

BS EN 60754-1:2014



BSI Standards Publication

# Test on gases evolved during combustion of materials from cables

Part 1: Determination of the halogen acid gas content

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**National foreword**

This British Standard is the UK implementation of EN 60754-1:2014. It is identical to IEC 60754-1:2011, incorporating corrigendum November 2013. Together with BS EN 60754-2:2014 it supersedes BS EN 50267-1:1999, BS EN 50267-2-1:1999, BS EN 50267-2-2:1999 and BS EN 50267-2-3:1999 which will be withdrawn on 27 January 2017.

IEC corrigendum November 2013 corrects the French language title.

The UK participation in its preparation was entrusted by Technical Committee GEL/20, Electric cables, to Subcommittee GEL/20/18, Electric Cables - Fire testing.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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**Amendments/corrigenda issued since publication**

Date	Text affected
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English version

**Test on gases evolved during combustion of materials from cables -  
Part 1: Determination of the halogen acid gas content  
(IEC 60754-1:2011 + corrigendum Nov. 2013)**

Essai sur les gaz émis lors de la  
combustion des matériaux prélevés  
sur câbles -  
Partie 1: Détermination de la quantité  
de gaz acide halogéné  
(CEI 60754-1:2011 + corrigendum Nov.  
2013)

Prüfung der bei der Verbrennung der  
Werkstoffe von Kabeln und isolierten  
Leitungen entstehenden Gase -  
Teil 1: Bestimmung des Gehaltes an  
Halogenwasserstoffsäure  
(IEC 60754-1:2011 + Corrigendum Nov.  
2013)

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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CENELEC member.

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Comité Européen de Normalisation Electrotechnique  
Europäisches Komitee für Elektrotechnische Normung

**CEN-CENELEC Management Centre: Avenue Marnix 17, B - 1000 Brussels**

## Foreword

This document (EN 60754-1:2014) consists of the text of IEC 60754-1:2011 + corrigendum November 2013, prepared by IEC/TC 20 "Electric cables".

The following dates are fixed:

- latest date by which the document has to be implemented at national level by publication of an identical national standard or by endorsement (dop) 2015-01-27
- latest date by which the national standards conflicting with the document have to be withdrawn (dow) 2017-01-27

This document supersedes EN 50267-1:1998 (PART), EN 50267-2-1:1998 (PART), EN 50267-2-2:1998 (PART) and EN 50267-2-3:1998 (PART).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CENELEC [and/or CEN] shall not be held responsible for identifying any or all such patent rights.

This standard covers the Principle Elements of the Safety Objectives for Electrical Equipment Designed for Use within Certain Voltage Limits (LVD - 2006/95/EC).

## Endorsement notice

The text of the International Standard IEC 60754-1:2011 + corrigendum November 2013 was approved by CENELEC as a European Standard without any modification.

In the official version, for Bibliography, the following note has to be added for the standard indicated :

IEC 60684-2      NOTE      Harmonized as EN 60684-2.

**Annex ZA**  
(normative)**Normative references to international publications  
with their corresponding European publications**

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.

NOTE When an international publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

<u>Publication</u>	<u>Year</u>	<u>Title</u>	<u>EN/HD</u>	<u>Year</u>
ISO 385	-	Laboratory glassware - Burettes	EN ISO 385	-
ISO 1042	-	Laboratory glassware - One-mark volumetric flasks	EN ISO 1042	-
ISO 3696	-	Water for analytical laboratory use - Specification and test methods	EN ISO 3696	-

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## 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  $(110 \pm 10)$  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<sup>2</sup>/h.

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

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);

$\rho$  is the flow rate of air (ml/h);

$V$  is the speed of air (ml/mm<sup>2</sup>/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 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  $\pm 0,1$  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  $\rho$  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  $(750 \pm 250)$  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  $(23 \pm 2)$  °C and a relative humidity of  $(50 \pm 5)$  %.

### 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  $(40 \pm 5)$  min in order to raise the temperature recorded by the thermocouple to  $(800 \pm 10)$  °C, after which it shall be maintained at that temperature for  $(20 \pm 1)$  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.

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 test.

The test specimen shall be heated using the heating regime determined in 7.3.1.

**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 NOTE 2 of 5.1.

#### **7.4 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 distilled or demineralized water, 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 1 000 ml.

#### **7.5 Determination of halogen acid content**

##### **7.5.1 Blank test**

Prior to conducting a test on a test specimen, a blank test shall be carried out using the test procedure given in 7.3.2 but without a test specimen in the combustion boat.

