BRITISH STANDARD

BS EN
60811-4-1:2004

Insulating and sheathing materials of electric and optical cables — Common test methods —

Part 4-1: Methods specific to polyethylene and polypropylene compounds — Resistance to environmental stress cracking — Measurement of the melt flow index — Carbon black and/or mineral filler content measurement in polyethylene by direct combustion — Measurement of carbon black content by termogravimetric analysis (TGA) — Assessment of carbon black dispersion in polyethylene using a microscope

The European Standard EN 60811-4-1:2004 has the status of a British Standard

ICS 29.035.20; 29.060.20

BSI
British Standards

National foreword

This British Standard is the official English language version of EN 60811-4-1:2004. It is identical with IEC 60811-4-1:2004. It supersedes BS EN 60811-4-1:1995 which is withdrawn.

The UK participation in its preparation was entrusted by Technical Committee GEL/20, Electric cables, to Subcommittee GEL/20/17, Low voltage cables, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

Cross-references

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EUROPEAN STANDARD **EN 60811-4-1**

NORME EUROPÉENNE

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ICS 29.035.20; 29.060.20 Supersedes EN 60811-4-1:1995

English version

Insulating and sheathing materials of electric and optical cables – Common test methods
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(IEC 60811-4-1:2004)

Matériaux d'isolation et de gainage des câbles électriques et optiques - Méthodes d'essais communes
Partie 4-1: Méthodes spécifiques pour les mélanges polyéthylène et polypropylène - Résistance aux craquelures sous contraintes dues à l'environnement - Mesure de l'indice de fluidité à chaud - Mesure dans le polyéthylène du taux de noir de carbone et/ou des charges minérales par méthode de combustion directe - Mesure du taux de noir de carbone par analyse thermogravimétrique - Evaluation de la dispersion du noir de carbone dans le polyéthylène au moyen d'un microscope
(CEI 60811-4-1:2004)

Isolier- und Mantelwerkstoffe für Kabel und isolierte Leitungen - Allgemeine Prüfverfahren
Teil 4-1: Besondere Verfahren für Polyethylen- und Polypropylen-Verbindungen - Spannungsrissbeständigkeit - Messung des Schmelzindexes - Bestimmung des Ruß- und/oder Füllstoffgehaltes in Polyethylen durch direkte Verbrennung - Bestimmung des Rußgehaltes durch thermogravimetrische Analyse (TGA) - Bewertung der Rußverteilung in Polyethylen unter Verwendung eines Mikroskops
(IEC 60811-4-1:2004)

This European Standard was approved by CENELEC on 2004-07-01. CENELEC members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CENELEC member into its own language and notified to the Central Secretariat has the same status as the official versions.

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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

Ref. No. EN 60811-4-1:2004 E

Foreword

The text of document 20/687/FDIS, future edition 2 of IEC 60811-4-1, prepared by IEC TC 20, Electric cables, was submitted to the IEC-CENELEC parallel vote and was approved by CENELEC as EN 60811-4-1 on 2004-07-01.

This European Standard supersedes EN 60811-4-1:1995.

The principal changes with respect to EN 60811-4-1:1995 are:

a) the wrapping test after thermal ageing in air is deleted from this part of EN 60811. It is now given only in EN 60811-4-2;

b) a thermogravimetic method is added for determination of carbon black content;

c) a method is introduced for assessment of carbon black dispersion.

The following dates were fixed:

– latest date by which the EN has to be implemented at national level by publication of an identical national standard or by endorsement	(dop)	2005-04-01
– latest date by which the national standards conflicting with the EN have to be withdrawn	(dow)	2007-07-01

Annex ZA has been added by CENELEC.

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Endorsement notice

The text of the International Standard IEC 60811-4-1:2004 was approved by CENELEC as a European Standard without any modification.

In the official version, for Bibliography, the following notes have to be added for the standards indicated:

IEC 60811-4-2	NOTE	Harmonized as EN 60811-4-2:2004 (not modified).
ISO 1133	NOTE	Harmonized as EN ISO 1133:1997 (not modified).

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INSULATING AND SHEATHING MATERIALS OF ELECTRIC AND OPTICAL CABLES – COMMON TEST METHODS –

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1 General

1.1 Scope

This part of IEC 60811 specifies the test methods to be used for testing polymeric insulating and sheathing materials of electric and optical fibre cables for power distribution and telecommunications, including cables used on ships and in offshore applications. These test methods apply specifically to PE and PP compounds, including cellular compounds and foam skin for insulation.

