

BS EN 60811-605:2012



BSI Standards Publication

Electric and optical fibre cables — Test methods for non-metallic materials

Part 605: Physical tests — Measurement of carbon black and/or mineral filler in polyethylene compounds

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This British Standard is the UK implementation of EN 60811-605:2012. It is identical to IEC 60811-605:2012.

In the UK, the relationship between the supersessions of BS EN 60811 series can be summarized as follows.

BS EN 60811-100 together with	Supersedes -
-201, -202, -203, -501	BS EN 60811-1-1:1995
-301, -302, -411, -601, -602, -603, -604	BS EN 60811-5-1:2000
-401, -412	BS EN 60811-1-2:1995
-402, -502, -503, -606	BS EN 60811-1-3:1995
-403, -404, -507	BS EN 60811-2-1:1998
-405, -409	BS EN 60811-3-2:1995
-406, -511, -605, -607	BS EN 60811-4-1:2004
-407, -408, -410, -510, -512, -513	BS EN 60811-4-2:2004
-504, -505, -506	BS EN 60811-1-4:1995
-508, -509	BS EN 60811-3-1:1995

Superseded standards are withdrawn

The UK participation in its preparation was entrusted by Technical Committee GEL/20, Electric cables, to Subcommittee GEL/20/17, Electric Cables - Low voltage.

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

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Amendments issued since publication

Amd. No.	Date	Text affected
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English version

**Electric and optical fibre cables -
Test methods for non-metallic materials -
Part 605 : Physical tests -
Measurement of carbon black and/or mineral filler in polyethylene
compounds
(IEC 60811-605:2012)**

Câbles électriques et à fibres optiques -
Méthodes d'essai pour les matériaux non-
métalliques -
Partie 605: Essais physiques -
Mesure du taux de noir de carbone et/ou
des charges minérales dans les mélanges
en polyéthylène
(CEI 60811-605:2012)

Kabel, isolierte Leitungen und
Glasfaserkabel -
Prüfverfahren für nichtmetallene
Werkstoffe -
Teil 605: Physikalische Prüfungen -
Messung des Ruß- und/oder
Füllstoffgehalts in Polyethylenmischungen
(IEC 60811-605:2012)

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Foreword

The text of document 20/1314/FDIS, future edition 1 of IEC 60811-605, prepared by IEC/TC 20 "Electric cables" was submitted to the IEC-CENELEC parallel vote and approved by CENELEC as EN 60811-605:2012.

The following dates are fixed:

- latest date by which the document has to be implemented at national level by publication of an identical national standard or by endorsement (dop) 2013-01-17
- latest date by which the national standards conflicting with the document have to be withdrawn (dow) 2015-04-17

This document supersedes Clauses 11 and 12 of EN 60811-4-1:2004 (partially). Full details of the replacements are shown in Annex A of EN 60811-100:2012.

There are no technical changes with respect to EN 60811-4-1:2004, but see the Foreword to EN 60811-100:2012.

This standard is to be read in conjunction with EN 60811-100.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CENELEC [and/or CEN] shall not be held responsible for identifying any or all such patent rights.

This standard covers the Principle Elements of the Safety Objectives for Electrical Equipment Designed for Use within Certain Voltage Limits (LVD - 2006/95/EC)

Endorsement notice

The text of the International Standard IEC 60811-605:2012 was approved by CENELEC as a European Standard without any modification.

Annex ZA (normative)

Normative references to international publications with their corresponding European publications

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

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

<u>Publication</u>	<u>Year</u>	<u>Title</u>	<u>EN/HD</u>	<u>Year</u>
IEC 60811-100	2012	Electric and optical fibre cables - Test methods for non-metallic materials - Part 100: General	EN 60811-100	2012

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INTRODUCTION

The IEC 60811 series specifies the test methods to be used for testing non-metallic materials of all types of cables. These test methods are intended to be referenced in standards for cable construction and for cable materials.

NOTE 1 Non-metallic materials are typically used for insulating, sheathing, bedding, filling or taping within cables.

NOTE 2 These test methods are accepted as basic and fundamental and have been developed and used over many years principally for the materials in all energy cables. They have also been widely accepted and used for other cables, in particular optical fibre cables, communication and control cables and cables for ships and offshore applications.

ELECTRIC AND OPTICAL FIBRE CABLES – TEST METHODS FOR NON-METALLIC MATERIALS –

Part 605: Physical tests – Measurement of carbon black and/or mineral filler in polyethylene compounds

1 Scope

This Part 605 of IEC 60811 describes the test methods for measuring the content of carbon black added for UV stabilization of polyethylene and polyolefin compounds. These methods are not suitable for halogenated compounds.

Method A is suitable only for polyethylene and polypropylene compounds.

Method B is suitable for polyolefine compounds.

2 Normative references

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

IEC 60811-100:2012, *Electric and optical fibre cables – Test methods for non-metallic materials – Part 100: General*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC 60811-100 apply.

4 Test method

4.1 General

This part of IEC 60811 shall be used in conjunction with IEC 60811-100.

Unless otherwise specified, tests shall be carried out at room temperature.

4.2 Method A – Measurement of carbon black and/or mineral filler content in polyethylene by direct combustion

4.2.1 Sample and test piece preparation

A sample of the outer covering 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.

4.2.2 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 0,000 1 g. A sample of polyethylene

weighing $(1,0 \pm 0,1)$ g shall be placed in the boat and the whole weighed to the nearest 0,000 1 g. The weight of the boat shall be subtracted to give the weight of the polyethylene to the nearest 0,000 1 g (quantity A).

The boat and the sample shall then be placed in the middle of a hard glass, silica or porcelain combustion tube, with a bore approximately of 30 mm, and (400 ± 50) mm in length. 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 $(1,7 \pm 0,3)$ 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 then re-weighed. The weight of the residue is determined to the nearest 0,000 1 g (quantity B of residue).

Subsequently, the boat shall again be introduced 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 0,000 1 g (quantity C of residue).

4.2.3 Expression of results

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

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

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

4.3 Method B – Thermogravimetric analysis of the carbon black content in polyolefin compounds

NOTE This method may be used as an alternative to that in 4.2 when measuring carbon black content of polyethylene. In the event of dispute, the direct combustion method in 4.2 should be used as the reference method.

4.3.1 Principle

Heat a weighed test specimen 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 specimen with dry nitrogen with an oxygen content as described in 4.3.2. 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.

4.3.2 Reagents

The following reagents shall be used:

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

4.3.3 Apparatus

The apparatus comprises:

- a) a thermogravimetric analyser;
- b) a gas selector;
- c) a plotter or other suitable device;
- d) an analytical balance.

4.3.4 Procedure

4.3.4.1 Parameters of the apparatus

- a) starting temperature 100 °C;
- b) heating rate 20 K/min;
- c) end temperature 950 °C;
- d) weighed test specimen 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”.

4.3.4.2 Operation

Operate the apparatus according to the manufacturer's instructions and the parameters given in 4.3.4.1. Cover the bottom of the crucible with the test specimen, 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 as specified in 4.3.2 for at least 5 min.

4.3.4.3 Evaluation

The share of carbon black in the compound is determined for each single test specimen 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.

5 Test report

The test report shall be in accordance with that given in IEC 60811-100.

Bibliography

IEC 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 thermogravimetric analysis (TGA) – Assessment of carbon black dispersion in polyethylene using a microscope* (withdrawn)

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