After cooling to ambient temperature, 200 ml of the solution shall be measured into a conical flask using a suitable pipette, and the following reagents introduced successively:

- a) approximately 4 ml of concentrated nitric acid;
- b) 20 ml of 0,1 M silver nitrate, measured with a pipette;
- c) approximately 3 ml of either nitrobenzene, toluene or iso-amyl alcohol.

The contents shall be well shaken to achieve conglomeration and coating of the silver halide that is formed by the reaction.

1 ml of a 40 % (weight/volume) aqueous solution of ferric ammonium sulphate containing a few drops of 6 M nitric acid shall then be added and the whole mixed together. The solution shall then be titrated with 0,1 M ammonium thiocyanate solution using a burette and the whole shaken or stirred vigorously. The end-point shall be the red end-point for the titration.

The volume, *B*, of 0,1 M ammonium thiocyanate solution shall be recorded.

**NOTE** It is recommended that the blank test be carried out prior to each testing campaign and whenever a new batch of sodium hydroxide solution, silver nitrate solution, ammonium thiocyanate solution or distilled or demineralized water is started.

##### **7.5.2 Material test**

The test procedure given in 7.3.2 shall be carried out with a test specimen in the combustion boat.

After cooling to ambient temperature, 200 ml of the solution shall be measured into a conical flask using a suitable pipette and the following reagents introduced successively:

- a) approximately 4 ml of concentrated nitric acid;
- b) 20 ml of 0,1 M silver nitrate, measured with a pipette;
- c) approximately 3 ml of either nitrobenzene, toluene or iso-amyl alcohol.

The contents shall be well shaken to achieve conglomeration and coating of the silver halide that is formed by the reaction.

1 ml of a 40 % (weight/volume) aqueous solution of ferric ammonium sulphate containing a few drops of 6 M nitric acid shall then be added and the whole mixed together. The solution shall then be titrated with 0,1 M ammonium thiocyanate solution using a burette and the whole shaken or stirred vigorously. The end-point shall be the red end-point for the titration.

The volume,  $A$ , of 0,1 M ammonium thiocyanate solution shall be recorded.

### 7.5.3 Halogen acid content calculation

The amount of halogen acid, expressed as milligrams of hydrochloric acid per gram of test specimen taken, shall be determined according to the formula:

$$C = \frac{\left( 36,5 (B - A) M \times \frac{1\,000}{200} \right)}{m}$$

where

- $A$  is the volume in ml of 0,1 M ammonium thiocyanate solution used in the test specimen determination;
- $B$  is the volume in ml of 0,1 M ammonium thiocyanate solution used in the blank test determination;
- $C$  is the amount of halogen acid expressed in milligrams of hydrochloric acid per gram of the test specimen;
- $m$  is the mass of test specimen taken in grams;
- $M$  is the molarity of ammonium thiocyanate solution;
- 36,5 is the molar mass of hydrogen chloride.

## 8 Evaluation of the test results

The halogen acid gas content of the material,  $C_m$ , shall be taken as the mean of the determination of the two test specimens.

The individual values shall not vary from the mean by more than  $\pm 10\%$  where the halogen acid gas content is  $\geq 5$  mg/g.

Record  $C = 5$  when the amount of halogen acid is less than 5 mg/g.

## 9 Performance requirement

No requirements for conformity are included in this standard.

