BRITISH STANDARD

BS 6469 : Section 4.1 : 1992

Insulating and sheathing materials of electric cables

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

Section 4.1 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

(Implementation of HD 505.4.1 S2)

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

Amendments issued since publication

Amd. No.	Date	Text affected

The following BSI references relate to the work on this standard:
Committee reference CIL/20
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Contents

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 BS 6469 : Parts 1 to 5 and Part 99 supersede BS 6469 : 1990 which is withdrawn.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 which were derived from IEC 811 : 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 6234, 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 505.4.1 S2 : 1990, which is identical with IEC 811-4-1 : 1985 plus Amendment No. 1 (1988) plus corrigendum (1986).

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.

The complete IEC standard (IEC 811) will eventually replace IEC Publications 538 and 540. To enable users to compare the relevant clauses in all three publications, a table of cross-references is given in appendix B.

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 505.4.1 S2

March 1990

UDC 621.315.616 : 621.315.2/.3 : 620.1

Descriptors: Electric cable, insulated cable, electrical insulation, sheath, resistance to cracking, wrapping test, measurement, melt flow index, carbon black

English version

Common test methods for insulating and sheathing materials of electric cables

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 content measurement in PE

(IEC 811-4-1 : 1985 + Amendment 1 : 1988)

Méthodes d'essais communes pour les matériaux d'isolation et de gainage des câbles électriques
Quatriéme partie: Méthodes spécifiques pour les mélanges polyéthylène et polypropylène
Section un: Résistance aux craquelures sous contraintes dues à l'environnement — Essai d'enroulement après vieillissement thermique dans l'air — Mesure de l'indice de fluidité à chaud — Mesure dans le PE du taux de noir de carbone et/ou des charges minérales
(CEI 811-4-1 : 1985 + Modification 1 : 1988)

Allgemeine Prüfungen für Isolier- und Mantelwerkstoffe für Kabel und isolierte Leitungen
Teil 4: Besondere Methoden für Polyäthylen und Polypropylen Compounds
Hauptabschnitt 1: Widerstand gegen umgebungsbedingte Spannungsrisse — Wickelprüfung nach thermischer Luftalterung — Messung des Schmelzindex — Bestimmung des Ruß- und/oder Mineralstoffgehalts in PE
(IEC 811-4-1 : 1985 + Änderung 1 : 1988)

This Harmonization Document was approved by CENELEC on 1990-01-01. 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 Komitee für Elektrotechnische Normung

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

Foreword

The English and French versions of this Harmonization Document are provided by the text of the IEC publication and the German version is the official translation of the IEC text.

According to the CENELEC Internal Regulations the CENELEC member National Committees are bound:

to announce the existence of this Harmonization Document at national level by or before 1990-06-01

to publish their new harmonized national standard by or before 1990 12-01

to withdraw all conflicting national standards by or before 1990-12-01.

Harmonized national standards are listed on the HD information sheet, which is available from the CENELEC National Committees or from the CENELEC Central Secretariat.

CONTENTS

COMMON TEST METHODS FOR INSULATING AND SHEATHING MATERIALS OF ELECTRIC CABLES

Part 4: Methods specific to polyethylene and polypropylene compounds

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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

1. Scope

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

This Section One of Part Four gives the methods for measurement of the resistance to environmental stress cracking, for wrapping test after thermal ageing in air, for measurement of melt flow index and for measurement of carbon black and/or mineral filler content, which apply to PE and PP compounds, including cellular compounds and foam skin for insulation.

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		$\leqslant$ 0.925 g/cm^3	23 °C
Medium-density polyethylene	$>$ 0.925	$\leqslant$ 0.940 g/cm^3	
High-density polyethylene		$>$ 0.940 g/cm^3	

Note. – These densities refer to unfilled resins as determined by the method specified in Clause 8 of Publication 811-1-3: Common Test Methods for Insulating and Sheathing Materials of Electric Cables, Part 1: Methods for General Application – Section Three: Method for Determining the Density – Water Absorption Tests – Shrinkage Test.

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 is the mean of the two middle values if the number is even.

8. Resistance to environmental stress cracking

8.1 *General*

These test procedures apply only to the original granules used as sheathing materials.

Procedure A

Applies to materials which will encounter less severe cable system conditions and environments.

Procedure B

Applies to materials which will encounter more severe cable system conditions and environments.

8.2 *Apparatus*

8.2.1 Heatable press for producing moulded test sheets, with platens which are larger than the backing plates.

8.2.2 Two rigid metal backing plates 6 ± 0.5 mm thick and about 200 mm × 230 mm area, each drilled with a hole from one edge so that a temperature sensor can be located within 5 mm of the centre of the plate.