1.2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60811-1-3:1993, *Insulating and sheathing materials of electric cables – Common test methods – Part 1: General application – Section 3: Methods for determining the density – Water absorption tests – Shrinkage test*

ISO 18553:2002, *Method for the assessment of the degree of pigment or carbon black dispersion in polyolefin pipes, fittings and compounds*

2 Terms and definitions

For the purposes of this document, a distinction is made between low-density, medium-density and high-density PE as shown below:

Type of polyethylene	Density at 23 °C [a] g/cm^3
Low-density polyethylene	≤0,925
Medium-density polyethylene	>0,925 ≤0,940
High-density polyethylene	>0,940
[a] These densities refer to unfilled resins as determined by the method specified in Clause 8 of IEC 60811-1-3.	

3 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.

4 Applicability

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

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.

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 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

The apparatus shall comprise the following elements:

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 in 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.

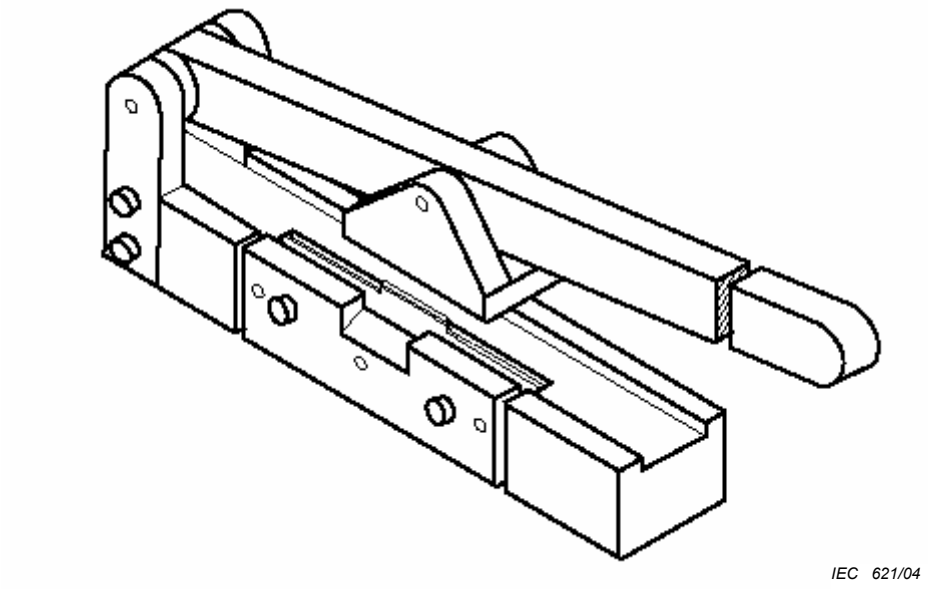
8.2.4 Suitable moulding chases for producing test sheets, 150 mm × 180 mm × (3,3 ± 0,1) mm with internal corners rounded to a radius of 3 mm.

8.2.5 Electrically heated air oven with forced air circulation and programming device which lowers temperature at a rate of (5 ± 0,5) K/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/cm^2 to 8 N/cm^2.

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



NOTE The blade is made of “Gem” blades as in Figure 2 – see also Annex A.

Figure 1 – Notching device

Dimensions in millimetres

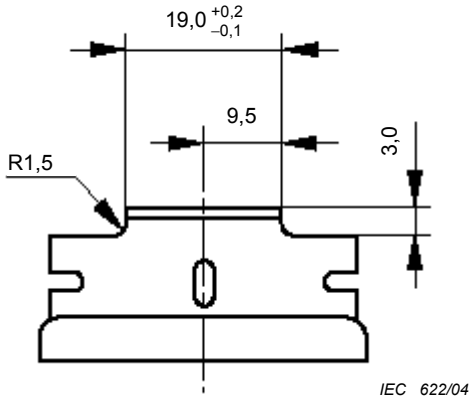
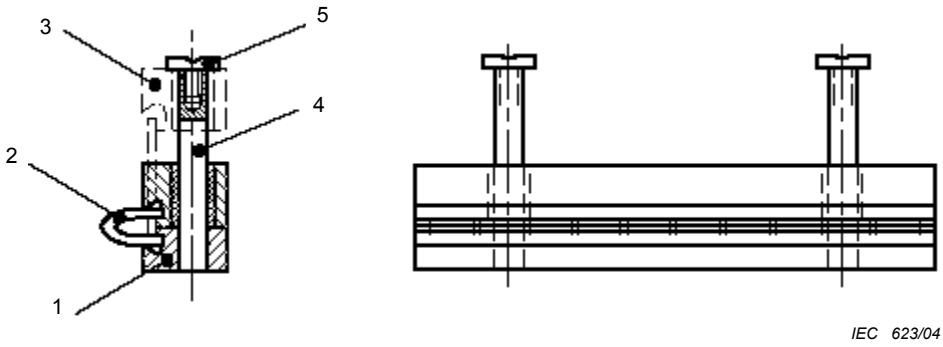