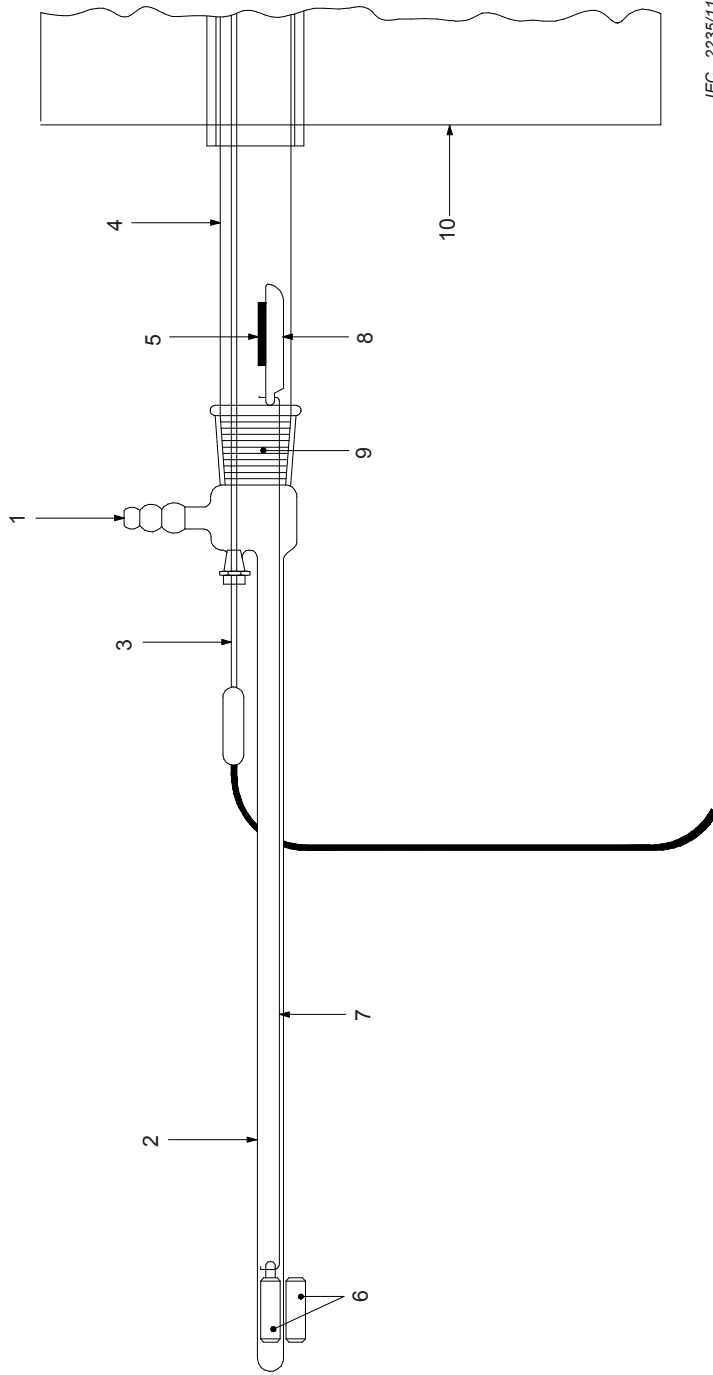
## 10 Test report

The test report shall include the following information:

- a) a full description of the material tested;
- b) the number of this standard;
- c) the halogen acid gas content of the material ( $C_m$ );
- d) the analytical method used for the determination of the halogen acid gas content (if not as given in this standard);
- e) the supplier's reference used for reagents 3) and 6) (as per 5.9) shall be reported;

- f) the type of test apparatus used (method 1, method 2 or method 3).