8.2.3 Two separator sheets, about 200 mm × 230 mm, for instance aluminium foil 0.1 mm to 0.2 mm thick.

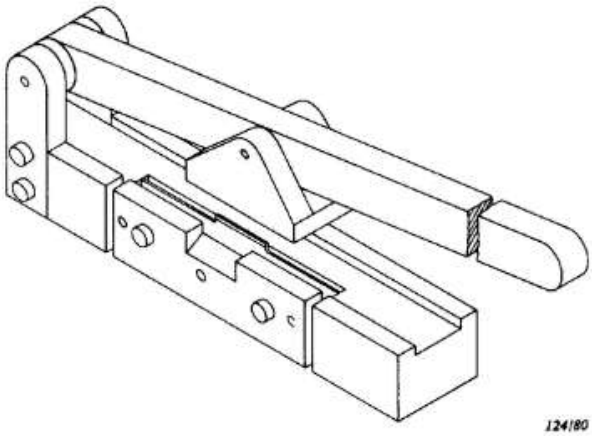
8.2.4 Suitable moulding chases for producing test sheets, 150 mm × 180 mm × 3.3 ± 0.1 mm with internal corners rounded to a radius of 3 mm.

8.2.5 Electrically heated air oven with forced air circulation and programming device which lowers temperature at a rate of 5 ± 0.5 °C/h.

8.2.6 Clean, sharp, undamaged blanking die with blanking press suitable for cutting test pieces 38.0 ± 2.5 mm × 13.0 ± 0.8 mm or other suitable devices.

8.2.7 Dial gauge, with plane gauging faces 4 mm to 8 mm in diameter and a gauging pressure of 5 N/cm² to 8 N/cm².

8.2.8 Notching device as in Figure 1 with blades as in Figure 2.



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The blade is made of "Gem" blades as in Figure 2 (see also Appendix A).

FIGURE 1

Dimensions in millimetres

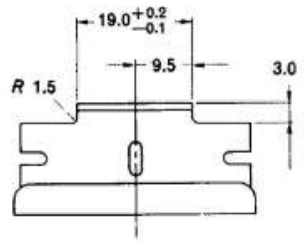

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FIGURE 2

8.2.9 Bending clamp assembly as in Figure 3 with vice or other suitable device ensuring the symmetrical closing of the clamping jaws.

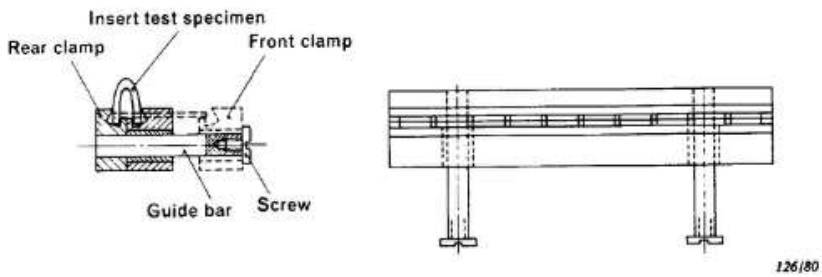

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FIGURE 3

8.2.10 Transfer tool assembly as in Figure 4 for shifting in one operation the bent test piece(s) from the bending clamp to the brass channel.

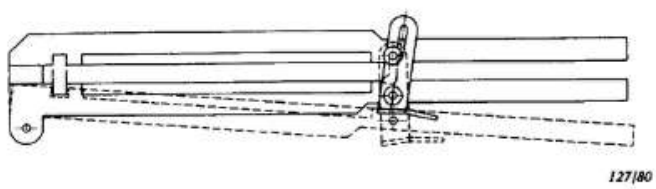

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FIGURE 4

8.2.11 Brass channel specimen holder as in Figure 5 for accommodating ten bent test pieces.

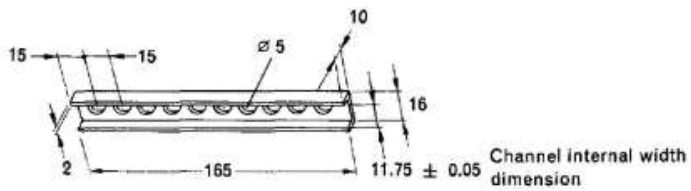


FIGURE 5 128/80

8.2.12 Hard glass test tubes 200 mm × 32 mm for accommodating the brass channel specimen holder with the bent test specimens. The tubes are plugged by suitable aluminium foil wrapped corks (see Figure 6).

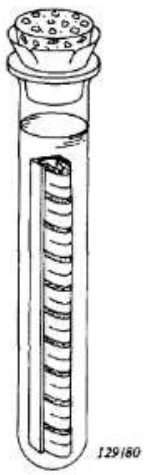


FIG. 6.— Test tube with inserted brass channel specimen holder as in Sub-clause 8.2.11, containing ten test specimens.

