, care being taken not to damage the insulation.

If withdrawal of the conductor is difficult, it should be stretched by any suitable means.

A length of 20 mm is marked by two parallel lines, centrally to each piece, immediately before the elongation test.

Note.- It should be emphasized that, in certain cases, such as that of stranded conductors having relatively thin insulation, it may be impossible to withdraw the conductor without damaging the insulation.

### 8.5 Elongation test after pre conditioning

Test pieces, pre-conditioned according to Sub-clause 8.2 and prepared according to Sub-clause 8.4, shall be subjected to an elongation test at ambient temperature. In case of doubt, the test shall be repeated at 23  $\pm$  2 °C.

The total length between the grips shall be about:

- 50 mm if tested with self-tightening grips;
- 85 mm if tested with non self-tightening grips.

The rate of separation shall be 25 ± 5 mm/min.

For routine tests, separation rates up to  $250 \pm 50$  mm/min are permitted.

### 8.6 Expression of results

The median of the values of elongation at break shall be recorded as the elongation at break.

### 9. Wrapping test after pre-conditioning

### 9.1 General

This test is intended for samples from filled cables of polyolefin insulation having a wall thickness of less than 0,8 mm.

### 9.2 Test procedure

The test shall be carried out in accordance with the method specified in Sub-clause 9.5 of IEC Publication 811-4-1 except that the ageing procedure shall be in accordance with Sub-clause 10.4 of this standard.

For cellular insulations having a wall thickness below or equal to 0.2 mm, the pull exerted on the exposed conductor shall be reduced to about  $7.5 \text{ N/mm}^2$  with respect to the conductor cross-section.

### 9.3 Evaluation of results

After cooling down to ambient temperature, the test pieces shall show no cracks when examined with normal or corrected vision without magnification. The test may be repeated once more if a test piece falls.

### 10. Wrapping test after thermal ageing in air

This test method shall be considered as an ageing method for polyolefin insulations and is therefore included in this section.

Note. - For cross-references this clause should be preferred to Clause 9 in Section One of IEC Publication 811-4-1, since Clause 9 will be deleted from Section One in the future.

### 10.1 General

This test is intended for polyolefin insulations of unfilled cables and of dry cores for filled cables having a wall thickness of less than 0.8 mm.

### 10.2 Apparatus

- 10.2.1 Smooth metal mandrel and loading elements.
- 10.2.2 Winding device, preferably with mechanically driven mandrel.
- 10.2.3 Electrically heated cabinet with natural air flow.

### 10.3 Sampling

The test shall be carried out on four test pieces for each length of cable or core to be tested.

Take a sample 2 m long and cut it into four test pieces of equal length.

Carefully remove the coverings and braidings, if any, from the test pieces and any filling compound which may adhere to the cores.

Leave the conductor within the insulation. Then straighten the test pieces.

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### 10.4 Ageing procedure

The test pieces prepared in accordance with Sub-clause 10.3 shall be suspended vertically for 14 x 24 h at 100  $\pm$  2 °C in the middle of the heating chamber in accordance with Sub-clause 10.2.3, so that each test piece is at least 20 mm from any other test piece. Not more than 2% of the chamber volume shall be occupied by the test pieces. Immediately after the ageing period, the test pieces are taken out of the chamber and left at ambient temperature, without being exposed to direct sunlight, for at least 16 h.

Note. The ageing time and/or ageing temperature may be increased if required by the relevant cable specifications.

### 10.5 Test procedure

Test pieces according to Sub-clause 10.3 shall be subjected, after ageing in accordance with Sub-clause 10.4, to a winding test at ambient temperature. For this purpose, the conductor shall be laid bare at one end. A weight shall be applied to the exposed conductor end, exerting a pull of about 15 N/mm²  $\pm$  20% with respect to the conductor cross-section. Ten windings shall be made on the other end of the test piece by means of a winding device in accordance with Subclause 10.2.2 on a metal mandrel at a speed of about one revolution/5 s. The winding diameter shall be 1 to 1,5 times the test piece diameter. Subsequently, the test pieces wound on the mandrel shall be removed from the latter and shall be kept in their helical form for 24 h at 70  $\pm$  2 °C in the vertical position, substantially in the middle of the heating chamber in accordance with Sub-clause 10.2.3.