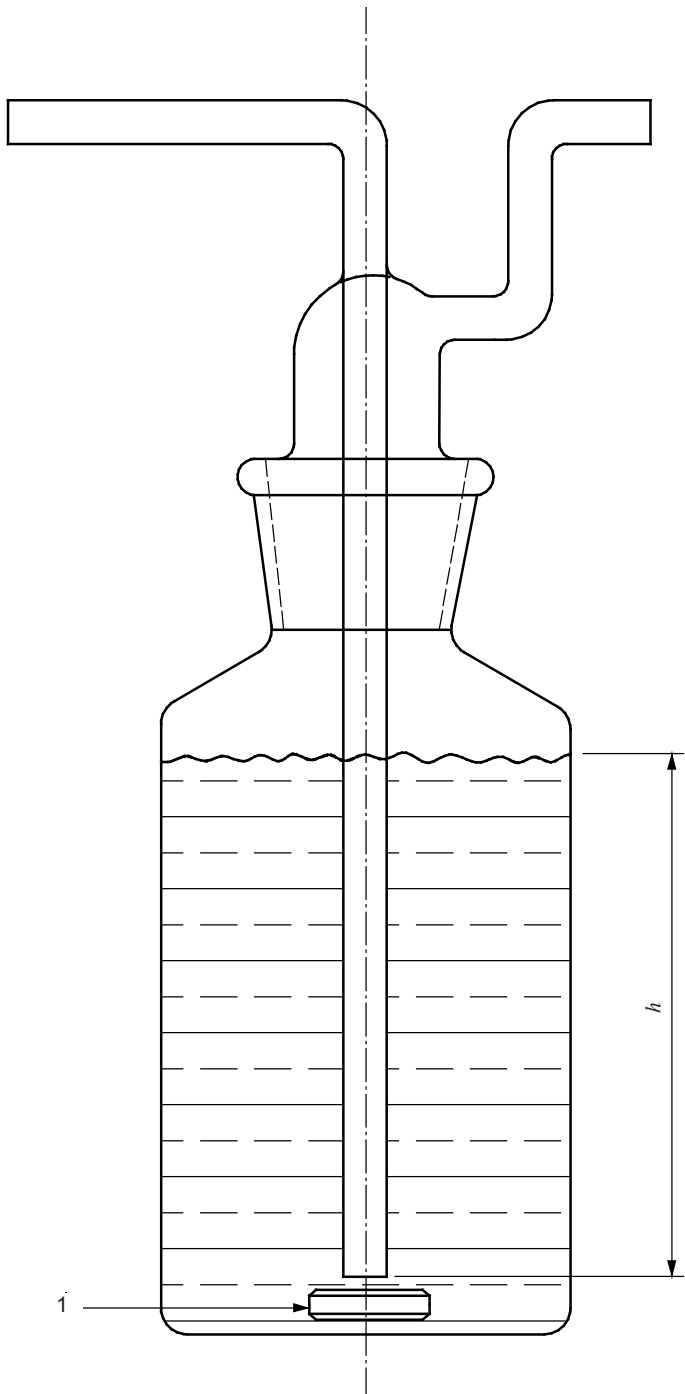




**Key**

- |   |   |    |   |
|---|---|----|---|
| 1 | Synthetic air or compressed air, filtered and dried | 6  | Bar magnets   |
| 2 | Glass tube  | 7  | Platinum wire   |
| 3 | Thermocouple  | 8  | Combustion boat   |
| 4 | Quartz glass tube                                   | 9  | Standard ground joint, size in correlation to the diameter of the quartz glass tube |
| 5 | Test specimen                                       | 10 | Furnace   |

