IEC 60754-3

Edition 1.0 2018-03

INTERNATIONAL STANDARD

NORME INTERNATIONALE

Test on gases evolved during combustion of materials from cables –
Part 3: Measurement of low level of halogen content by ion chromatography

Essai sur les gaz émis lors de la combustion des matériaux prélevés sur câbles –
Partie 3: Mesure d'une faible teneur en halogène par chromatographie ionique

IEC 60754-3:2018-03(en-fr)

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TEST ON GASES EVOLVED DURING COMBUSTION OF MATERIALS FROM CABLES –

Part 3: Measurement of low level of halogen content by ion chromatography

FOREWORD

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International Standard IEC 60754-3 has been prepared by IEC technical committee 20: Electric cables.

This bilingual version (2018-11) corresponds to the monolingual English version, published in 2018-03.

The text of this International Standard is based on the following documents:

FDIS	Report on voting
20/1784/FDIS	20/1791/RVD

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

The French version of this standard has not been voted upon.

This document 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 document will remain unchanged until the stability date indicated on the IEC website under "http://webstore.iec.ch" in the data related to the specific document. At this date, the document will be

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- withdrawn,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

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*

– Part 3: *Measurement of low level of halogen content by ion chromatography*

NOTE Guidance on the corrosivity of fire effluent is given in IEC 60695-5-1.

IEC 60754-1 and IEC 60754-2 were 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 and such corrosive effluents can cause extensive damage to electrical and electronic equipment not involved in the fire itself.

IEC 60754-1 provides a method for determining the amount of acid gases evolved by burning cable components so that limits can be agreed for cable specifications.

IEC 60754-2 provides a method for determining the acidity (by pH measurement) and conductivity of an aqueous solution of gases evolved during the combustion of materials so that limits can be agreed for cable specifications

IEC 60754-1 is not able to determine hydrofluoric acid and, 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.

This document provides a method for measurement of low level of halogen content of the gases evolved by burning cable and has a high accuracy in the low range of concentration.

The ion chromatic system has an inherently high accuracy. However, the overall accuracy of the test method is limited by other factors (see Annex A for further information).

This part of IEC 60754 is linked with IEC 60754-2, using the same test procedure for obtaining the absorption solution.

TEST ON GASES EVOLVED DURING COMBUSTION OF MATERIALS FROM CABLES –

Part 3: Measurement of low level of halogen content by ion chromatography

1 Scope

This part of IEC 60754 specifies the apparatus and procedure for the measurement of the amount of halogens evolved during the combustion of materials taken from electric or optical fibre cable constructions.

The method specified in this document is intended for the measurement of the content of chlorine (Cl), bromine (Br), fluorine (F) and iodine (I), by using the analytical technique of ion chromatography for analysing an aqueous solution resulting from the gases evolved during the combustion.

The heating (combustion) procedure in this part of IEC 60754 is the same as in IEC 60754-2.

The method is intended for materials with an individual halogen content not exceeding 10 mg/g.

The method specified in this document 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 1 The relevant cable standard indicates which components of the cable are tested.

NOTE 2 This test method is sometimes used to test materials to be used in cable manufacture.

For reasons of precision, this method is not recommended for detecting values of halogens less than 0,1 mg/g of the sample taken.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1042, *Laboratory glassware – One-mark volumetric flasks*

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

ISO 10304-1, *Water quality – Determination of dissolved anions by liquid chromatography of ions – Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1
halogen
element of periodic table group 17, specifically fluorine, chlorine, bromine, iodine

3.2
individual halogen content
amount of a halogen in a test specimen that can be converted to water soluble halide ion in the gaseous combustion effluent from the test specimen

Note 1 to entry: Halide ions are fluoride, chloride, bromide or iodide.

Note 2 to entry: The halogen in the test specimen may be in the form of organic or inorganic compounds.

Note 3 to entry: The individual halogen content is expressed as milligrams of halogen per gram of test specimen.

Note 4 to entry: Halide ions in any solid combustion residue that remains in the combustion boat are not measured by this test method.

4 Test method principle

The material under test shall be heated in a stream of dry air. The evolved gases shall be trapped by bubbling through wash bottles filled with distilled or demineralized water.

Each individual halogen content of the resulting solution shall then be determined by using ion chromatography (IC).

The ion chromatography analysis can be done off line and does not have to be in line.

5 Test apparatus

5.1 General

The apparatus is shown in Figure 1 to Figure 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. Quartz glass or polypropylene tubing shall be used for these connections, unless it is not required to measure and report fluorine, in which case glass or silicone rubber may be used.

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.

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.

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 boat

The combustion boat shall be made of porcelain, fused quartz or soapstone. In case the method is used to report fluorine, the combustion boat shall be made of quartz glass. The combustion boat 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.

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 with a precision of ≤ 0,1 mg until two identical consecutive weights are obtained. 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 trapped by bubbling through two wash bottles (see Figure 2), each containing approximately 450 ml of distilled or demineralized water of a purity at least Grade 3 in accordance with ISO 3696.

The wash bottles shall be made of quartz glass, unless it is not required to measure and report fluorine, in which case wash bottles made of glass may be used.

The pH value of the water shall be between 5,5 and 7,5, and the conductivity less than 0,5 µS/mm.

A magnetic stirrer shall be introduced in the first wash 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 wash bottle of internal diameter approximately 75 mm 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, ρ, shall be 20 m/h x (π/4) x D^2 with a tolerance of ± 10 %, where D is the internal diameter of the quartz tube.

EXAMPLES

If D = 30 mm, 20 m/h × (π/4) × D^2 = 14,1 l/h, and the flow rate can be in the range 12,7 l/h to 15,5 l/h.