8.2.13 Reagents

Procedure A

100% Igepal CO-630 (Antarox CO-630) or any other reagent having the same chemical composition (see Notes 1 and 2 and Appendix A).

Procedure B

10% solution (by volume) in water of Igepal CO-630 (Antarox CO-630) or any other reagent having the same chemical composition (see Notes 1, 2 and 3 and Appendix A).

Notes 1. — The reagent must not be used more than once.

2. — In the case of unexpectedly short failure times, the reagent should be checked for water content as small increases in water content beyond the specified maximum of 1% will cause a significant increase in reagent activity.

3. — Water solution of Igepal CO-630 or similar material should be prepared by paddle-stirring the mixture at 60 °C to 70 °C for at least 1 h. The solution should be used within one week of preparation.

8.2.14 A heated container of sufficient size and depth to accept racks which will hold the filled test tubes (Figure 6). The temperature shall be maintained at 50 ± 0.5 °C by means of suitable equipment and the thermal capacity shall be high enough to ensure that the temperature does not drop below 49 °C even when the test tubes are inserted.

8.3 *Preparation of the test sheets*

8.3.1 For preparing a test, a clean separator foil as in Sub-clause 8.2.3 shall be placed on the backing plate as in Sub-clause 8.2.2, the moulding chase as in Sub-clause 8.2.4. The chase shall be filled with 90 ± 1 g of granules or mill-massed material forming a uniform layer on top of which the second separator foil and then the second backing plate shall be placed. No release agent shall be used.

8.3.2 The mould assembly shall be placed in the moulding press as in Sub-clause 8.2.1, preheated to 170 °C, and the press shall be closed, using a force ≤1 kN.

8.3.3 When the temperature, as indicated by the sensors in the backing plates has reached 165 °C to 170 °C, a full force in the range 50 kN to 200 kN shall be applied to the mould by means of the press, for a period of 2 min during which the sensors should continue to indicate values in the range 165 °C to 170 °C. On completion of the full force phase the heating of the mould assembly shall be stopped either by removing from the press or by fast cooling in the press under full force.

8.4 *Conditioning of the test sheets* *

After removing the backing plates without disturbing the separator foil, the moulded test sheet shall be placed in an oven as in Sub-clause 8.2.5 so as to permit free circulation of air around it, so that the moulding is well supported on thermally conducting horizontal surfaces and so that good contact is maintained between the separator foils and the polyethylene.

The temperature as measured not further than 5 mm above the centre of the horizontal surface of the moulded sheet shall then be controlled as follows:

The oven test temperature shall be maintained for 1 h at 145 ± 2 °C for low density polyethylene, 155 ± 2 °C for medium density polyethylene, and 165 ± 2 °C for high density polyethylene. Cooling shall be at the rate of 5 ± 2 °C/h to 29 ± 1 °C. It is also permissible to cool the moulded test sheets while in the press. The actual cooling rate shall be recorded by a graphical recorder.

Note. – Conditioning of the test sheets should be optional. In case of dispute, a conditioned specimen should be used.

8.5 *Visual examination of the test sheets*

The sheet shall exhibit a smooth surface and shall not contain any bubbles, lumps or sink marks except within 10 mm of the edge.

8.6 *Test procedure*

8.6.1 *Preparation of the test pieces*

Using the blanking die and blanking press as in Sub-clause 8.6.2 or other suitable devices, ten test pieces as in Sub-clause 8.6.2 shall be cut from a test sheet more than 25 mm from the edges of the sheet so that the web between the holes after removal of the test pieces is not damaged during handling.

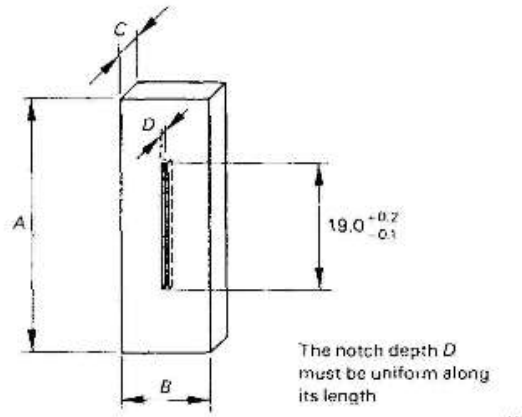
The thickness of the test pieces determined using the dial gauge as in Sub-clause 8.2.7 shall be in accordance with Sub-clause 8.6.2. The test pieces shall be cut with square edges. Bevelled edges may lead to erroneous results.