Figure 2 – Blade

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



1	Rear clamp	4	Guide bar
2	Insert test specimen	5	Screw
3	Front clamp		

Figure 3 – Bend clamp assembly

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.

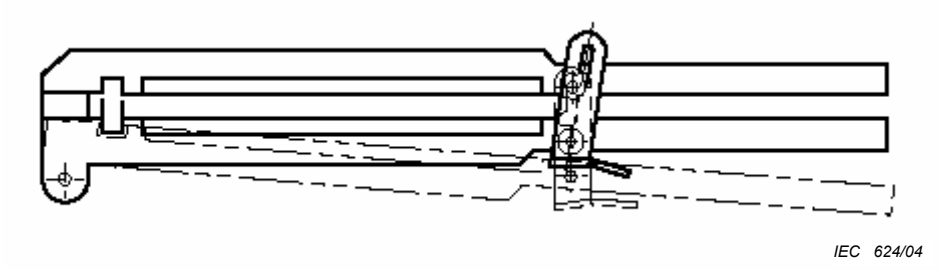
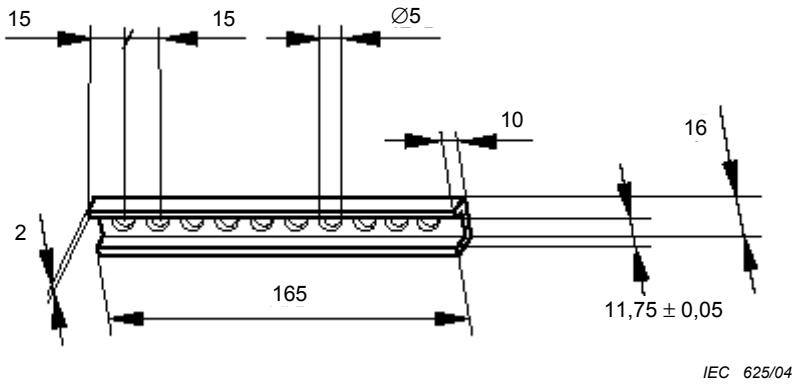


Figure 4 – Transfer tool assembly

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

Dimensions in millimetres



The dimension 11,75 mm ± 0,05 mm is the channel internal width.

Figure 5 – Brass channel specimen holder

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).

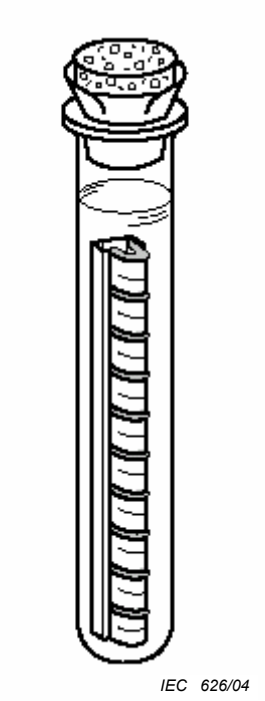


Figure 6 – Test tube with inserted brass channel specimen holder as in 8.2.11, containing ten test specimens

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 below and Annex 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 below and Annex A).

NOTE 1 The reagent should not be used more than once.

NOTE 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.

NOTE 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 (see 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 8.2.3 shall be placed on the backing plate as in 8.2.2, the moulding chase as in 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 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 plate, 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 shall 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

Conditioning of test sheets 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 subclause shall be used as a reference treatment.