### 10.6 Evaluation of results

After cooling down to ambient temperature the test pieces shall show no cracks when examined with normal or corrected vision without magnification. The test may be repeated once more if a test piece fails.

### 11. Mass increase of insulation

### 11.1 General

This test is used to examine possible interaction between insulation material and filling compound of filled cable. It is intended only for the purpose of material selection.

### 11.2 Sampling

Three samples of each colour of core are taken from a cable before the filling process. Each sample of about 2 m is cut into three pieces of length 600 mm, 800 mm and 600 mm.

### 11.3 Test procedure

The 800 mm test piece is immersed in about 200 g of filling compound contained in a glass vessel and pre-heated to the following temperature:

 $60 \pm 1$  °C for filling compound having a drop-point above 50 °C and up to and including 70 °C;

70 ± 1 °C for filling compound having a drop-point above 70 °C.

At least 500 mm of the middle part of this test piece shall be immersed in the compound without contact with the glass vessel or another specimen. The ends of the test piece shall be kept out of the compound.

The glass vessel shall be stored for  $10 \times 24$  h in an oven and the temperature shall be maintained continuously at the value specified above for the relevant filling compound.

At the end of this period, the test piece shall be removed from the filling compound and carefully cleaned with absorbent paper. Then the ends of the test piece shall be cut away leaving at least 500 mm of the middle part immersed in the filling compound. The two dry 600 mm pieces shall be cut back to the same length as the immersed test piece and the conductor shall be removed from all three. The three test pieces shall be weighed at ambient temperature to the nearest 0,5 mg.

### 11.4 Calculation

The mass increase W shall be determined as:

$$W = \frac{M_2 - M_1}{M_1} \times 100$$

where:

 $M_1$  = mean mass of the two dry test pieces.

 $M_2$  = mass of test piece immersed in the filling compound.

### APPENDIX A

### LONG-TERM STABILITY TEST

Note. This test method is only applicable to telecommunications cables. A similar test method applicable to electric cables for power distribution is under consideration.

### A1. General

The need to establish whether or not the quality of a cable's components will be satisfactory over the proposed life of the cable is well recognized. In particular, the polyethylene insulation must have sufficient resistance to ageing in service. For polyethylene-filled cables, the compatibility of the combination of the insulation and filling compound shall be assessed.

The definition of test duration, temperature, atmosphere and failure criteria shall be carefully chosen. One method found suitable for material selection is given in this appendix. The test duration makes the test unsuitable for routine quality control testing. Therefore, the test should be considered only as a material selection test to ensure that the chosen materials are satisfactory for the intended life of the cable.

For routine quality control purposes, short duration tests are required.

### A2. Apparatus

- A2.1 An air oven generally in accordance with ISO 188 and complying especially with the following criteria:
  - test temperature: 105 ± 1 °C

Note. - The test temperature should be further studied.

- exchange of clean, dry air: at least 6 changes of air per hour; in case of dispute, the maximum rate of change shall be 10 changes per hour.
- Alternatively, a testing apparatus consisting of one or more cells having the following dimensions may be used, provided that the above criteria are followed.

Cell height: Cell diameter:

at least 250 mm. at least 75 mm. between 3:1 and 4:1.

Height diameter ratio:

- A2.2 An air flow meter with a measuring range dependent on the size of the air oven according to Sub-clause A2.1.
- A2.3 A thermocouple or thermometer allowing a reading of 0,2 °C.
- A2.4 A balance accurate to ±0,0005 g and readable and repeatable to 0,1 mg.

### A3. Sampling

Three samples of each colour and 2 m in length shall be taken either from an unfilled or from a filled cable core. Each length constitutes a test piece.