8.6.2 *Notching and inserting of the test pieces*

Shortly before placing into the reagent, each of the test pieces shall be given a notch (Figure 7) using the notching device as in Sub-clause 8.2.8. The blade shall be neither dull nor damaged and,

* Conditioning of test pieces shall be agreed between the interested parties since it may substantially affect the test results. If such an agreement does not exist, the treatment given in this sub-clause shall be used as a reference treatment.

therefore, shall be replaced as required. Even under favourable conditions it should not be used for more than 100 notches.



Density of PE-sheathing compounds	*A* (mm)	*B* (mm)	*C* (mm)	*D* (mm)
≤ 0.940 g/cm³	38 ± 2.5	13.0 ± 0.8	3.00 to 3.30	0.50 to 0.65
> 0.940 g/cm³	38 ± 2.5	13.0 ± 0.8	1.75 to 2.0	0.30 to 0.40

FIGURE 7

Ten test pieces shall then be placed, with the notch up, in the bending clamp as in Sub-clause 8.2.9. The clamp shall be closed for 30 s to 35 s by means of a vice or a motor-driven arbor press at a constant speed.

The bent test pieces shall be lifted with the transfer tool as in Sub-clause 8.2.10 from the bending clamp and placed in the brass channel as in Sub-clause 8.2.11. If some test pieces are riding too high in the holder they shall be forced down by manual pressure.

The holder shall be inserted in a tube as in Sub-clause 8.2.12, 5 min to 10 min after the test pieces have been bent. The test tube shall be filled with the appropriate reagent as in Sub-clause 8.2.13 until all the test pieces are covered by the liquid, and shall be closed by a cork.

The filled test tube shall be placed immediately in a rack in the heated container as in Sub-clause 8.2.14. Care shall be taken that the test pieces do not touch the test tube during the test. The moment of insertion in the heated container shall be noted.

8.7 *Evaluation of results*

In general, environment stress cracking starts at the notch and runs at right angles to it. The first sign of a crack when examined with normal or corrected vision without magnification constitutes a failure of the test piece.

Procedure A

After 24 h in the heated container no more than five test pieces shall have failed. If six test pieces have failed, the test is to be considered as not passed. The test may be repeated once using ten test pieces from a new test sheet, and no more than five test pieces shall fail.

Procedure B

After 48 h in the heated container no test pieces shall have failed. If one test piece has failed, the test is to be considered as not passed. The test may be repeated once using ten test pieces from a new sheet and no test piece shall fail.

8.8 *Summary of test conditions and requirements for procedures A and B*

Conditions and/or requirements		Procedure A	Procedure B
Preparation of the test sheets:			
– Temperature	°C	165 to 170	
– Force	kN	50 to 200	
– Time	min	2	
Conditioning of test sheets:			
– Temperature range	°C	From (145 ± 2) to (30^{+0}_{-2})	
– Cooling rate	°C/h	5 ± 2	
Test conditions:			
– Reagent* – concentration	%	100	10
– Temperature	°C	50 ± 0.5	
– Duration (minimum)	h	24	48
Requirements:			
– Failure rate	max.	5 test pieces (F 50)	0 test pieces (F 0)

* Igepal CO-630 or any other reagent having the same chemical composition.

9. Wrapping test after thermal ageing in air

9.1 *General*

The scope of this test is to show that the polyethylene compound used for insulation is protected against oxidation.

The test applies to products with diameter ≤10 mm, and/or a wall thickness less than 0.8 mm and for materials with a density ≤0.940 g/cm³.

For materials with a density >0.940 g/cm³ the test is left under consideration.

Note. – A long-term stability test is under consideration.

9.2 *Apparatus*

9.2.1 Smooth metal mandrel and loading elements.

9.2.2 Winding device, preferably with mechanically driven mandrel.

9.2.3 Electrically heated cabinet with natural air flow.

9.3 *Sampling*

The test shall be done on four test pieces for each length of the cable or cores 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.

9.4 *Ageing procedure*

The test pieces prepared in accordance with Sub-clause 9.3 shall be suspended vertically for 14 × 24 h at 100 ± 2 °C, in the middle of the heating chamber in accordance with Sub-clause 9.2.3 so that each test piece is at least 20 mm from any other test piece. Not more than 2% of the cabinet volume shall be occupied by the test pieces. Immediately after the ageing period, the test pieces are taken out of the cabinet and left at room 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.

9.5 *Test procedure*

Test pieces according to Sub-clause 9.3 shall be subjected, after ageing in accordance with Sub-clause 9.4, to a winding test at room 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 ± 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 Sub-clause 9.2.2 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 ± 2 °C in the vertical position, substantially in the middle of the heating chamber in accordance with Sub-clause 9.2.3.

9.6 *Evaluation of results*

After cooling down to room 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.

10. Measurement of the melt flow index

10.1 *General*

The melt flow index (MFI) of polyethylene and polyethylene compounds is the quantity of material extruded in 2.5 min or 10 min at 190 °C through a specified die under the action of a load determined by the method used.

