



Edition 1.1 2017-07 CONSOLIDATED VERSION

# INTERNATIONAL STANDARD

# NORME INTERNATIONALE



Electric and optical fibre cables – Test methods for non-metallic materials – Part 410: Miscellaneous tests – Test method for copper-catalyzed oxidative degradation of polyolefin insulated conductors

Câbles électriques et à fibres optiques – Méthodes d'essai pour les matériaux non-métalliques –

Partie 410: Essais divers – Méthode d'essai pour la mesure de la dégradation par oxydation catalytique par le cuivre des conducteurs isolés aux polyoléfines





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INTERNATIONAL ELECTROTECHNICAL COMMISSION

COMMISSION ELECTROTECHNIQUE INTERNATIONALE

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# **REDLINE VERSION**

# **VERSION REDLINE**



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EC 60811-410:2012

# INTERNATIONAL ELECTROTECHNICAL COMMISSION

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

# Part 410: Miscellaneous tests – Test method for copper-catalyzed oxidative degradation of polyolefin insulated conductors

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IEC 60811-410 edition 1.1 contains the first edition (2012-03) [documents 20/1294/FDIS and 20/1343/RVD] and its amendment 1 (2017-07) [documents 20/1734/FDIS and 20/1739/RVD].

In this Redline version, a vertical line in the margin shows where the technical content is modified by amendment 1. Additions are in green text, deletions are in strikethrough red text. A separate Final version with all changes accepted is available in this publication.

- 4

International Standard IEC 60811-410 has been prepared by IEC technical committee 20: Electric cables.

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

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 the base publication and its amendment 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

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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.

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

# Part 410: Miscellaneous tests – Test method for copper-catalyzed oxidative degradation of polyolefin insulated conductors

# 1 Scope

This Part 410 of IEC 60811 gives the procedure for copper-catalyzed oxidative degradation of a polyolefin, which is typically used for insulation in communication cables.

Full test conditions, such as temperature, duration, 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.

## 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

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

# 4.2 Apparatus

For the purposes of this test, the different equipment used is as follows:

- a) A differential thermal analyser or differential scanning calorimeter, capable of heating at rates of up to at least (20  $\pm$  1) K/min and maintaining the test temperature isothermally within 0,2 K and of automatic recording of differences in temperature (or differences in heat transfer) between the sample and a reference material to the required sensitivity and precision.
- b) A recorder capable of displaying heat flow or temperature difference on the Y-axis, and time on the X-axis. The time base shall be accurate to  $\pm$  1 % and be readable to 0,1 min.

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- c) A gas-selector switch and regulators for high-purity nitrogen and oxygen;  $N_2$ ,  $O_2$  with purity:
  - $N_2 5.0 = 99,999 \%$  purity;
  - $O_2$  4.5 = 99,995 % purity.
- d) An analytical balance capable of weighing  $\frac{30 \text{ g}}{}$  3 mg to 5 mg, and readable and repeatable to  $\frac{\pm 0,1 \text{ mg}}{} \pm 1 \text{ µg}$ . The mass rounding is in 10 µg.
- e) Test pieces holders: aluminium holders, each of approximately 6 mm to 7 mm in diameter, or of similar dimensions as supplied by the manufacturer of the instrument.

# 4.3 Sample and test pieces preparation

From a sample of the cable, prepare an appropriate number, e.g. four test pieces of different colours, of approximately 4 mm length containing the core. The test pieces shall be cut from an insulated core to yield 3 mg to 5 mg of insulating material.

Insert one test piece in an aluminium holder.

## 4.4 Test procedure

Before carrying out a test series, the instrument shall be calibrated and prepared as described in Annex A.

Open valves on both the nitrogen and oxygen gas cylinders. Place the gas selector switch to the nitrogen  $(N_2)$  position and adjust the flow rate to  $(50 \pm 5)$  ml/min using a flowmeter.

Place the prepared test piece holder (see 4.3) in the differential thermal analyser and an empty aluminium holder in the reference position.

NOTE Crimping the test piece with a suitable aluminium or stainless steel screen is optional. It may provide a better contact with the test piece holder. If the piece holder is closed, the oxidation cannot be made.

