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Test on gases evolved during combustion of materials from cables – Part 1: Determination of the halogen acid gas content

Essai sur les gaz émis lors de la combustion des matériaux prélevés sur câbles – Partie 1: Détermination de la quantité de gaz acide halogéné

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

TEST ON GASES EVOLVED DURING COMBUSTION OF MATERIALS FROM CABLES –

Part 1: Determination of the halogen acid gas content

FOREWORD

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International Standard IEC 60754-1 has been prepared by IEC technical committee 20: Electric cables.

It has the status of a group safety publication in accordance with IEC Guide 104.

This third edition cancels and replaces the second edition, published in 1994, and constitutes a technical revision.

The significant technical changes with respect to the previous edition are as follows:

– improved definition of safety requirements relating to capture of gases and use of reagents;
– introduction of guidance on preparation of test specimens for a more even combustion;
– improvements to the procedure for establishing the heating regime;
– improved expression of tolerances and precision;

– definition of the procedure for the blank test;

– introduction of an informative annex giving details of a methodology for the determination of the halogen acid gas content of a sample representative of a cable construction.

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

FDIS	Report on voting
20/1266/FDIS	20/1276/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.

A list of all the parts in the IEC 60754 series, published under the general title *Test on gases evolved during combustion of materials from cables*, 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- withdrawn,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.

The contents of the corrigendum of November 2013 have been included in this copy.

INTRODUCTION

IEC 60754 consists of the following parts, under the general title *Test on gases evolved during combustion of materials from cables*:

– *Part 1: Determination of the halogen acid gas content*

– *Part 2: Determination of acidity (by pH measurement) and conductivity*

IEC 60754-1 was developed due to concerns expressed by cable users over the amount of acid gas which is evolved when some cable insulating, sheathing and other materials are burned, as this acid can cause extensive damage to electrical and electronic equipment not involved in the fire itself.

This standard provides a method for determining the amount of acid gases evolved by burning cable components so that limits can be agreed for cable specifications. As the test is not carried out on a complete cable test piece, for a hazard assessment the actual material volumes of the cable components should be taken into consideration.

TEST ON GASES EVOLVED DURING COMBUSTION OF MATERIALS FROM CABLES –

Part 1: Determination of the halogen acid gas content

1 Scope

This part of IEC 60754 specifies the apparatus and procedure for the determination of the amount of halogen acid gas, other than hydrofluoric acid, evolved during the combustion of compounds based on halogenated polymers and compounds containing halogenated additives taken from electric or optical fibre cable constructions.

NOTE 1 This test method is not able to determine hydrofluoric acid. A suitable method may be found in IEC 60684-2.

NOTE 2 This test method may be used to test materials to be used in cable manufacture, but a declaration of cable performance should not be made based on such a test.

NOTE 3 The relevant cable standard should indicate which components of the cable should be tested.

NOTE 4 For the purposes of this standard, the term “electric cable” covers all insulated metallic conductor cables used for the conveyance of energy or signals.

The method specified in this standard is intended for the testing of individual components used in a cable construction. The use of this method will enable the verification of requirements which are stated in the appropriate cable specification for individual components of a cable construction.

NOTE 5 By agreement between the producer and purchaser, the methodology given in this standard may be used to test combinations of materials representing a cable construction, but a declaration of cable performance to this standard should not be made based on such a test. Information on such a method is given in Annex A.

For reasons of precision this method is not recommended for reporting values of halogen acid evolved less than 5 mg/g of the sample taken.

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.

ISO 385, *Laboratory glassware – Burettes*

ISO 1042, *Laboratory glassware – One-mark volumetric flasks*

ISO 3696, *Water for analytical laboratory use – Specification and test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
halogen acid gas content

amount of halogen acid gas evolved, except hydrofluoric acid, expressed as milligrams of hydrochloric acid per gram of total test specimen

4 Test method principle

The material under test shall be heated in a stream of dry air and the gases shall be absorbed in 0,1 M sodium hydroxide solution contained in wash bottles. The amount of halogen acid shall then be determined by acidifying the solution with nitric acid, adding a measured volume of 0,1 M silver nitrate solution and back titrating the excess with 0,1 M ammonium thiocyanate, using ferric ammonium sulphate as the indicator.