Note. — The melt flow index is not applicable to flame retarding polyethylene.

10.2 *Apparatus*

The apparatus is basically an extrusion plastometer, the general design being as shown in Figure 8, page 30. Polyethylene, which is contained in a vertical meter cylinder, is extruded through a die by a loaded piston under controlled temperature conditions. All surfaces of the apparatus in contact with the material under test shall have a high polish.

The apparatus consists of the following essential parts:

a) Steel cylinder

A steel cylinder fixed in a vertical position and thermally insulated for operation at 190 °C. The cylinder is at least 115 mm long with an internal diameter of between 9.5 mm and 10 mm and complying with the requirements in Item *b)* of Sub-clause 10.2. The base of the cylinder shall be thermally insulated if the area of the exposed metal exceeds 4 cm^2 and it is recommended that the insulating material used be polytetrafluoroethylene (thickness about 3 mm) in order to avoid sticking of the extruded material.

b) Steel hollow piston

A steel hollow piston with a length at least the same as that of the cylinder. The axes of the cylinder and of the piston shall coincide and the effective length of the piston shall be a maximum of 135 mm. There is a head of length 6.35 ± 0.10 mm. The diameter of the head shall be less than the internal diameter of the cylinder at all points along the working length of the cylinder by 0.075 ± 0.015 mm. In addition, for calculating the load (see Item *c)* of Sub-clause 10.2) this diameter should be known within ± 0.025 mm. The lower edge of the head has a radius of 0.4 mm and the upper edge has its sharp edge removed. Above the head, the piston is relieved to about 9 mm diameter. A stud may be added at the top of the piston to support the removable load, but the piston is thermally insulated from this load.

c) Removable load on top of the piston

The combined masses of the load and the piston shall be such that the force P applied is:

P = 21.2 N in the case of method A (see Sub-clause 10.5).

P = 49.1 N in the case of method C (see Sub-clause 10.6).

d) Heater

A heater to maintain the polyethylene in the cylinder at a temperature of 190 ± 0.5 °C. An automatic temperature control is strongly recommended.

e) Temperature measuring device

A temperature measuring device located as closely as possible to the die, but situated within the body of the cylinder, the measuring device being calibrated to permit temperature measurement to an accuracy of ± 0.1 °C.

f) Die

A die of length 8.000 ± 0.025 mm made of hardened steel, the mean internal diameter being between 2.090 mm and 2.100 mm and uniform along its length to within ± 0.005 mm (see Figure 9, page 31). The die shall not project beyond the base of the cylinder.

g) Balance

A balance accurate to ± 0.0005 g.

10.3 *Test samples*

A sample of insulation or sheath of sufficient mass shall be taken from one end of the cable or wire. The sample shall be cut in pieces, the dimension of which shall not exceed 3 mm in any direction.

Note. — If necessary, the insulating material may be taken from different cores.

10.4 *Cleaning and maintenance of the apparatus*

The apparatus shall be cleaned after each test.

On no account should abrasives or materials likely to damage the surfaces of the piston, cylinder or die be used in removing superficial polyethylene or in manipulating any part of the apparatus.

Suitable solvents for cleaning the apparatus are xylene, tetrahydronaphthalene or odourless kerosene. The piston shall be cleaned while still hot with a cloth dipped in the solvent, and the cylinder, also while still hot, with a swab dipped in the solvent. The die shall be cleaned with a closely-fitting brass reamer or wooden peg, and then immersed in boiling solvent.

It is recommended that, at fairly frequent intervals, for example once a week for apparatus in constant use, the insulating plate and the die-retaining plate, if fitted, (see Figure 8, page 30) be removed and the cylinder cleaned thoroughly.

10.5 *Method A*

10.5.1 *General*

Method A is suitable for determining the melt flow index (MFI) of a sample of polyethylene whose MFI is unknown.

10.5.2 *Test procedure*

The apparatus shall be cleaned (see Sub-clause 10.4). Before beginning a series of tests, the temperature of the cylinder and piston should be at 190 ± 0.5 °C for 15 min and this temperature maintained during the extrusion of the polyethylene.

It is recommended that the temperature measuring device (see Item *e)* of Sub-clause 10.2) be a mercury-in-glass thermometer located permanently within the mass of the cylinder (see note below). A low melting-point alloy, such as Wood's metal, improves the thermal contact and its use is recommended.

Note. — If any other temperature measuring device is used, it should be calibrated at 190 ± 0.5 °C before the beginning of each series of tests in comparison with a mercury-in-glass thermometer, conforming to Item *e)* of Sub-clause 10.2 above, placed within the cylinder and immersed in polyethylene to its appropriate depth of immersion.

