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INTERNATIONAL STANDARD

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NORME HORIZONTALE

Determination of certain substances in electrotechnical products – Part 3-2: Screening – Total bromine in polymers and electronics by Combustion – Ion Chromatography

Détermination de certaines substances dans les produits électrotechniques – Partie 3-2: Méthodes d'essai – Brome total dans les polymères et les produits électriques par Combustion – Chromatographie d'Ionisation

IEC 62321-3-2:2013

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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FOREWORD

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International Standard IEC 62321-3-2 has been prepared by IEC technical committee 111: Environmental standardization for electrical and electronic products and systems.

It has the status of a horizontal standard in accordance with IEC Guide 108.

The first edition of IEC 62321:2008 was a 'stand alone' standard that included an introduction, an overview of test methods, a mechanical sample preparation as well as various test method clauses.

This first edition of IEC 62321-3-2 introduces a new clause in the IEC 62321 series.

Future parts in the IEC 62321 series will gradually replace the corresponding clauses in IEC 62321:2008. Until such time as all parts are published, however, IEC 62321:2008 remains valid for those clauses not yet re-published as a separate part.

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

FDIS	Report on voting
111/300/FDIS	111/310/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 parts in the IEC 62321 series can be found on the IEC website under the general title: *Determination of certain substances in electrotechnical products.*

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- withdrawn,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 widespread use of electrotechnical products has drawn increased attention to their impact on the environment. In many countries all over the world this has resulted in the adaptation of regulations affecting wastes, substances and energy use of electrotechnical products.

The use of certain substances (e.g. lead (Pb), cadmium (Cd) and polybrominated diphenyl ethers (PBDE's)) in electrotechnical products, is a source of concern in current and proposed regional legislation.

The purpose of the IEC 62321 series is therefore to provide test methods that will allow the electrotechnical industry to determine the levels of certain substances of concern in electrotechnical products on a consistent global basis.

WARNING – Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions

DETERMINATION OF CERTAIN SUBSTANCES IN ELECTROTECHNICAL PRODUCTS –

Part 3-2: Screening – Total bromine in polymers and electronics by Combustion – Ion Chromatography

1 Scope

Part 3-2 of IEC 62321 specifies the screening analysis of the total bromine (Br) in homogeneous materials found in polymers and electronics by using the analytical technique of combustion ion chromatography (C-IC).

This test method has been evaluated for ABS (acrylonitrile butadiene styrene), EMC (epoxy molding compound), and PE (polyethylene) within the concentration ranges as specified in Table 1.

The use of this method for other types of materials or concentration ranges outside those specified below has not been evaluated.

Table 1 – Tested concentration ranges for bromine by C-IC in various materials

Substance/element	Bromine		
Parameter	Unit of measure	Medium/material tested	
		ABS	EMC	PE
Concentration or concentration range tested	mg/kg	124 to 890	195 to 976	96

This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

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 60754-1:2011, *Test on gases evolved during combustion of materials from cables – Part 1: Determination of the halogen acid gas content*

IEC 62321-1, *Determination of certain substances in electrotechnical products – Part 1: Introduction and overview*[1]

IEC 62321-2, *Determination of certain substances in electrotechnical products – Part 2: Disassembly, disjointment and mechanical sample preparation*[1]

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1 To be published.

IEC 62321-3-1, *Determination of certain substances in electrotechnical products – Part 3-1: Screening –Lead, mercury, cadmium, total chromium and total bromine in electrotechnical products using X-ray fluorescence spectrometry*[2]

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

ISO 8466-1, *Water quality – Calibration and evaluation of analytical methods and estimation of performance characteristics – Part 1: Statistical evaluation of the linear calibration function*

ISO/DIS 10304-1:2006, *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, definitions and abbreviations

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC 62321-1 as well as the following, apply.

3.1.1
accuracy
closeness of agreement between a test result and an accepted reference value

Note 1 to entry: The term accuracy, when applied to a set of test results, involves a combination of random components and a common systematic error or bias component.

[ISO 5725-1:1995, definition 3.6] [1]

3.1.2
laboratory control sample
a known matrix spiked with compound(s) representative of the target analytes, used to document laboratory performance

[Based on US EPA SW-846] [2]

3.1.3
repeatability limit
value less than or equal to which the absolute difference between two test results obtained under repeatability conditions may be expected to be with a probability of 95 %

Note 1 to entry: The symbol used is *r*.