### A4. Test procedure

### A4.1 For unfilled cables

- A4.1.1 The test piece shall be wound into a loose coil of about 60 mm diameter. There shall be no twists or kinks in the test piece. If necessary, the coil may be secured with two loose ties of aluminium wire.
- A4.1.2 The test piece shall be weighed to the nearest 0,1 mg and shall be suspended in the lower part of the air oven by means of an aluminium wire hook attached to the lid. A thermocouple or a suitable thermometer shall be used to check that the air temperature at the centre of the coil is maintained at  $105 \pm 1$  °C.

The three samples of each colour shall be tested. If an apparatus consisting of ageing cells is used, it would be preferable to age each test piece in a separate cell. However, if necessary, up to three test pieces may be aged together in one cell, provided they are suspended 3 to 5 mm apart so as not to touch each other or the cell wall.

- A4.1.3 At the end of the test period of 42 days, the test piece shall be removed from the air oven, cooled to ambient temperature and:
  - Visually examined for splits or cracks in the insulation and for other signs of polymer breakdown; the colours shall be readily identifiable.
  - The mass increase shall not be greater than 1 mg, when reweighed to the nearest 0,1 mg.
- A4.1.4 The test pieces, having undergone the procedure described in Sub-clause A4.1.3, shall then be subjected to the following test:

Five 200 mm lengths shall be cut from the test piece. These shall be cut at equidistant intervals, the first being taken at 0.2 m from one end of the test piece. One end of each 200 mm length shall be wound manually around the other end to give at least ten contiguous turns, and shall be visually examined for cracks and splits. The five test pieces so formed shall be suspended in an air-circulating oven at  $60 \pm 1$  °C for seven days.

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At the end of this period, the test pieces shall be visually examined for cracks and splits.

### A4.2 For fully filled cables

- A4.2.1 The test pieces shall be pre-conditioned for seven days in the associated filling compound at the following temperatures:
  - 60 ± 1 °C for filling compounds having a drop-point above 50 °C and up to and including 70 °C.
    - 70 ± 1 °C for filling compounds having a drop-point above 70 °C.
  - Note. For the definition of the drop-point, see Clause 4 of IEC Publication 811-5-1.

Pre-conditioning may be performed either on single samples by immersion in about 200 g of filling compound (except for the ends) contained in a glass vessel, or on a cable. If a cable is used, care shall be taken in removing the test pieces after pre-conditioning.

- A4.2.2 After pre-conditioning, the test pieces shall be carefully wiped free of excess filling compound using an absorbent lint-free tissue. The ends which were not immersed in the filling compound shall be discarded, and the test pieces shall be cut to length as specified in Clause A3.
- A4.2.3 The test procedure described in Sub-clauses A4.1.1 to A4.1.4 shall then be followed.

### APPENDIX B

TEST METHOD FOR COPPER-CATALYSED OXIDATIVE DEGRADATION OF POLYOLEFIN INSULATED CONDUCTORS (OIT-TEST)

### B1. General

The need for a manufacturer to monitor his cable production to ensure that it has adequate resistance to oxidation is well established. The OIT test has been found suitable for monitoring both raw materials and cables for compliance with this requirement, once suitable materials have been selected. The OIT test is not suitable for the selection of materials. For this purpose, long-term thermal ageing tests are preferred.

Having established the suitability of materials and material compatibility by a long-term stability test, the performance of the material may then be determined by the OIT test. To ensure material compliance with long-term stability behaviour, a relationship between the OIT test and the long-term stability test must be established.

This relationship is used to monitor materials and production, and may vary from laboratory to laboratory.

All insulation and insulation/filling compound combinations used in cable manufacture need to be assessed in this way.

An OIT testing procedure found suitable for testing copper-catalysed oxidative degradation is given in this appendix.

### B2. Apparatus

- B2.1 A differential thermal analyser or differential scanning calorimeter, capable of heating at rates of up to at least  $20 \pm 1$  K/min and of automatic recording of differences in temperature (or differences in heat transfer) between the sample and a reference material to the required sensitivity and precision.
- B2.2 A recorder capable of displaying heat flow or temperature difference on the Y-axis, and time on the X-axis. The time base must be accurate to ±1% and be readable to 1 min.
- B2.3 A gas-selector switch and regulators for high-purity nitrogen and oxygen.
- B2.4 An analytical balance capable of weighing 30 g, and readable and repeatable to  $\pm 0.1$  mg sample.