The cylinder shall then be charged with a portion of the sample (see Table 1) and the unloaded piston reinserted into the top of the cylinder.

Six minutes after introducing the sample, during which time the temperature of the cylinder should have returned to 190 ± 0.5 °C, the load is placed on the piston to extrude the polyethylene through the die. The rate of extrusion is measured by cutting the extruded material at regular intervals of time at the die with a suitable sharp-edged instrument to give short lengths of extruded material which will be referred to as "cut-offs". The time intervals at which each cut-off is taken are given in Table 1.

Several cut-offs shall be taken within 20 min of the introduction of the sample into the cylinder. The first cut-off and any containing air bubbles shall be ignored. The remaining successive cut-offs, of which there shall be at least three, shall be weighed individually to the nearest milligram and the average mass determined. If the difference between the maximum and the minimum values of the individual weighings exceeds 10% of the average, the test results shall be discarded and the test repeated on a fresh portion of the sample.

10.5.3 *Expression of results*

The MFI should be reported to two significant figures (see Note 1) and expressed in symbols as MFI.190.20.A (see Note 2):

$$\text{MFI.190.20.A} = \frac{600 \times m}{t}$$

where:

MFI is expressed in grams per 10 min

m is the average mass of cut-offs, expressed in grams

t is the time interval of cut-offs, expressed in seconds

Notes 1. — The MFI of polyethylene may be affected by previous thermal and mechanical treatments, and in particular oxidation will tend to reduce the MFI. Oxidation occurring during the test will usually cause a systematic reduction in the masses of successive cut-offs. This phenomenon is not exhibited by polyethylene compounds containing an anti-oxidant.

2. — MFI — melt flow index.
190 = temperature of tests, expressed in degrees Celsius.
20 (or 50) — approximate load, expressed in newtons applied to the melt.

10.6 *Method C*

10.6.1 *General*

Method C is suitable for determining the MFI of a sample of polyethylene whose MFI, measured in accordance with method A, is below 1.

10.6.2 *Test procedure*

The test procedure is the same as for method A.

The time intervals used in obtaining the cut-offs and the mass of the charge put into the cylinder are given in Table I.

10.6.3 *Expression of results*

The MFI should be reported to two significant figures (see Note 1 above) and expressed in symbols as MFI.190.50.C (see Note 2 above):

$$\text{MFI.190.50.C} = \frac{150 \times m}{t}$$

Note. — The use of shorter cut-off time (150 s) with a heavier load (50 N) gives results quoted on scale C which agree approximately with results that would have been obtained had method A and scale A been used. There is, however, no direct correlation between scales A and C.

TABLE 1

Time intervals (as a function of melt flow index) used in obtaining the cut-offs and mass of the charge put into the cylinder for methods A and C

Melt flow index (IF)	Mass of the charge put into the cylinder (g)	Time intervals (s)
0.1 to 0.5	4 to 5	240
0.5 to 1	4 to 5	120

11. Carbon black and/or mineral filler content measurement in PE

11.1 *Sampling*

A sample of the insulation or sheath of sufficient weight shall be taken from one end of the cable. The sample shall be cut in pieces, the dimensions of which shall not exceed 5 mm in any direction.

11.2 *Test procedure*

A combustion boat about 75 mm long shall be heated until it is red hot, allowed to cool in the desiccator for at least 30 min and weighed to the nearest 0.0001 g. A sample of polyethylene weighing 1.0 ± 0.1 g shall be placed in the boat and the whole weighed to the nearest 0.0001 g. The weight of the boat shall be subtracted to give the weight of the polyethylene to the nearest 0.0001 g (quantity A).

The boat and the sample shall then be placed in the middle of a hard glass, silica or porcelain combustion tube, bore approximately 30 mm, length 400 ± 50 mm. A stopper carrying a thermometer for temperature measurements from 300 °C to 550 °C and a tube for the admission of nitrogen shall then be inserted into one end of the combustion tube so that the end of the thermometer touches the boat. Nitrogen with an oxygen content of less than 0.5% shall be passed through the combustion tube at 1.7 ± 0.3 l/min and this rate of flow shall be maintained during the subsequent heating.

Note. — In case of doubt, the oxygen content of the nitrogen shall be limited to 0.01%.

The combustion tube shall be placed in a furnace and its outlet connected to two cold traps in series, both containing trichlorethylene, the first being cooled with solid carbon dioxide. The outlet tube from the second trap shall lead to a fume hood or the outside atmosphere. Alternatively, it is permissible for the outlet from the combustion tube to lead directly to the outside atmosphere.