[ISO 5725-1:1994, definition 3.16]

3.1.4
reproducibility limit
value less than or equal to which the absolute difference between two test results obtained under reproducibility conditions may be expected to be with a probability of 95 %

Note 1 to entry: The symbol used is *R*.

[ISO 5725-1:1994, definition 3.20]

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2 To be published.

3.1.5
test sample
sample prepared from the laboratory sample and from which test portions will be taken

[ISO 6206:1979, definition 3.2.13] [3]

3.1.6
test portion
the quantity of material drawn from the test sample (or from the laboratory sample if both are the same) and on which the test or observation is actually carried out

[ISO 6206:1979, definition 3.2.14]

3.2 Abbreviations

ABS	Acrylonitrile butadiene styrene
CCV	Continuing calibration verification
CD	Conductivity detector
C-IC	Combustion – Ion chromatography
EMC	Epoxy molding compound
IC	Ion chromatography
IS	Internal standard
IUPAC	International Union of Pure and Applied Chemistry
KRISS	Korea Research Institute of Standards and Science
LCS	Laboratory control sample
LOD	Limit of detection
LOQ	Limit of quantification
MDL	Method detection limit
PBBs	Polybrominated biphenyls
PBDEs	Polybrominated diphenyl ethers
PE	Polyethylene
PP	Polypropylene
XRF	X-Ray fluorescence spectroscopy
US EPA	United States Environmental Protection Agency

4 Principle

4.1 Overview

The concept of 'screening' has been developed to reduce the amount of testing. Executed as a predecessor to any other test analysis, the main objective of screening is to quickly determine whether the screened part or section of a product:

– contains a certain substance at a concentration significantly higher than its value or values chosen as criterion, and therefore may be deemed unacceptable;

– contains a certain substance at a concentration significantly lower than its value or values chosen as criterion, and therefore may be deemed acceptable;

contains a certain substance at a concentration so close to the value or values chosen as criterion that when all possible errors of measurement and safety factors are considered, no conclusive decision can be made about the acceptable absence or presence of a certain

substance and, therefore, a follow-up action may be required, including further analysis using verification testing procedures.

This test method is designed specifically to screen for bromine (Br) in polymers and electronics in electrotechnical products. C-IC provides information on the total quantity of bromine present in the sample, but does not identify compounds or valence states of the bromine. Therefore, special attention shall be paid when screening for bromine, where the result will reflect only the total bromine present. The presence of brominated flame retardants PBB or PBDE shall be confirmed by a verification test procedure. When applying this method to electronics “as received”, which, by the nature of their design, are not uniform, care shall be taken in interpreting the results.

4.2 Principle of test

A sample of known weight or volume is placed into a sample boat and introduced at a controlled rate into a high-temperature combustion tube. There the sample is combusted in an oxygen-rich pyrohydrolytic environment. The gaseous by-products of the combusted sample are trapped in an absorption medium where the hydrogen bromide (HBr) formed during the combustion disassociates into its respective ion, Br^-. An aliquot of known volume of the absorbing solution is then manually or automatically injected into an ion chromatograph (IC) by means of a sample injection valve. The halide anions, including bromide, are separated into individual elution bands on the separation column of the IC. The conductivity of the eluent is reduced with an anion suppression device prior to the ion chromatograph’s conductivity detector, where the anions of interest are measured. Quantification of the bromine in the original combusted sample is achieved by calibrating the system with a series of standards containing known amounts of bromide and then analysing unknown samples under the same conditions as the standards. The combined system of pyrohydrolytic combustion followed by ion chromatographic detection is referred to as combustion-ion chromatography (C-IC).

5 Reagents and materials

WARNING – All recognized health and safety precautions shall be in effect when carrying out the operations specified in this International Standard. Failure to heed the directions contained in this International Standard, or those of the manufacturer of the devices used, may result in injury or equipment damage.

Use only reagents of recognized analytical grade. Weigh the reagents with an accuracy of ± 1 % of the nominal mass, unless stated otherwise. The reagents listed in 5 b) and 5 g) to 5 k) may be considered representative examples for the preparation of eluents (5 i)).

a) Water, complying with grade 1 as defined in ISO 3696.

b) Hydrogen peroxide, a mass fraction of 30 %, (H_2O_2).

