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INTERNATIONAL STANDARD

NORME INTERNATIONALE

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

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

IEC 60811-605:2012

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CONTENTS

INTERNATIONAL ELECTROTECHNICAL COMMISSION

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

FOREWORD

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International Standard IEC 60811-605 has been prepared by IEC technical committee 20: Electric cables.

This Part 605 of IEC 60811 cancels and replaces Clauses 11 and 12 of IEC 60811-4-1:2004, which is withdrawn. Full details of the replacements are shown in Annex A of IEC 60811-100:2012.

There are no specific technical changes with respect to the previous edition, but see the foreword to IEC 60811-100:2012.

The text of this standard is based on the following documents:

FDIS	Report on voting
20/1314/FDIS	20/1363/RVD

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

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

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

A list of all the parts in the IEC 60811 series, published under the general title *Electric and optical fibre cables – Test methods for non-metallic materials*, can be found on the IEC website.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC web site under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

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- withdrawn,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

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 ± 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 ± 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

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

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

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)