**Figure 1 – Device for inserting combustion boat and test specimen**



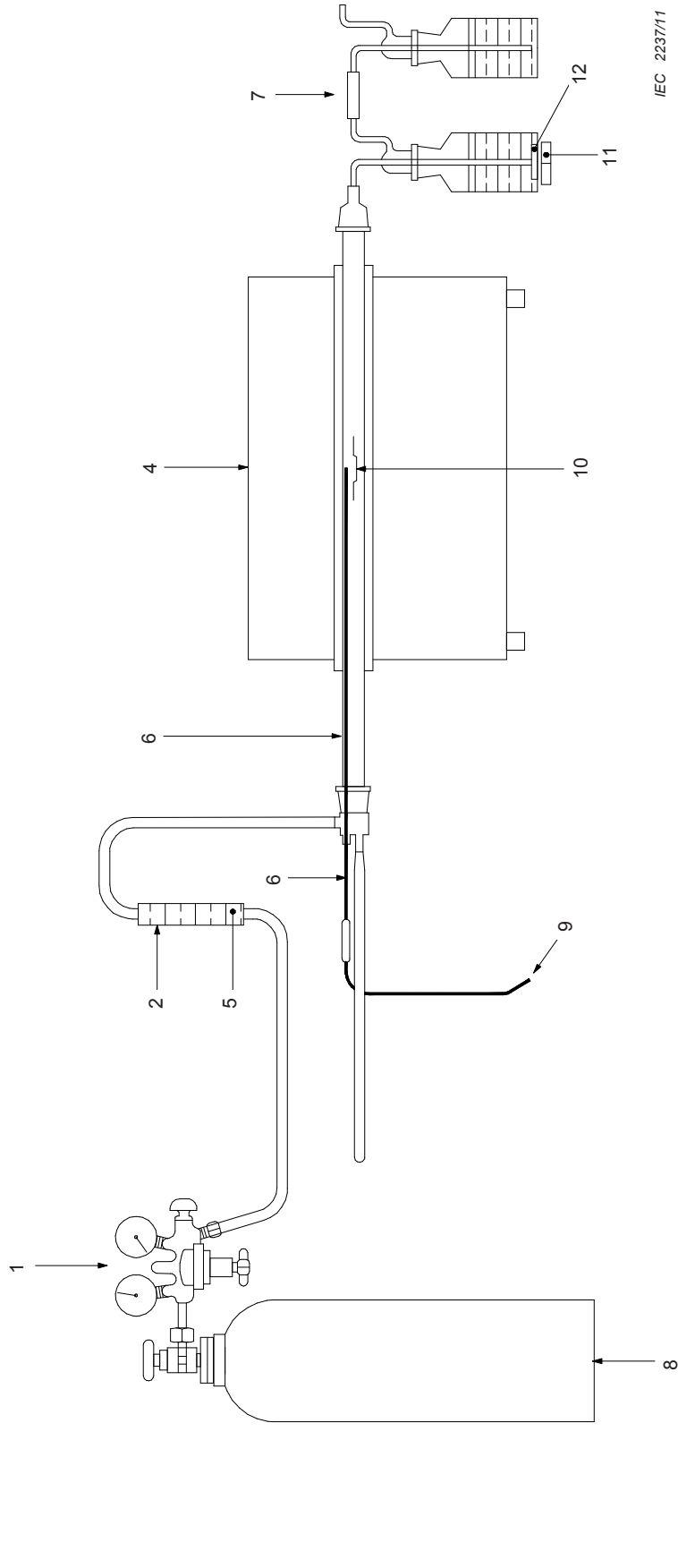
IEC 2236/11

**Key**

1 Magnetic stirring bar

*h* 100 mm to 120 mm

**Figure 2 – Example of a gas washing bottle**

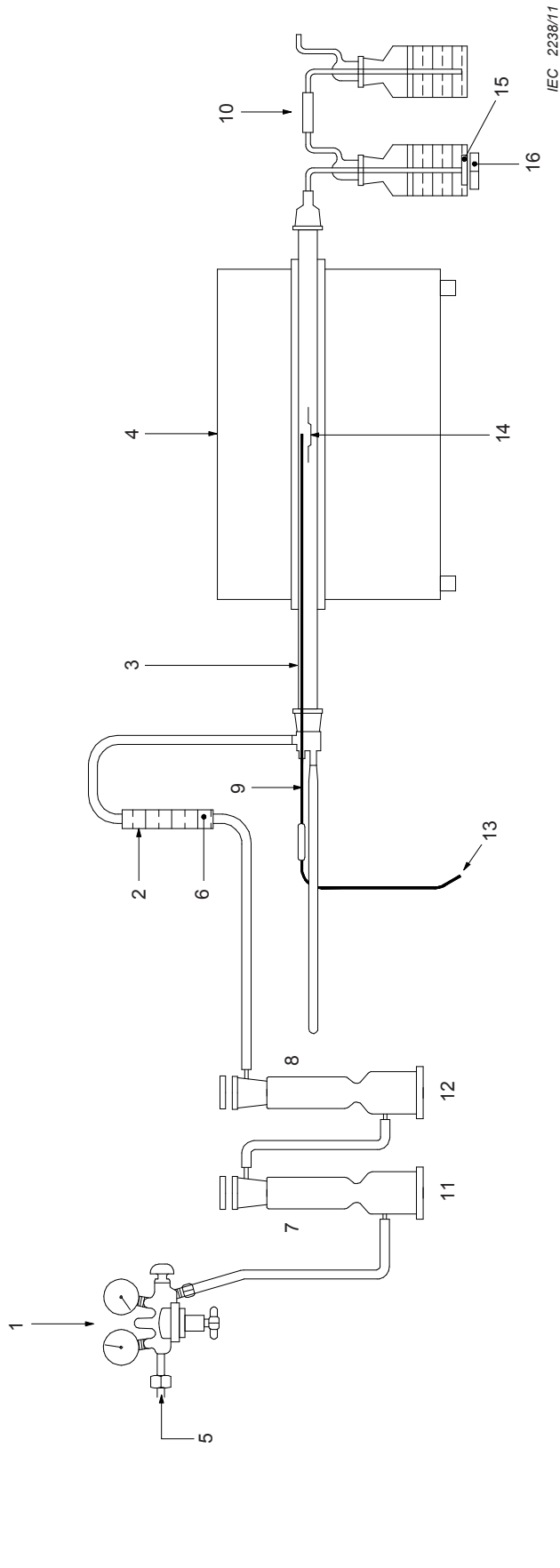


IEC 2237/11

**Key**

- |   |                         |    |   |
|---|-------------------------|----|---|
| 1 | Pressure reducing valve | 7  | Gas washing bottles   |
| 2 | Flow meter              | 8  | Synthetic air   |
| 3 | Quartz glass tube       | 9  | Device for inserting combustion boat containing test specimen |
| 4 | Furnace                 | 10 | Combustion boat containing test specimen                      |
| 5 | Needle valve            | 11 | Magnetic stirrer  |
| 6 | Thermocouple            | 12 | Magnetic stirring bar   |