   Hydrogen peroxide is very caustic, thus the operator shall wear goggles and gloves and shall work under a fume hood when handling this reagent. As this method uses a gas (oxygen) at a high temperature and under high pressure, precautions shall be taken by the operator.

c) Quartz wool, fine grade or other suitable medium.

d) Argon, carrier gas of minimum 99,9 %.

   Purification scrubbers to ensure the removal of containments are recommended such as moisture (molecular sieve) and hydrocarbon trap filters (activated charcoal or equivalent) are recommended.

e) Oxygen, combustion gas of minimum 99,6 %.

f) Burning aids, tungsten oxide (WO_3) or iron oxide (Fe_3O_4) etc. Minimum particle size of burning aids should be less than 50 μm.

g) Blank solution, fill a volumetric flask (e.g. 100 ml flask) with water (5 a)).

h) Calibration standard solutions

Certified calibration standards from commercial sources, or calibration standards prepared in the laboratory, containing the elements of interest at the concentrations of interest are used. Depending on the concentrations expected in the sample, use the standard solution to prepare, e.g. 5 to 10 calibration solutions distributed as evenly as possible over the expected working range.

NOTE 1 The solution is either prepared from a primary standard or calibrated by some other means.

NOTE 2 Many standard reference solutions which can be used to prepare standard solutions are commercially available.

i) Eluents

Eluents are used as a solvent in separating materials in elution. The choice of eluent depends on the chosen column and detector (seek advice from column supplier). Eluent preparation is carried out as specified in 5.10 of ISO 10304-1:2006:

1) sodium hydrogen carbonate, $NaHCO_3$;
2) sodium carbonate, Na_2CO_3;
3) sodium hydroxide, NaOH;
4) potassium hydroxide, KOH.

j) Internal standard (IS) solution (optional)

An internal standard can be used to correct for analytical errors.

The internal standard used in the absorption solution should not contain any of the sample components, and should be selected based on the condition of column and mobile phase (e.g. phosphate, citric acid, oxalic acid, methane sulfonic acid, etc.)

k) Absorption solution, used for quantifying bromine – 3 ml of H_2O_2 (5 b)) are poured into a 1 000 ml volumetric flask and water is added to the scale and mixed. This solution contains 900 mg/kg of H_2O_2.

l) Reference materials – Reference material can be used to ensure recovery rates of bromine fall within 90 % to 110 %. Certified reference material is the best one for that purpose. If certified reference material is not available, a reference material can be prepared by mixing certain amounts of bromine compounds. It can be made by mixing certain amounts of bromine compounds, diluting with cellulose or aluminium oxides to obtain a suitable concentration, and then pulverizing the mixture to homogenize.

6 Apparatus

The following apparatuses shall be used:

a) balance; analytical, with sensitivity to 0,000 1 g (0,1 mg);
b) scissors;
c) combustion system – in general, it consists of the following components (see Figure C.1):
   1) auto sampler (optional) – auto sampler is capable of accurately delivering 1 mg to 100 mg of sample into the sample boat. The auto sampler may be used as long as the accuracy and performance of the method are not degraded;
   2) sample boat – boat is made of quartz, nickel, ceramics, platinum or stainless steel;
   3) sample introduction system – the system provides a sampling port for the introduction the sample into the sample boat and is connected to the inlet of the pyrohydrolytic combustion tube. The system is swept by a humidified inert carrier gas and shall be capable of allowing the quantitative delivery of the material to be analyzed into the pyrohydrolytic oxidation zone at a controlled and repeatable rate;
   4) electric furnace – it can be heated 900 °C to 1 100 °C and have the quartz tube installed inside of the device and connected to the equipment for injecting sample. Therefore, it is designed so that the combustion gas of the sample can be discharged without loss;

5) pyrohydrolytic combustion tube – pyrohydrolytic combustion tube is made of quartz and constructed such that when the sample is combusted in the presence of humidified oxygen, the by-products of combustion are swept into the humidified pyrohydrolytic combustion zone. The inlet end shall allow for the stepwise introduction and advancement of a sample boat into the heated zone and shall have a side arm for the introduction of the humidified carrier gas and oxygen. The pyrohydrolytic combustion tube must be of ample volume, and have a heated zone with quartz wool or other suitable medium providing sufficient surface area so that the complete pyrohydrolytic combustion of the sample is ensured. If the sample contains bromine of high concentration, a trap column should be installed between the absorption tube and the combustion tube.