B2.5 Sample holders: aluminium holders, each of approximately 6 - 7 mm, or of similar dimensions as supplied by the manufacturer of the instrument.

### B3. Sampling

An appropriate number of test pieces of approximately 4 mm length containing the conductor are cut from an insulated conductor to yield 3 to 5 mg of insulating material.

### 84. Instrument calibration

- B4.1 Calibrate the instrument according to manufacturer's instructions before start of runs. Use analytical-grade indium as a temperature reference material.
- B4.2 Place 2 ± 0,5 mg of analytical-grade indium in an aluminium holder covered with an aluminium cover. Place the sample thus prepared, and a reference aluminium holder and cover in the instrument.

Should it be necessary to clean the sample and the aluminium reference holder and cover, use petroleum ether or other suitable solvent to remove contaminants.

- B4.3 Temperature-programme the scanner from 145 °C to 165 °C at a rate of 1 K/min, while recording the thermogram.
- B4.4 Calibrate the instrument according to manufacturer's instructions to obtain an indium first order transition temperature of 156,6 °C. For calibration purposes, the melting-point 156,6 °C is defined as the intersection of the extrapolated peak onset and the extrapolated baseline (see Figure B1).

### B5. Instrument preparation

- B5.1 Open valves on both the nitrogen and oxygen gas cylinders. Place gas selector switch to nitrogen  $(N_2)$  position and adjust flow rate to  $50 \pm 5$  ml/min using flowmeter.
- B5.2 Insert the wire sample in accordance with Clause B3 in an aluminium holder (see Sub-clause B4.2).
- B5.3 Place the prepared test piece of insulated wire in the sample holder of the instrument, and an empty aluminium holder in the reference position.
  - Note. Crimping the sample with a suitable aluminium or stainless steel screen is optional. It may provide a better contact with the sample holder.
- B5.4 Purge with nitrogen for 5 min. Check the flow rate and readjust to  $50 \pm 5$  ml/min if required.

- B5.5 Set the instrument at zero and set signal amplification and recorder sensitivity to the maximum pen deflection associated with the exothermic reaction.
- B5.6 Set heating rate to 20 K/min.
- B6. Test procedure
- B6.1 Commence programmed heating, and record the thermogram.
- B6.2 Continue heating until the specified test temperature, controlled to ±1 °C, is reached. Discontinue programmed heating and equilibrate the sample to a constant temperature. Test temperatures in the range of 190 °C 200 °C have been found appropriate for polyethylene.

Once temperature equilibrium has been established (steady recorder signal), change purge gas to oxygen, and adjust the flow rate to  $50 \pm 5$  ml/min. Mark this point on the recorder. This change-over point to oxygen purge is considered the zero time of the experiment  $(T_0)$ .

B6.3 Continue the isothermal operation until maximum pen deflection is attained after commencement of the oxidative exotherm as shown on the recorder (see Figure B2).

In the case of a multi-step exotherm, continue the isothermal operation until maximum pen deflection occurs.

- B6.4 When the test is completed, turn off the recorders and switch the gas selector back to nitrogen.
- B6.5 Allow the instrument to cool to the start temperature.
- B6.6 Repeat the entire test on a new sample four more times, thus generating a total of five thermograms. The use of a fresh aluminium reference holder for each sample is optional.

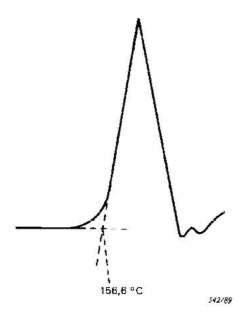
The procedure described in Sub-clauses B6.2 and B6.3 may be simplified by omitting the programmed pre-heating in N<sub>2</sub>. When this procedure is used, the instrument cell is heated to the required temperature in oxygen. First the reference holder is positioned in the cell, then, once the cell has attained the specified test temperature, the sample in its aluminium holder is placed in position. This point corresponds to the start of the test  $(T_{_{\rm O}})$ .