After removing the backing plates without disturbing the separator foil, the moulded test sheet shall be placed in an oven, as in 8.2.5, so as to permit free circulation of air around it. The moulding shall be well supported on thermally conducting horizontal surfaces and a good contact 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 °C ± 2 °C for low-density polyethylene, 155 °C ± 2 °C for medium-density polyethylene, and 165 °C ± 2 °C for high-density polyethylene. Cooling shall be at the rate of (5 ± 2) K /h until it reaches 29 °C ± 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

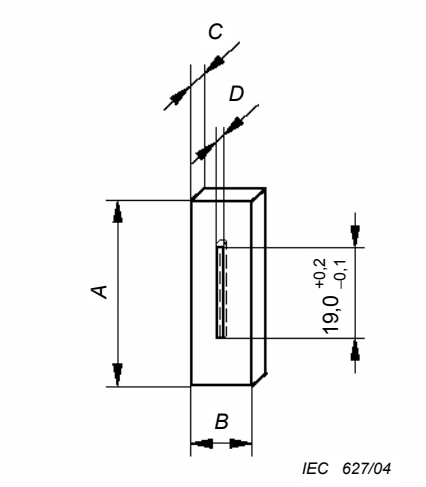
Using the blanking die and blanking press as in 8.2.6 or other suitable devices, ten test pieces as in 8.6.2 shall be cut from a test sheet more than 25 mm from the edges of the sheet so that the web between the holes after removal of the test pieces is not damaged during handling.

The thickness of the test pieces determined using the dial gauge as in 8.2.7 shall be in accordance with 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 (see Figure 7) using the notching device as in 8.2.8. The blade shall be neither dull nor damaged and, therefore, shall be replaced as required. Even under favourable conditions, it should not be used for more than 100 notches.

Dimensions in millimetres



Density of PE-sheathing compounds [a]	*A* mm	*B* mm	*C* mm	*D* [b] mm
≤0,940 g/cm^3	38,0 ± 2,5	13,0 ± 0,8	3,00 to 3,30	0,50 to 0,65
>0,940 g/cm^3	38,0 ± 2,5	13,0 ± 0,8	1,75 to 2,0	0,30 to 0,40

[a] The density is for the unfilled resin, according to Clause 4.

[b] The depth *D* shall be uniform along its length.

Figure 7 – Notched test pieces

Ten test pieces shall then be placed, with the notch up, in the bending clamp as in 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 8.2.10 from the bending clamp and placed in the brass channel as in 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 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 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 8.2.14. Care shall be taken so 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		Method A	Method 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	See note a	
– Cooling rate	K/h	5 ± 2	
Test conditions:			
– Reagent [b] – concentration	%	100	10
– Temperature	°C	50,0 ± 0,5	
– Duration (minimum)	h	24	48
Requirements:			
– Failure rate	Max.	5 test pieces (F 50)	0 test pieces (F 0)

[a] Starting temperature varies according to polymer type:
145 °C ± 2 °C for low-density polyethylene;
155 °C ± 2 °C for medium-density polyethylene;
165 °C ± 2 °C for high-density polyethylene.
Final temperature 29 °C ± 1 °C.

[b] Igepal CO-630 or any other reagent having the same chemical composition.

9 Wrapping test after thermal ageing in air

NOTE Wrapping after thermal ageing in air is now covered only by Clause 10 in IEC 60811-4-2.

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 1,5 min or 10 min at 190 °C through a specified die under the action of a load determined by the method used.

NOTE 1 The same method is also specified in ISO 1133.

NOTE 2 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. Polyethylene, which is contained in a vertical 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 shall be 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) below. 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) this diameter should be known within ±0,025 mm. The lower edge of the head shall have 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 10.5);

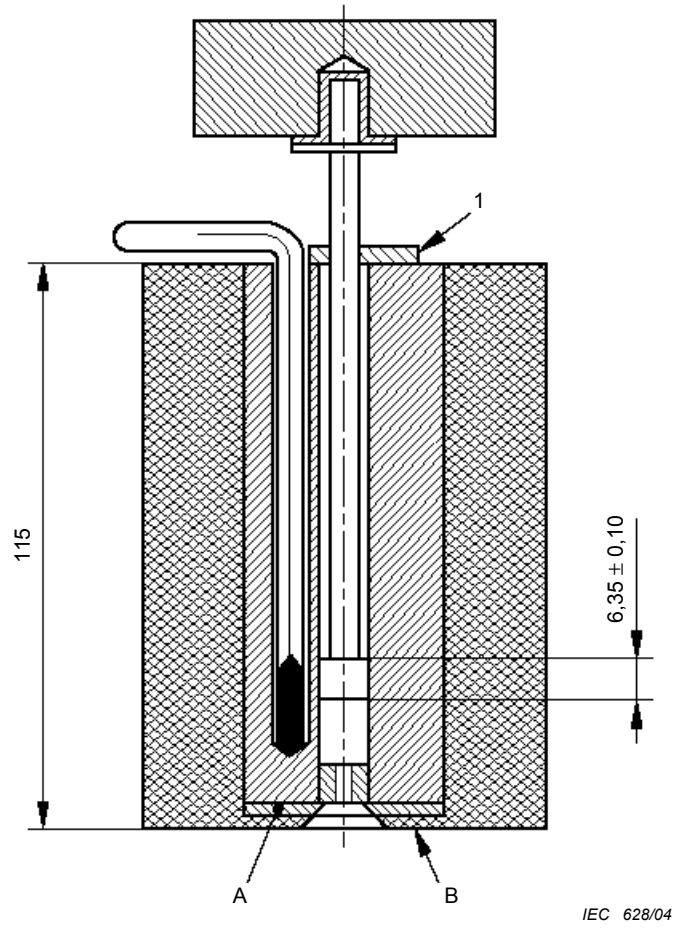
P = 49,1 N in the case of method C (see 10.6);