6) water supply device – this device is capable of delivering grade 1 water (5 a) to the combustion tube at a controlled rate sufficient to provide a pyrohydrolytic environment;

7) absorption tube – a glass pipe size is capable of maintaining about one-half of the total volume by putting 10 ml to 20 ml of the absorption solution. This has the configuration that the discharge gas pipe of the heating furnace is submerged in the absorption solution to absorb the discharged gas. Further, it has the configuration that the absorption solution of ion chromatograph can be injected through the connecting device. For preventing contamination from other samples, the absorption tube should be washed after sample analysis.

d) ion chromatographic system – in general, it consists of the following components (see Figure C.2):

1) eluent reservoir;

2) IC pump;

3) sample injection system – incorporating a sample loop of appropriate volume (e.g. 0,02 ml) or auto sampler device;

4) precolumn or guard column;

5) separation column;

6) suppressor;

7) conductivity detector (CD);

8) recording device, e.g. computer, integrator.

7 Sampling

Sampling shall be carried out as described in IEC 62321-2. It should be done randomly and the collected segments should represent the entire sample.

a) Solid sample

The sample shall be cut into small pieces (approximately less than 3 mm × 3 mm) using scissors (6 b)).

b) Liquid sample

For sampling of liquid sample, sampling should be performed after rinsing the inside of the pipette a few times with the sample liquid.

8 Procedure

8.1 Combustion

General combustion procedures by using electric furnace are described in Clause 7 (Test procedure) of IEC 60754-1:2011.

a) After a sample boat is heated sufficiently in the electric furnace to remove the contaminants, 1 mg to 100 mg of samples are weighed with precision of 0,1 mg and loaded into the sample boat. If samples are difficult to combust (e.g. flux, solder paste),

burning aids (e.g. WO_3) have to be used. Generally a 5 to 1 ratio of burning aids to sample is sufficient. If any burning aid is being used apply approximately 100 mg of it in a thin layer over the surface of the sample boat, evenly spread the weighed sample on it, and then cover the sample with approximately 300 mg of the burning aid.

b) It is then heated in the combustion furnace for 10 min to 20 min together with argon, oxygen and the water by using the sample injection device located at the center of the quartz tube of the combustion furnace. An example of combustion conditions is described in Table F.1. If the combustion boat shows evidence of soot generation or unburned sample particles, the combustion shall be judged to be insufficient and the procedure shall be repeated. The contaminated area shall be cleaned thoroughly before repeating the procedure.

c) Upon completion of combustion operations, wash the tubing at the combustion gas discharge outlet, and pour all washing solutions into the absorbing bottle for measuring.

d) For the blank test, perform a similar operation without inserting the sample or the combustion boat, and use this absorption solution obtained as the blank solution.

NOTE If the combustion furnace and IC are connected and operated automatically, the absorption solution absorbing the combustibles can be injected into the IC.

8.2 IC analysis

The general rules on ion chromatographic analysis as set out in ISO 10304-1 shall be followed:

a) set up the IC according to the instrument manufacturer's instructions. Typical operating conditions for IC are shown in Table G.1;

b) run the eluent and wait for a stable baseline;

c) perform the calibration as described in 8.5. Measure the samples, calibration (5 h)) and blank solution (5 g)) as described in 8.5.

Operating conditions should be selected and stabilized according to the device manufacturer.

8.3 Blank test

Blank test is performed by quantifying the blank solution (5 g)) which is prepared by following exactly the same procedure described above but without actual sample. A blank solution (5 g)) which does not contain bromine (lower than 0,05 mg/l) can be used as a method blank sample.

8.4 Cleaning and recalibration

Clean any coke or soot as per the manufacturer's instructions. After any cleaning or adjustment, assemble the apparatus and check for leaks. Run a check standard to determine if the instrument needs to be recalibrated.

8.5 Calibration

A calibration curve shall be developed for quantitative analysis. The calibration curve is prepared by using a standard solution of bromide.

When the analytical system is first evaluated and at intervals afterwards, establish a calibration function (e.g. as specified in ISO 8466-1) for the measurement. An example is shown in Table G.2.

The following calibration solutions are prepared from the stock solution of the bromine (1 000 mg/l). The volumes indicated in Table G.2 are placed in a 1 000 ml volumetric flask with a pipette and filled with water (5 a)) up to the mark and 0,5 ml to 8 ml of 1 000 mg/l bromine standard solution are added to the mark and mixed. This solution contains 0,5 mg/l to 8,0 mg/l of bromine:

a) prepare the calibration standard solutions (5 h));