### B7. Calculation

B7.1 Extend the recorded base line from time zero beyond the oxidative exotherm. Extrapolate the steepest part of the exotherm to intercept the extended baseline (see Figure B2).

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- B7.2 The oxidative induction time (OIT) is measured from zero time to the smallest time interval practical, not exceeding 1 min.
- B8. Report
- B8.1 Identification of sample
- B8.2 Test temperature
- B8.3 Average and Standard deviation OIT of the five determinations in minutes.



Le point de fusion (156,6 °C) est défini comme l'intersection de la tangente prolongée de la première pente du pic endothermique et de la ligne de base extrapolée.

The melting-point (156.6  $^{\circ}$ C) is defined as the intersection of the extrapolated peak onset and the extrapolated baseline.

Figure B1 - Endotherme de fusion typique pour l'indium.

Representative melting endotherm for Indium.

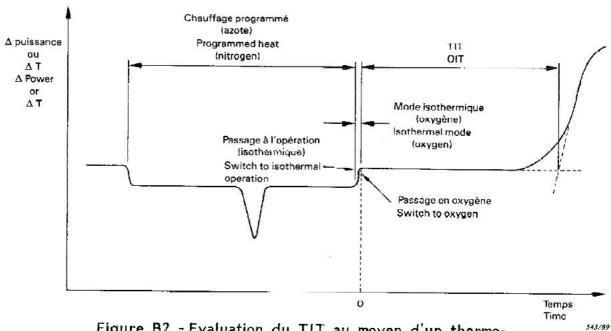


Figure B2 - Evaluation du TIT au moyen d'un thermogramme basé sur le temps enregistré. Evaluation of OIT from recorded-time-based thermogram.

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### ANNEX ZA (normative)

# OTHER INTERNATIONAL PUBLICATIONS QUOTED IN THIS STANDARD WITH THE REFERENCES OF THE RELEVANT EUROPEAN PUBLICATIONS

When the International publication has been modified by CENELEC common modifications, indicated by (mod), the relevant EN/HD applies.

| IEC Publication | Date | Title  | EN/HD          | Date |
|-----------------|------|--|----------------|------|
| 811-1-3         | 1985 | Common test methods for insulating and sheathing materials of electric cables  Part 1: Methods for general application  Section Three Methods for determining the density  Water absorption tests — Shrinkage test   | HD 505,1,3 S1  | 1988 |
| 811-4-1         | 1985 | Part 4: Methods specific to polyethylene and polypropylene compounds Section One — Resistance to environmental stress cracking — Wrapping test after thermal ageing in air — Measurement of the melt flow index — Carbon black and/or mineral filler content measurement in PE | HD 504,4,1 S1* | 1988 |
| 811-5-1         | 1989 | Part 5: Methods specific to filling compounds. Section One — Drop-point — Separation of oil — Lower temperature brittleness — Total acid number — Absence of corrosive components — Permittivity at 23°C — D.C. resistivity at 23°C and 100°C                                  | HD 505.5.1 S1  | 1992 |

### Other publications

ISO 188: 1982 - Rubber, vulcanized - Accelerated ageing or heat-resistance tests

<sup>\*</sup>Superseded by HD 505.4.1 S2: 1990, which is based on IEC 811-4-1: 1985 + A1: 1988

### National annex NA (informative)

### Cross-references

Publication referred to Corresponding British Standard BS 6469 Insulating and sheathing materials of electric cables IEC 811-1-3: 1985 Section 1.3: 1992 Methods for determining the density — Water absorption tests Shrinkage test IEC 811-4-1: 1985 Section 4.1: 1992 Resistance to environmental stress cracking — Wrapping test after thermal ageing in air - Measurement of the melt flow index - Carbon black and/or mineral content measurement in PE IEC 811-5-1: 1989 Section 5.1: 1992 Drop-point Separation of oil — Lower temperature brittleness - Total acid number - Absence of corrosive components - Permittivity at 23 °C - D.C. resistivity at 23 °C and 100 °C ISO 188: 1982 BS 903 Physical testing of rubber Part A19: 1986 Heat resistance and accelerated ageing tests