The furnace shall then be heated so that the temperature is between 300 °C and 350 °C after about 10 min, about 450 °C after 10 min further, and 500 ± 5 °C after a third period of 10 min. This temperature shall then be maintained for 10 min at the end of which the outlet tube shall be disconnected from the cold traps, if these are used, and the tube containing the boat withdrawn from the furnace and allowed to cool for 5 min, the flow of nitrogen being maintained at the same rate as before.

The boat shall then be removed from the combustion tube through the nitrogen inlet end, allowed to cool in the desiccator for 20 min to 30 min and reweighed, the weight of the residue being determined to the nearest 0.0001 g (quantity B of residue).

Subsequently, the boat shall be introduced again into the combustion tube; instead of nitrogen, air or oxygen shall be blown through the tube at an adequate flow rate at a temperature of 500 ± 20 °C, and the remaining carbon black shall be burnt. After it has cooled in the test assembly, the boat shall be removed and weighed again, the mass of the residue being determined to the nearest 0.0001 g (quantity C of residue).

11.3 *Expression of results*

$$\text{Carbon black content} = \frac{B - C}{A} \cdot 100\%$$

$$\text{Mineral filler content} = \frac{C}{A} \cdot 100\%$$

$$\text{Filler content} = \frac{B}{A} \cdot 100\%$$

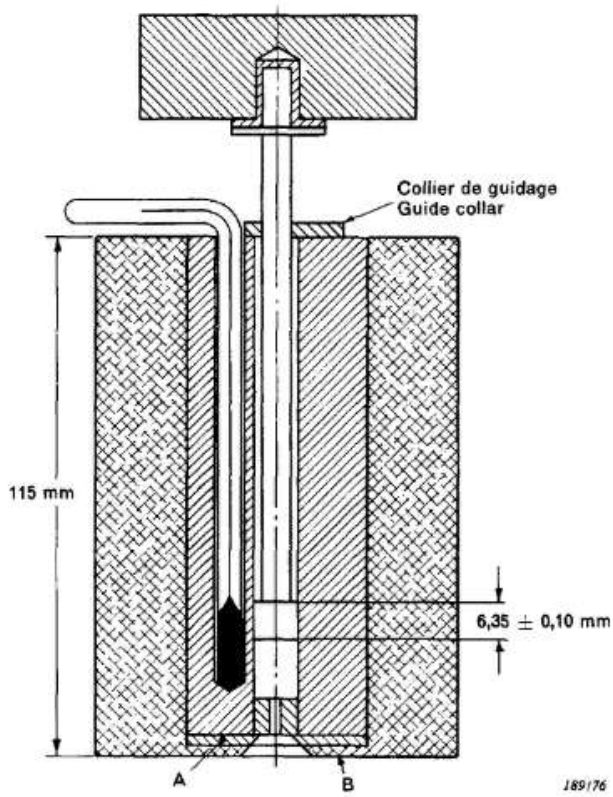


FIG. 8. — Appareil pour déterminer l'indice de fluidité à chaud (montrant le cylindre à grand diamètre extérieur, la plaque de fixation A de la filière et la plaque isolante B).
Apparatus for determining melt flow index (showing large external diameter cylinder, die-retaining plate A and insulating plate B).

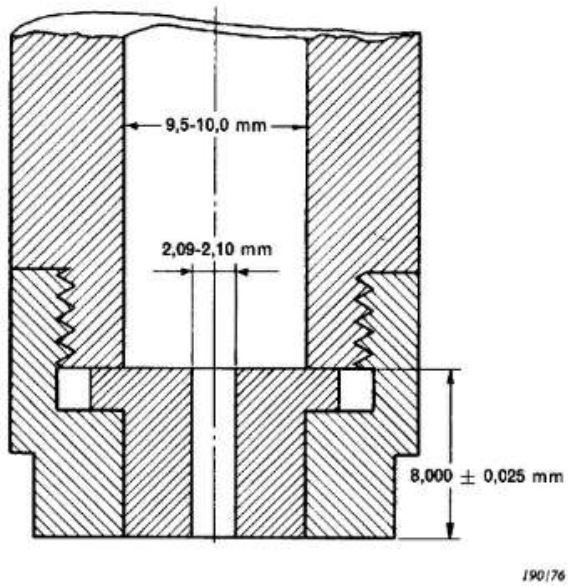


FIG. 9. — Filière (montrant le cylindre de petit diamètre extérieur et un exemple du montage de la filière).
Die (showing small external diameter cylinder with an example method of retaining the die).

APPENDIX A

TOOLS AND REAGENTS

Tools

The tools indicated in Sub-clauses 8.2.8, 8.2.9 and 8.2.10 can be obtained from:

Messrs. Custon Scientific Instruments Inc.
541 Deven Street
Arlington, N.J.
U.S.A.