If D = 46 mm, 20 m/h × (π/4) × D^2 = 33,2 l/h, and the flow rate can be in the range 29,9 l/h to 36,5 l/h.

NOTE The flow rate of air, ρ, is related to the velocity, v, according to the formula

$$\rho = v \times \frac{\pi D^2}{4} \quad [1]$$

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).

If v = 20 m/h, this becomes, ρ = 15,7 m/h × D^2

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 filtered and dried and shall be introduced on the inlet side of the quartz glass tube (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 or better.

5.8 Laboratory glassware

The following laboratory glassware shall be available:

– one mark volumetric flask in accordance with ISO 1042 class B with 1 000 ml capacity.

The volumetric flask shall be made of quartz glass or polypropylene, unless it is not required to measure and report fluorine, in which case a volumetric flask made of glass may be used.

5.9 Ion chromatographic system

In general, the ion chromatographic system consists of the following components (see Figure 6):

– eluent reservoir;
– IC pump;
– sample injection system, incorporating a sample loop of appropriate volume (e.g. 0,02 ml) or auto sample device;
– precolumn or guard column;
– separation column;
– suppressor;
– conductivity detector.

6 Test specimen

6.1 General

Two test specimens, each consisting of (1 000 ± 5) mg of the material to be tested, shall be prepared. Each test specimen shall be taken from a cable 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 stored 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) with a precision 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 with a precision of ≤ 0,1 mg. The weight (m_2) shall be recorded.

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

$$m = m_2 - m_1 \quad [2]$$

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.

7 Test procedure

7.1 General

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

After a satisfactory blank test procedure (see 7.2), the test procedure and measurement of the individual halogen content shall be carried out on each test specimen.

7.2 Blank test

A blank test, i.e. with no test specimen in the boat, shall be carried out under the same conditions as when a test specimen is present.

If the result of this blank test is too high compared with the limit of measurement, check that all components of the test apparatus are clean, especially the boat and quartz glass tube. Repeat the blank test procedure until a satisfactory result is obtained.

7.3 Test apparatus and arrangement

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

7.4 Heating procedure

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

The temperature shall be measured by a thermocouple suitably protected against corrosion and placed inside the quartz glass tube. The heating system shall be adjusted such that the temperature at the designated position for the boat shall be not less than 935 °C and not more than 965 °C. The temperature at a position approximately 300 mm from the designated position in the direction of the air flow shall be not less than 900 °C.

The boat containing the test specimen shall be quickly inserted into the heating zone of the tube to the designated position determined by the thermocouple measurement and the timer shall be started. The combustion boat shall be placed in such a way that the distance between the boat and the exit end of the effective heating zone is at least 300 mm. The effective heating zone is that zone where a temperature of not less than 900 °C has been determined by the thermocouple measurement.

The combustion procedure, under the air flow condition, shall be continued for (30 ± 1) min in the furnace.

WARNING The operator should take precautions, e.g. the wearing of eye protection and suitable protective clothing, because certain materials ignite quickly and can cause "blow back" of hot gases. Care should also be taken to avoid over-pressurization of the system, and to allow for venting of exhaust gases. Guidance on the avoidance of "blow back" is given in 5.1.

7.5 Washing procedure

Following the heating procedure, all the bottles used shall be disconnected, and the contents washed into a 1 000 ml volumetric flask. Using water with the properties given in 5.5, the bottles, the connecting links and, after cooling, the end of the quartz glass tube including the silica wool (if used) shall also be washed into the flask, and the contents made up to the 1 000 ml mark.

7.6 Measurement of the halogens

The absorption solution obtained shall be analyzed using ion chromatography.

The ion chromatographic analysis shall be carried out according to ISO 10304-1.

a) Set up the IC equipment according to the instrument manufacturer’s instructions.
b) Run the eluent and wait for a stable baseline.
c) Perform the calibration.
d) Measure the samples.

For the purpose of this test, five calibration solutions shall be made up with concentrations of the anions in the range of 0,1 mg/g to 1 mg/g.

The concentration of anions in the absorption solution may be higher than 1 mg/g. Therefore, in order to complete the analysis, a dilution shall be made and an appropriate correction applied. The individual halogen content, expressed as milligrams per gram of test specimen taken, shall be determined for each halogen.

In case where the ion chromatography analysis cannot be performed immediately, the absorption solution should be stored at not more than 6 °C in a polypropylene container or otherwise as recommended by the IC specialist.

8 Evaluation of the test results

Calculate the individual halogen content, in units of mg/g, using the equation [3]:

$$X_i = (C_i \cdot V)/m \quad [3]$$

where

X_i is the individual halogen content ("i" = Cl, Br, I or F);

C_i is the concentration of this element, "i", in the absorption solution;

V is the final volume of the absorption solution;

m is the mass of the test specimen.

EXAMPLE In the case where the concentration of chloride ion in the absorption solution, C_{Cl}, is 1 mg/l, the final volume of the absorption solution is 1 000 ml and the mass of the test specimen is 1 000 mg, then X_{Cl} becomes (1 mg/l × 1 000 ml / 1 000 mg) = (1 mg/l × 1 l / 1 g) = 1 mg/g.

The individual halogen content shall be taken as the mean of the determination of the two test specimens.

If the difference in two results is greater than 0,1 mg/g and if the ratio of the standard deviation to the mean is greater than 0,25, the test results should be discarded and the test procedure should be repeated.

9 Performance requirement

No performance requirements are included in this document.

If no performance requirement is given in the relevant cable specification, it is recommended that the guidance in Annex A is followed.

10 Test report

The test report shall include the following information:

a) a full description of the material tested and the cable from which it was taken;

b) the number of this document;

c) the individual halogen content for fluorine, for chlorine, for bromine and for iodine;

d) a description of the ion chromatography equipment.