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 shall be calibrated to permit temperature measurement to an accuracy of ±0,1 °C.



Key

1 Guide collar

Figure 8 – Apparatus for determining melt flow index (showing large external diameter cylinder, die-retaining plate A and insulating plate B)

Dimensions in millimetres

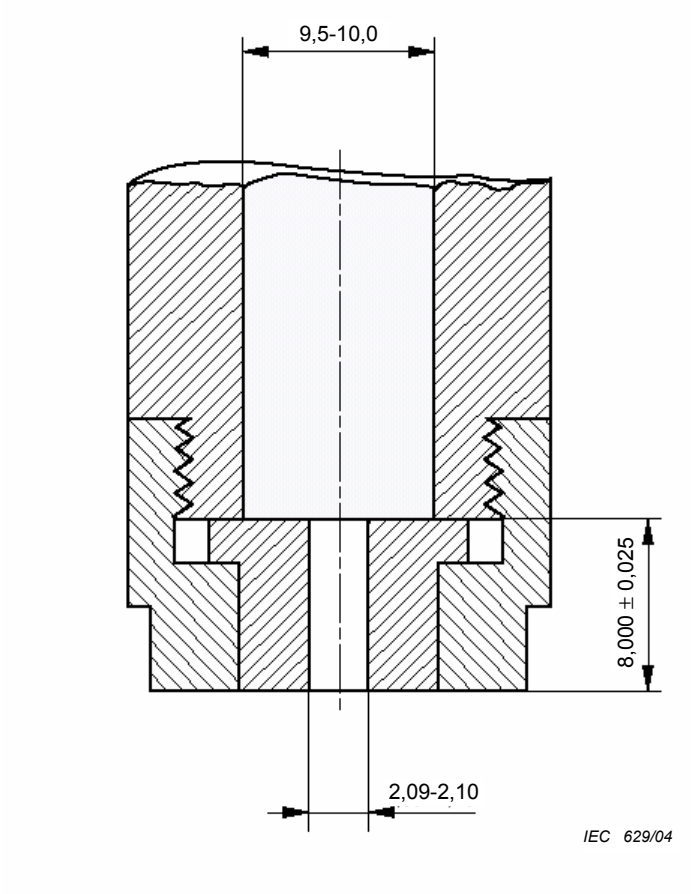


Figure 9 – Die (showing small external diameter cylinder with an example method of retaining the die)

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). 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), 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 10.4). Before beginning a series of tests, the temperature of the cylinder and piston shall 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 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 10.2, 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.

Four minutes after introducing the sample, during which time the temperature of the cylinder shall 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 shall be 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 melt flow index (MFI) shall be reported to two significant figures (see Note 1) and expressed in g/600 s 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.

NOTE 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.

NOTE 2

190 = temperature of tests, expressed in degrees Celsius.

20 (or 50 for Method C) = 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 1.

10.6.3 Expression of results

The MFI shall be reported to two significant figures (see Note 1 of 10.5.3) and expressed in g/150 s as MFI.190.50.C (see Note 2 of 10.5.3):

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

NOTE The use of shorter reference 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 MFI	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
1 to 3,5	4 to 5	60

11 Carbon black and/or mineral filler content measurement in polyethylene – Direct combustion method

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 650 °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.

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 another 10 min; and (600 ± 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 is 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 for a temperature of (600 ± 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 is determined to the nearest 0,0001 g (quantity C of residue).