Purge with nitrogen for 5 min. Check the flow rate and readjust to (50  $\pm$  5) ml/min if required.

Set signal amplification and recorder sensitivity to the full scale pen deflection associated with the exothermic reaction and set the temperature base at zero.

Set the heating rate to 20 K/min and start the programmed heating.

Continue heating until the specified test temperature, controlled to  $\pm$  1 °C, is reached. Discontinue programmed heating and equilibrate the test piece to a constant temperature. Start to record the thermogram. A test temperature of 200 °C has been found appropriate for polyethylene. To simplify the procedure, it is allowed to omit the programmed pre-heating and start directly at the test temperature.

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

Continue the isothermal operation until maximum pen deflection is attained after commencement of the oxidative exotherm as shown on the recorder (see Figure 1).

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

When the test is completed, turn off the recorder and switch the gas selector back to nitrogen.

Allow the differential thermal analyser to cool to the ambient temperature.

Repeat the entire test on a new test piece, three more times, thus generating a total of four thermograms. The use of a fresh aluminium reference holder for each test piece is optional.

### 4.5 Measurements

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

The OIT shall be measured from zero to the smallest time interval practical, not exceeding 1 min.

# 5 Test report

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

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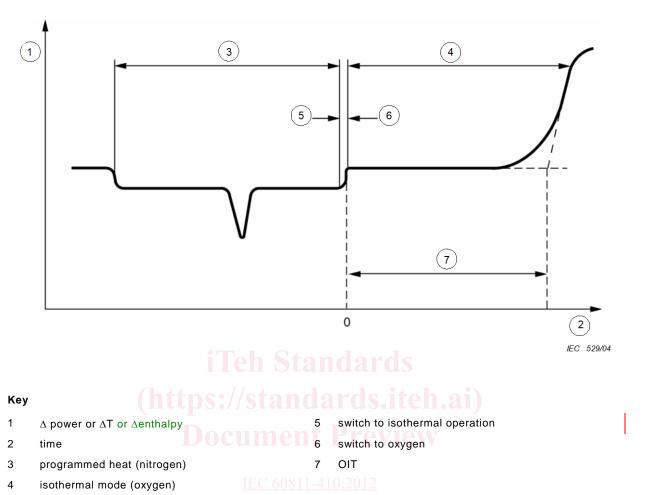


Figure 1 – Evaluation of OIT from recorded-time-based thermogram

# Annex A (normative)

# Instrument calibration

The instruments shall be calibrated as follows:

- a) Calibrate the instrument according to the manufacturer's instructions before starting. Use analytical-grade indium as a temperature reference material.
- b) Place analytical-grade indium into an aluminium holder with an aluminium cover. Place the sample, typically 6 mg, thus prepared, and a reference aluminium holder with cover into the instrument.
  - Should it be necessary to clean the sample and the aluminium reference holder and cover, use petroleum ether or other suitable solvent to remove contaminants.
- c) Programme the temperature of the scanner from 145 °C to 165 °C at a rate of 1 K/min, while recording the thermogram.
- d) Calibrate the instrument according to the manufacturer's instructions in order to obtain an indium first order transition temperature of 156,6 °C. For calibration purposes, the melting point 156,6 °C is defined as the intersection of the extrapolated peak onset and the extrapolated baseline (see Figure A.1).

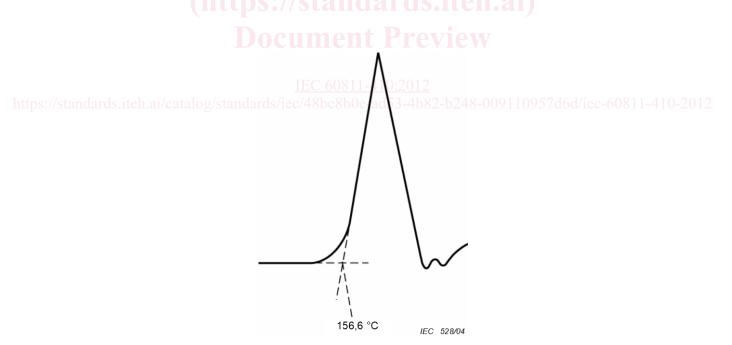


Figure A.1 – Representative melting endothermic for indium