IEC 62321-3-2

Edition 2.0 2020-06
REDLINE VERSION

INTERNATIONAL STANDARD

colour inside

HORIZONTAL STANDARD

Determination of certain substances in electrotechnical products – Part 3-2: Screening – Fluorine, chlorine and ~~total~~ bromine in polymers and electronics by combustion-ion chromatography (C-IC)

IEC 62321-3-2:2020-06 RLV(en)

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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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.

This second edition cancels and replaces the first edition published in 2013. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

a) In the previous edition, a screening test method for bromine (Br) content only was provided. In this edition, a screening test method by C-IC for fluorine (F), chlorine (Cl) and bromine (Br) has been added to the normative part of the document.
b) A screening test method by C-IC for iodine (I) has been added in Annex D (informative).

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

FDIS	Report on voting
111/573/FDIS	111/577/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.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts in the IEC 62321 series, published under the general title *Determination of certain substances in electrotechnical products* 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- withdrawn,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.

IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

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), polybrominated diphenyl ethers (PBDEs) and phthalates) 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.

The first edition of IEC 62321-3-2 (2013) was published to address screening for total bromine.

This document (revised edition of IEC 62321-3-2) describes the test methods to quantify halogen (fluorine, chlorine and bromine) in polymers and electronics by C-IC in the normative section and to quantify iodine (I) in an informative Annex D.

In addition, information on oxygen bomb combustion-ion chromatography and oxygen flask-ion chromatography is provided in Annex A (informative) and Annex B (informative).

WARNING – Persons using this document should be familiar with normal laboratory practice. This document 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 – Fluorine, chlorine and ~~total~~ bromine in polymers and electronics by combustion-ion chromatography (C-IC)

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 part of IEC 62321 specifies the screening analysis of fluorine, chlorine and bromine in polymers and electronics using combustion-ion chromatography (C-IC). A C-IC screening analysis procedure for iodine can be found in Annex D.

This test method has been evaluated for ABS (acrylonitrile butadiene styrene), EMC (epoxy moulding compound), PE (polyethylene) and PC (polycarbonate) within the concentration ranges as specified in Table 1, Table 2 and Table 3. (Detailed results are shown in Table E.1 to Table E.6, and in Annex F (Table F.1 and Table F.2).

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 fluorine by C-IC in PC

Substance/element	Fluorine	
Polymer	Unit of measure mg/kg	PC
Concentration or concentration range tested		575

Table 2 – Tested concentration ranges for chlorine by C-IC in PE

Substance/element	Chlorine	
Polymer	Unit of measure mg/kg	PE
Concentration or concentration range tested		102,2

Table 3 – Tested concentration ranges for bromine by C-IC in various materials

Substance/element	Bromine		
~~Parameter~~	Unit of measure mg/kg	~~Medium/material tested~~	
Polymer		ABS	EMC	PE
Concentration or concentration range tested		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.~~

This horizontal standard is primarily intended for use by technical committees in the preparation of standards in accordance with the principles laid down in IEC Guide 108.

One of the responsibilities of a technical committee is, wherever applicable, to make use of horizontal standards in the preparation of its publications. The contents of this horizontal standard will not apply unless specifically referred to or included in the relevant publications.

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.

~~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~~ disjunction and mechanical sample preparation*~~[1]~~

~~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~~2007, *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 abbreviated terms

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 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

~~1 To be published.~~

~~2 To be published.~~

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.

[SOURCE: ISO 5725-1:~~1995~~1994, 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]~~

3.1.2
precision
closeness of agreement between independent test results obtained under stipulated conditions

[SOURCE: ISO 5725-1:1994, 3.12, modified – The notes have been deleted.]

3.1.3
repeatability
precision under repeatability conditions

[SOURCE: ISO 5725-1:1994, 3.13]

3.1.4
repeatability limit
r
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*.~~

[SOURCE: ISO 5725-1:1994, 3.16]

3.1.5
reproducibility
precision under reproducibility conditions

[SOURCE: ISO 5725-1:1994, 3.17]

3.1.6
reproducibility limit
R
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*.~~

[SOURCE: ISO 5725-1:1994, 3.20]

3.1.7
screening
analytical procedure to determine the presence or absence of substances in the representative part or section of a product, relative to the value or values chosen as the criterion for presence, absence or further testing

Note 1 to entry: If the screening method produces values that are not conclusive, then additional analysis or other follow up actions may be necessary to make a final presence/absence decision

[SOURCE: IEC 62321-1:2013, 3.1.10]

3.1.8
test sample
sample prepared from the laboratory ~~sample~~ and from which test portions will be taken

[SOURCE: ISO 6206:1979, 3.2.13] ~~[3]~~

3.1.9
test portion
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

[SOURCE: ISO 6206:1979, 3.2.14]

3.2 Abbreviated terms

ABS	acrylonitrile butadiene styrene
CCV	continuing calibration verification
CD	conductivity detector
C-IC	combustion-ion chromatography
CRM	certified reference material
EMC	epoxy moulding compound
IC	ion chromatography
ICV	initial calibration verification
IS	internal standard
IUPAC	International Union of Pure and Applied Chemistry
KRISS	Korea Research Institute of Standards and Science
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LOD	limit of detection
LOQ	Limit of quantification
MDL	method detection limit
~~PBBs~~	~~Polybrominated biphenyls~~
~~PBDEs~~	~~Polybrominated diphenyl ethers~~
PC	polycarbonate
PE	polyethylene
PP	polypropylene
~~XRF~~	~~X-Ray fluorescence spectroscopy~~
SOP	standard operation procedure
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~~ halide (HF, HCl, HBr) formed during the combustion dissociates into its ~~respective ion~~ specific anion (F^-, Cl^-, and Br^-) and cation (H_30^+).

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 fluoride, chloride and 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~~ halogen in the original combusted sample is achieved by calibrating the system with a series of standards containing known amounts of fluoride, bromide and chloride 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 document. Failure to heed the directions contained in this document, 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 Clause 5 b) and g) to k) may be considered representative examples for the preparation of eluents (Clause 5 i)). All reagents used shall not contain an amount of halides above the limit of detection (LOD).

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