Detail drawings of the tools are obtainable from:

American Society for Testing and Materials (ASTM)
1916 Race Street
Philadelphia 19103, Pa.
U.S.A.

Reagents

The reagent 100% IGEPAL CO-630 of density 1.06 at 25 °C can be obtained from:

GAF Corp., Dyestuff and Chemical Div.
140 West 51 Street
New York, N.Y. 10020
U.S.A.

and must contain less than 1% water. Because it is hygroscopic, it should be stored in closed metal or glass containers.

APPENDIX B

CORRESPONDING CLAUSES AND SUB-CLAUSES IN IEC PUBLICATIONS 538 AND 540 AND IEC PUBLICATION 811

B1. Corresponding clauses and sub-clauses in IEC Publications 538 and 811

Heading of clause in Publication 538*	538	811		
	Clause or sub-clause	Part	Section	Clause or sub-clause
General	1	All	All	1 to 7
Mechanical properties of insulation	2	1	1	9.1
Mechanical properties of sheath	3	1	1	9.2
Melt flow index (MFI)	4	4	1	10
Density	5	1	3	8
Ageing test for insulation and sheath	6.1	1	2	8
Shrinkage test for insulation	6.2	1	3	10
Bending test at low temperature				
insulation	6.3.1	1	4	8.1
sheath	6.3.2	1	4	8.2
Carbon black and/or mineral filler content	7	4	1	11
Measurement of thicknesses and diameters	Appendix A	1	1	8
Melt flow index	Appendix B	4	1	10

Heading of clause in Publication 538A**	538A	811		
	Clause	Part	Section	Clause
Wrapping test after thermal ageing in air	1	4	1	9
Resistance to environmental stress cracking	2	4	1	8

* Publication 538: Electric Cables, Wires and Cords: Methods of Test for Polyethylene Insulation and Sheath.

** Publication 538A: First Supplement to Publication 538 (1976): Additional Methods for Test for Polyethylene Insulation and Sheath of Electric Cables, Wires and Cords Used in Telecommunication Equipment and in Devices Employing Similar Techniques.

B2. Corresponding clauses in I E C Publications 540, 811 and 885 *

Heading of clause in Publication 540 *	540	811			885
	Clause	Part	Section	Clause	Part
Partial discharge tests	3	–	–	–	2
Measurement of thicknesses and diameters **	4	1	1	8	–
Tests for determining the mechanical properties of insulating and sheathing compounds	5	1	1	9	–
Thermal ageing methods	6	1	2	8	–
Loss of mass test for PVC insulations and sheaths	7	3	2	8	–
Pressure test at high temperature for PVC insulations and sheaths	8	3	1	8	–
Tests at low temperature for PVC insulations and sheaths	9	1	4	8	–
Tests for resistance of PVC insulations and sheaths to cracking	10	3	1	9	–
Method for determining the density of elastomeric and thermoplastic compounds	11	1	3	8	–
Measurement of the melt flow index of thermoplastic polyethylene	12	4	1	10	–
Ozone resistance test	13	2	1	8	–
Hot set test	14	2	1	9	–
Mineral oil immersion test for elastomeric sheaths	15	2	1	10	–
Electrical tests for cables, cords and wires for voltages up to and including 450/750 V	16	–	–	–	1
Thermal stability of PVC insulations and sheaths	17	3	2	9	–
Carbon black and/or mineral filler content in PE	18	4	1	11	–
Water absorption tests	19	1	3	9	–
Shrinkage test	20	1	3	10	–

* Publication 540: Test Methods for Insulations and Sheaths of Electric Cables and Cords (Elastomeric and Thermoplastic Compounds).
Publication 885: Electrical Test Methods for Electric Cables.

** Technically not identical.

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

National annex NB (informative)

Table NB.1. Corresponding clauses or sub-clauses in BS 6469 : 1990 and BS 6469 : 1992

Clause in BS 6469 : 1990	BS 6469 : 1990	BS 6469 : 1992		
	Clause or sub-clause	Part	Section	Clause or sub-clause
General	1.1 to 1.3	1 to 4 and 99 5	All 5.1	1 to 7 1 to 3
Measurement of thicknesses 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: Original granules	5.2.2	4	4.1	8
Test for resistance to environmental stress cracking: Complete cable	5.2.3	99	99.1	11
Carbon black content	5.3.2	4	4.1	11
Carbon black dispersion[1]	5.3.3	-	-	-
Wrapping test after thermal ageing[2]	5.4	4 4	4.1 4.2	9 10

[1] Methods given in BS 2782 : Methods 823A or 823B.

[2] It is intended that a formal proposal to IEC will result in the test in clause 9 of Section 4.1 being withdrawn.

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National annex NC (informative)

Table NC.1. Tests included in BS 6469 : 1992 not previously covered

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 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 4.1 : 1992

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