IEC 60811-603

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INTERNATIONAL STANDARD

NORME INTERNATIONALE

Electric and optical fibre cables – Test methods for non-metallic materials – Part 603: Physical tests – Measurement of total acid number of filling compounds

Câbles électriques et à fibres optiques – Méthodes d'essai pour les matériaux non-métalliques – Partie 603: Essais physiques – Mesure de l'indice d'acide total des matières de remplissage

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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

Part 603: Physical tests – Measurement of total acid number of filling compounds

FOREWORD

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International Standard IEC 60811-603 has been prepared by IEC technical committee 20: Electric cables.

This Part 603 of IEC 60811 cancels and replaces Clause 7 of IEC 60811-5-1:1990, which is withdrawn.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
20/1312/FDIS	20/1361/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- withdrawn,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 603: Physical tests – Measurement of total acid number of filling compounds

1 Scope

This Part 603 of IEC 60811 gives the test methods to examine the filling compound for corrosive elements.

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.

This test is used to examine the filling compound for corrosive elements.

The total acid number is defined as the quantity of base, expressed in milligrams of potassium hydroxide (KOH), that is required to titrate all acidic constituents present in 1 g of sample.

4.2 Apparatus

The apparatus comprises a 50 ml burette graduated in 0,1 ml subdivisions or a 10 ml burette graduated in 0,05 ml subdivisions.

4.3 Reagents

4.3.1 General

The reagent shall be of an analytical reagent quality.

Distilled water shall be used throughout.

4.3.2 Potassium hydroxide solution, standard alcoholic (0,1 N)

Add 6 g of solid KOH to approximately 1 l of anhydrous isopropyl alcohol (containing less than 0,9 % water) in a 2 l Erlenmeyer flask. Boil the mixture gently for 10 min to 15 min, stirring to prevent the solids from forming a cake on the bottom. Add at least 2 g of barium hydroxide ($Ba(OH)_2$) and again boil gently for 5 min to 10 min. Cool to room temperature, allow to stand for several hours, and filter the supernatant liquid through a fine sintered glass or porcelain filtering funnel. Avoid unnecessary exposure to carbon dioxide (CO_2) during filtration. Store the solution in a chemically resistant dispensing bottle out of contact with cork, rubber or saponifiable stopcock lubricant and protected by a guard tube containing soda, lime or soda asbestos. Standardize frequently enough to detect changes of 0,000 5 N, preferably against pure potassium acid phthalate in about 100 ml of CO_2-free water using phenolphthalein to detect the end point.

NOTE 1 To simplify calculations, the standard KOH solution may be adjusted so that 1,00 ml is equivalent to 5,00 mg of KOH.

NOTE 2 Sodium hydroxide (NaOH) may be substituted for KOH.

4.3.3 p-Naphtholbenzein indicator solution

Dissolve 10 g of p-naphtholbenzein per litre of titration solvent as defined in 4.3.4.

p-Naphtholbenzein shall conform with Annex A.

4.3.4 Titration solvent

Add 500 ml of toluene and 5 ml of water to 495 ml of anhydrous isopropyl alcohol.

4.4 Test procedure

Introduce approximately 25 g of the filling compound, weighed to the nearest 0,1 g, into a 250 ml Erlenmeyer flask. Add 100 ml of the titration solvent and 0,5 ml of the indicator solution and, without stoppering, swirl until the sample is entirely dissolved by the solvent. Titrate immediately at a temperature below 30 °C. Add 0,1 N KOH solution in increments and swirl to disperse the KOH as necessary. Shake vigorously near the end point, but avoid dissolving carbon dioxide (CO_2) in the solvent.

The end point is considered to have occurred when the colour change persists for 15 s or if it reverses with two drops of 0,1 N HCl.

NOTE In the case of acidic compounds, the orange colour changes to green or green-brown as the end-point is approached.

Make a blank titration on 100 ml of the titration solvent and 0,5 ml of the indicator solution and add 0,1 N KOH solution in 0,05 ml or 0,1 ml increments. Record the quantity of 0,1 N KOH solution required to reach the end point (orange to green).

4.5 Calculation

The total acid number can be calculated as follows:

$$\text{Total acid number, mg of KOH/g} = \frac{(A - B)N \times 56{,}1}{W}$$

where

A is the number of millilitres of KOH solution required for titration of the sample;

B is the number of millilitres of KOH solution required for titration of the blank;

N is the normality of the KOH solution;

W is the amount of grams of sample used.

5 Test report

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

Annex A
(normative)

Specification for p-Naphtholbenzein

A.1 Appearance

p-Naphtholbenzein shall be in the form of a red amorphous powder.

A.2 Chlorides

It shall contain less than 0,5 % chlorides.

A.3 Solubility

A quantity of 10 g shall dissolve completely in 1 l of titration solvent defined in 4.3.4.

A.4 Minimum absorbance

After having dissolved exactly 0,1 000 g of the sample in 250 ml of methanol and having converted 5 ml of this solution to 100 ml with the aid of a pH 12 buffer, the final dilution shall have a minimum absorbance of 1,20 when read at 6,50 µm peak, using a Beckmann DU or alternative type spectrophotometer, 1 cm cells and water serving as the blank.

A.5 pH range

The indicator shall turn to the first clear green at a relative pH of 11 ± 0,5 when tested by the method for pH range of the p-Naphtholbenzein indicator defined in 4.3.3.

Not more than 0,5 ml of 0,01 N KOH above that for the blank shall bring the indicator solution to the first clear green. Not more than 1,0 ml of 0,01 N KOH above that for the blank shall turn the indicator solution to a blue colour.

The initial pH of the indicator solution shall be at least as high as that of the blank.