11.3 Expression of results

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

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

$$\text{Total filler content} = \frac{B}{A} \cdot 100\,\%$$

12 Thermogravimetric analysis of the carbon black content in polyolefine compounds

NOTE This method may be used as an alternative to that in Clause 11 when measuring carbon black content of polyethylene. In the event of dispute, the direct combustion method in Clause 11 should be used as the reference method.

12.1 Principle

Heat a weighed test portion in a thermogravimetric analyser starting at 100 °C with 20 K/min up to 950 °C.

NOTE 1 A starting temperature of 100 °C is practical, as the subsequent measurements can be carried out earlier because of the shorter cooling time.

At first, purge the test portion with dry nitrogen which shall be free of oxygen. When the temperature of 850 °C is reached, switch from dry nitrogen to “synthetic air”. With the switch to air the combustion of the carbon black that is present will follow.

NOTE 2 Weight loss during the purging stage with nitrogen, up to approximately 800 °C, is due to degradation of the polymer and loss of other minor ingredients.

12.2 Reagents

– Dry nitrogen with an oxygen content of less than 10 mg/kg.
– Dry “synthetic air” (a mixture of 80 % nitrogen and 20 % oxygen).

12.3 Apparatus

– Thermogravimetric analyser.
– Gas selector.
– Plotter.
– Analytical balance.

12.4 Procedure

12.4.1 Parameters of the apparatus

a) Starting temperature 100 °C.
b) Heating rate 20 K/min.
c) End temperature 950 °C.
d) Weighed test portion 5 mg to 10 mg.
e) Purging gas up to 850 °C dry nitrogen.
f) Purging gas from 850 °C to 950 °C dry “synthetic air”.

12.4.2 Operation

Operate the apparatus according to the manufacturer’s instructions and the parameters given in 12.4.1. Cover the bottom of the crucible with the test portion, which should consist of a sheet which is as thin as possible. Before the start of the heating period, ensure that an oxygen-free atmosphere is obtained by purging with nitrogen for at least 5 min.

12.4.3 Evaluation

The share of carbon black in the compound is determined for each single test portion from the weight change during burning in dry “synthetic air” from 850 °C to 950 °C. The ignition residue at 950 °C is, at the same time, the ash content.

13 Test for the assessment of carbon black dispersion in polyethylene

13.1 General

The assessment shall be carried out in accordance with ISO 18553. The method is suitable for use with a polyethylene compound or an extrusion (for example a sheath).

NOTE The method applies only to polyethylene with less than 3 % carbon black content.

ISO 18553 gives two procedures for the preparation of test specimens. Either may be used, but the following recommendations apply:

– the compression procedure is primarily intended for use with polyethylene compounds, but may be used for extrusions;
– the microtome procedure is intended for use with polyethylene extrusions.

13.2 Procedure

In accordance with ISO 18553, prepare the specified number of test specimens.

Using the microscopic examination technique given in ISO 18553, examine the test specimens for

a) degree of dispersion.
b) rating of appearance.

13.3 Expression of results

Express the results of the examination in the manner given in ISO 18553.

13.4 Requirements

Unless otherwise stated in the relevant cable standard, the limits recommended in Annex D of ISO 18553 shall be taken to indicate an acceptable degree of dispersion.

NOTE Annex D of ISO 18553 says:

"The following limits are recommended:

Grading: mean (see 5.1) ≤ 3.

Appearance rating: not worse than micrograph B in Annex B (i.e. only dispersion ratings comparable to photomicrographs A1, A2, A3 and B are acceptable)."

Annex A
(informative)

Tools and reagents

Tools

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

MM. 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.

Bibliography

[1] IEC 60811-4-2:2004, *Insulating and sheathing materials of electric and optical cables – Common test methods – Part 4-2: Methods specific to polyethylene and polypropylene compounds – Tensile strength and elongation at break after conditioning at elevated temperature – Wrapping test after conditioning at elevated temperature – Wrapping test after thermal ageing in air – Measurement of mass increase – Long-term stability test – Test method for copper-catalyzed oxidative degradation*

[2] ISO 1133, *Plastics – Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics*

Annex ZA
(normative)

Normative references to international publications with their corresponding European publications

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

NOTE Where an international publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

Publication	Year	Title	EN/HD	Year
IEC 60811-1-3	1993	Insulating and sheathing materials of electric and optical cables - Common test methods Part 1-3: General application - Methods for determining the density - Water absorption tests - Shrinkage test	EN 60811-1-3	1995
ISO 18553	2002	Method for the assesment of the degree of pigment or carbon black dispersion in polyolefin pipes, fittings and compounds	-	-

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