### National annex NB (informative)

| se in BS 6469 : 1990 BS 6469 : 1992  |                         |               |                   |                         |
|--|-------------------------|---------------|-------------------|-------------------------|
|  | Clause or<br>sub-clause | Part          | Section           | Clause or<br>sub-clause |
| General  | 1.1 to 1.3              | 1 to 4 and 99 | All<br>5.1        | 1 to 7<br>1 to 3        |
| Measurement of thickness and diameters                                     | 2.1                     | 1             | 1.1               | 8                       |
| Determination of tensile strength and elongation at break                  | 2.2                     | 1             | 1.1               | 9                       |
| Thermal ageing methods   | 2.3                     | 1             | 1.2               | 8                       |
| Methods for determining density  | 2.4                     | 1             | 1.3               | 8                       |
| Shrinkage test   | 2.5                     | 1             | 1.3               | 10                      |
| Gravimetric water absorption test  | 2.6                     | 1             | 1.3               | 9.2                     |
| Green/yellow proportions   | 2.7                     | 99            | 99.1              | 8                       |
| Mineral oil immersion test   | 3.1                     | 2             | 2.1               | 10                      |
| Ozone resistance test  | 3.2                     | 2             | 2.1               | 8                       |
| Hot set test   | 3.3                     | 2             | 2.1               | 9                       |
| Tear resistance  | 3.4                     | 99            | 99.1              | 9                       |
| Loss of mass tests   | 4.1                     | 3             | 3.2               | 8                       |
| Pressure tests at high temperature   | 4.2                     | 3             | 3.1               | 8                       |
| Tests at low temperature   | 4.3                     | 1             | 1.4               | 8                       |
| Tests for resistance to cracking   | 4.4                     | 3             | 3.1               | 9                       |
| Hot deformation test   | 4.5                     | 99            | 99.1              | 10                      |
| Thermal stability test for insulations and sheaths                         | 4.6                     | 3             | 3.2               | 9                       |
| Determination of melt flow index (MFI)                                     | 5.1                     | 4             | 4.1               | 10                      |
| Test for resistance to environmental stress cracking:<br>Original granules | 5.2.2                   | 4             | 4.1               | 8                       |
| Test for resistance to environmental stress cracking:<br>Complete cable    | 5.2.3                   | 99            | 99.1              | 11                      |
| Carbon black content   | 5.3.2                   | 4             | 4.1               | 11                      |
| Carbon black dispersion <sup>1)</sup>                                      | 5.3.3                   | -             | -                 | -                       |
| Wrapping test after thermal ageing <sup>2</sup>                            | 5.4                     | 4 4           | $\frac{4.1}{4.2}$ | 9                       |

<sup>1)</sup> Methods given in BS 2782 : Methods 823A or 823B.

<sup>2)</sup> It is intended that a formal proposal to IEC will result in the test in clause 9 of Section 4.1 being withdrawn.

# National annex NC (informative)

| Test  | Part | Section | Clause |
|---|------|---------|--------|
| Water absorption test: electrical                                     | 1    | 1.3     | 9.1    |
| Tests specific to filling compounds:                                  |      |         |        |
| Drop-point  | 5    | 5.1     | 4      |
| Separation of oil   | 5    | 5.1     | 5      |
| Low temperature brittleness   | 5    | 5.1     | 6      |
| Total acid number   | 5    | 5.1     | 7      |
| Absence of corrosive components                                       | 5    | 5.1     | 8      |
| Permittivity at 23 °C   | 5    | 5.1     | 9      |
| D.C. resistivity at 23 °C and 100 °C                                  | 5    | 5.1     | 10     |
| Determination of linear swell after ageing in oil                     | 99   | 99.1    | 12     |
| Alternative ozone resistance test method (low concentration)          | 99   | 99.1    | 13     |
| Method of test for insulation resistance constant $(K \text{ value})$ | 99   | 99.2    | 8      |
| Method of test for power factor and permittivity                      | 99   | 99.2    | 9      |
| Water absorption determined by the capacitance method                 | 99   | 99.2    | 10     |

BS 6469: Section 4.2: 1992 IEC 811-4-2: 1990

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