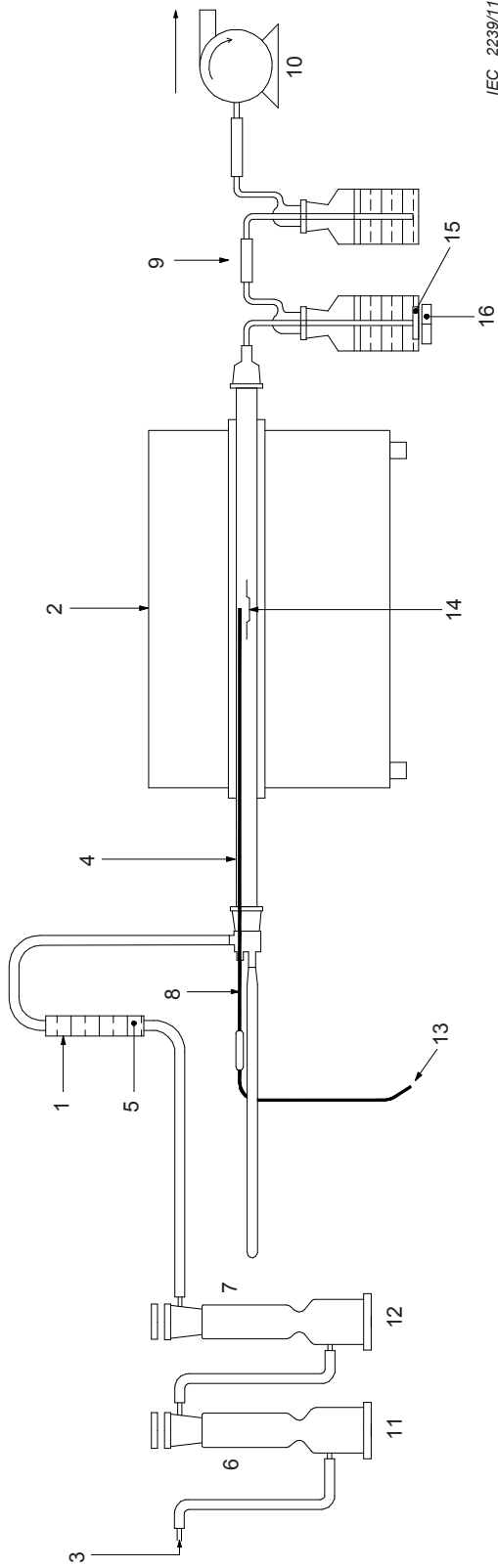
**Figure 3 – Test apparatus: method 1 – Use of synthetic or compressed air from a bottle**



**Key**

1	Pressure reducing valve	7	Activated charcoal	13	Device for inserting combustion boat containing test specimen
2	Flow meter	8	Silica gel	14	Combustion boat containing test specimen
3	Quartz glass tube	9	Thermocouple	15	Magnetic stirring bar
4	Furnace	10	Gas washing bottles	16	Magnetic stirrer
5	Compressed air	11	Air filtering		
6	Needle valve	12	Air drying		

**Figure 4 – Test apparatus: method 2 – Use of laboratory compressed air supply**



<b>Key</b>	
1	Flow meter
2	Furnace
3	Ambient air
4	Quartz glass tube
5	Needle valve
6	Activated charcoal
7	Silica gel
8	Thermocouple
9	Gas washing bottles
10	Suction pump
11	Air filtering
12	Air drying
13	Device for inserting combustion boat containing test specimen
14	Combustion boat containing test specimen
15	Magnetic stirring bar
16	Magnetic stirrer

**Figure 5 – Test apparatus: method 3 – Use of ambient air sucked by means of a suction pump**

## **Annex A** (informative)

### **Determination of the halogen acid gas content of a sample representative of a cable construction**

If agreed between the cable producer and the customer, a representative sample of all the non-metallic components of the cable construction may be used as the test specimen.

The representative sample should be prepared by the following method:

A sample of approximate length 15 mm to 25 mm of the complete cable should be cut into small pieces after all metal elements have been removed. The sample should be of sufficient length to give the required weight of test specimen according to 6.1. A sharp knife or a razor blade should be used to cut the cable components into smaller pieces complying with the requirements of 6.1.

The pieces should be well mixed and the required number of test specimens in accordance with 6.1 should be taken from the mixture.

## Bibliography

IEC 60684-2, *Flexible insulating sleeving – Part 2: Methods of test*

IEC Guide 104, *The preparation of safety publications and the use of basic safety publications and group safety publications*

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