NOTE 1 Other analytical methods having at least the same precision may be used, but in case of dispute the method given in this standard is the one to use.

NOTE 2 Although both hydrogen chloride and hydrogen bromide are detected by this analytical method, the halogen acid content is reported as if all the halogen acid is hydrogen chloride.

5 Test apparatus

5.1 General

The apparatus is shown in Figures 1 to 5.

The assembly of the components which constitute the test apparatus shall be leak-tight. The connecting distances between the quartz glass tube and the first bottle and between subsequent bottles shall be as short as possible. Glass or silicone rubber tubing shall be used for these connections.

NOTE 1 At the exit side of the quartz glass tube, as close to the end as possible, it is permitted to place a plug of silica wool to aid collection of condensates.

NOTE 2 A third empty bottle, of the same size as the gas washing bottles, placed before the gas washing bottles, may be used to improve safety, i.e. to prevent suck back of water into the quartz glass tube.

5.2 Tube furnace

The length of the heating zone of the furnace shall be within the range 480 mm to 620 mm, and its inside diameter shall be within the range 38 mm to 62 mm. It shall be equipped with an adjustable electrical heating system.

5.3 Quartz glass tube

For the test, a quartz glass tube shall be introduced into the tube furnace. The tube shall be approximately concentric to the furnace. It shall be resistant to the action of corrosive gases.

The inside diameter of the tube shall be within the range 30 mm to 46 mm. The tube shall protrude on the entrance side of the furnace by a length of between 60 mm to 200 mm, and on the exit side by between 60 mm to 100 mm. The initial clearance shall allow for thermal expansion. For the purposes of measurement of the protrusion distances, the tube shall be regarded as that part of essentially constant diameter.

NOTE The outer diameter of the tube should be chosen with due regard to the inside diameter of the tube furnace.

Prior to each test, the tube shall be cleaned throughout its length by being calcined at approximately 950 °C.

5.4 Combustion boats

The combustion boat shall be made of porcelain, fused quartz or soapstone and shall have the following dimensions:

– external length: within the range 45 mm to 100 mm;

– external width: within the range 12 mm to 30 mm;
– internal depth: within the range 5 mm to 10 mm.

NOTE The dimensions of the boat should be chosen with due regard to the inside diameter of the quartz tube.

The preferred method for insertion of the combustion boat into the quartz glass tube is shown in Figure 1.

Prior to each test, the combustion boat shall be washed and calcined in a muffle furnace at approximately 950 °C for 4 h after which it shall be introduced into a desiccator and cooled to ambient temperature. The combustion boat shall then be weighed to an accuracy of 0,1 mg. This weight m_1 shall be recorded.

5.5 Bubbling devices for gases

At the exit of the quartz glass tube, the evolved gases shall be passed through two wash bottles (see Figure 2), each containing at least 220 ml of 0,1 M sodium hydroxide solution.

A magnetic stirrer shall be introduced in the first gas washing bottle to get a good swirling motion and an effective absorption of the combustion gases. The tubes into the wash bottles shall have a maximum internal diameter at their tip of 5 mm, in order to aid absorption.

The height of the liquid above the end of the tube shall be (110 ±10) mm in each bottle.

NOTE Use of a standard laboratory glass bottle of approximately 50 mm internal diameter will enable this requirement to be met.

5.6 Air supply system

The gas used for combustion shall be air.

The flow rate of air introduced into the quartz glass tube shall be adjusted according to the actual internal cross-sectional area of the tube, such that the speed of air flowing across the sample is approximately 20 ml/mm^2/h.

The speed of air shall be regulated by reference to the flow rate of air. The flow rate of air shall be (0,0157 × D^2) l/h with a tolerance of ±10 %.

NOTE The derivation of the flow rate of air from the speed of air is:

$$\rho = V \times \frac{\pi D^2}{4}$$

where

D is the internal diameter of the tube (mm);

ρ is the flow rate of air (ml/h);

V is the speed of air (ml/mm^2/h).

The air supply shall be adjusted and controlled by a needle valve, and the flow rate monitored by a flowmeter of the appropriate range.

The air supplied shall be selected from one of the following methods:

Method 1

This method uses synthetic air or compressed air from a bottle. The air shall be introduced on the inlet side of the quartz glass tube (see Figure 3).

Method 2

This method uses a laboratory compressed air supply. The air shall be introduced on the inlet side of the quartz glass tube and shall be be filtered and dried (see Figure 4).

Method 3

This method uses the ambient air of the laboratory. The air shall be filtered and dried. In this case, the mixture of air and combustion gas shall be sucked by a pump. (See Figure 5.)

5.7 Analytical balance

The balance shall have a precision of ±0,1 mg.

5.8 Laboratory glassware

For the titration, the following laboratory glassware shall be available:

– 20 ml pipette;
– 100 ml pipette;
– one mark volumetric flask in accordance with ISO 1042 with 1 000 ml capacity;
– conical flask with 250 ml to 500 ml capacity;
– burette in accordance with ISO 385-1.

5.9 Reagents

For the analysis, the following reagents of a recognized analytical quality shall be used. Demineralized or distilled water shall be of a purity at least Grade 3 in accordance with ISO 3696.

a) concentrated nitric acid: about 65 %, with a specific gravity ρ of approximately 1,40 g/ml;
b) nitric acid, approximately 6 M;
c) 0,1 M silver nitrate;
d) nitrobenzene, toluene or iso-amyl alcohol;
e) an approximately 40% weight/volume solution of ferric ammonium sulphate;
f) 0,1 M ammonium thiocyanate solution.

WARNING Nitrobenzene is regarded as highly toxic. Toluene or iso-amyl alcohol are safer alternatives.

6 Test specimen

6.1 General

Two test specimens, each consisting of (750 ± 250) mg of the material to be tested, shall be prepared. Each test specimen shall be taken from a sample representative of the material. Each test specimen shall be cut into a number of smaller pieces.

NOTE Pieces with a maximum dimension of 3 mm have been found to be suitable.

6.2 Conditioning of specimen

The prepared test specimens shall be conditioned for at least 16 h at a temperature of (23 ±2) °C and a relative humidity of (50 ± 5) %.

6.3 Mass of specimen

Weigh the combustion boat (m_1) to an accuracy of 0,1 mg (see 5.4). After conditioning, the test specimen shall be put into the combustion boat and evenly distributed on the bottom of the boat, which shall be weighed to an accuracy of 0,1 mg, The weight (m_2) shall be recorded.

The mass m of the test specimen shall be calculated as follows:

$$m = m_2 - m_1$$

where

m is the mass of the test specimen in grams;

m_1 is the mass of the combustion boat in grams;

m_2 is the mass of the combustion boat with the test specimen, in grams.

NOTE Modern weighing equipment with suitable automatic zeroing could allow direct measurement of m.

7 Test procedure

7.1 General

The test procedure and determination shall be carried out on each test specimen.

7.2 Test apparatus and arrangement

The test procedure defined in this clause shall be carried out using the apparatus detailed in Clause 5.

7.3 Heating procedure

7.3.1 Determination of heating regime

The empty combustion boat shall be inserted into the quartz glass tube and placed approximately in the centre of the tube furnace.

The flow rate of air shall be adjusted by means of a needle valve to the value specified in 5.6 and shall be kept constant during the determination.

Position a thermocouple, or other suitable temperature measuring device (suitably protected against corrosion), at the test specimen point in the empty combustion boat. The combustion boat shall be heated at an approximately uniform heating rate over a period of (40 ± 5) min in order to raise the temperature recorded by the thermocouple to (800 ± 10) °C, after which it shall be maintained at that temperature for (20 ± 1) min.

Determine from this procedure a heating regime which will ensure that the required test specimen heating rate and temperature is achieved.

7.3.2 Test specimen heating procedure

The combustion boat containing the test specimen shall be inserted into the quartz glass tube and placed approximately in the centre of the tube furnace.