

INTERNATIONAL STANDARD

REDLINE VERSION

HORIZONTAL STANDARD

**Determination of certain substances in electrotechnical products -
Part 3-1: Screening - Lead, mercury, cadmium, total chromium, total bromine,
total phosphorus, total chlorine, total tin and total antimony content by X-ray
fluorescence spectrometry**

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

**Determination of certain substances in electrotechnical products -
Part 3-1: Screening - Lead, mercury, cadmium, total chromium, total
bromine, total phosphorus, total chlorine, total tin and total antimony
content by X-ray fluorescence spectrometry**

FOREWORD

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This redline version of the official IEC Standard allows the user to identify the changes made to the previous edition IEC 62321-3-1:2013. A vertical bar appears in the margin wherever a change has been made. Additions are in green text, deletions are in strikethrough red text.

IEC 62321-3-1 has been prepared by IEC technical committee 111: Environmental standardization for electrical and electronic products and systems. It is an International Standard.

This second edition cancels and replaces the first edition published in 2013 and the first edition of IEC 62321 published in 2008. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous editions of IEC 62321-3-1:2013 and IEC 62321:2008:

- a) This second edition of IEC 62321-3-1 includes the analysis of additional elements as indicators for additional substances. The selection is based on IEC TR 62936:2016. There are also comments about using the same methodology for screening for content of critical raw materials (CRMs).

This document has been given the status of a horizontal document in accordance with the ISO/IEC Directives, Part 1.

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

Draft	Report on voting
111/871/FDIS	111/887/RVD

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

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 language used for the development of this International Standard is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

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 webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn, or
- revised.

INTRODUCTION

The widespread use of electrotechnical products has drawn increased attention to their impact on the environment. In many countries 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 (PBDEs)) in electrotechnical products, is a source of concern in current and proposed regional legislation. With the actual revision the following elements are added: phosphorus (P), assuming the source of P is related to TCEP, Trixylyl-phosphate, chlorine (Cl), assuming the source of Cl is related to SCCP, TCEP, TBTC, tin (Sn), assuming the source of Sn is related to restricted organo-tin compounds, antimony (Sb), assuming the source of Sb is related to Pyrochlore, antimony lead yellow.

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.~~

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.

The first edition of IEC 62321-3-1 was a partial replacement of IEC 62321:2008, forming a structural revision and generally replacing Clauses 6 and Annex D.

1 Scope

This part of IEC 62321 describes the screening analysis of ~~five~~ substances, specifically lead (Pb), mercury (Hg), cadmium (Cd), total chromium (Cr), total bromine (Br), total phosphorus (P), assuming the source of P is related to TCEP (CAS 115-96-8), Trixylyl-phosphate (CAS 25155-23-1), total chlorine (Cl), assuming the source of Cl is related to SCCP (CAS 85535-84-8), TCEP (CAS 115-96-8), TBTC (CAS 1461-22-9), total tin (Sn), assuming the source of Sn is related to restricted organo-tin compounds, total antimony (Sb), assuming the source of Sb is related to Pyrochlore, and antimony lead yellow (CAS 8012-00-8) in uniform materials found in electrotechnical products, using the analytical technique of X-ray fluorescence (XRF) spectrometry.

The same methodology can also be used for screening of substances discussed as critical raw materials in various countries (for example currently discussed in the EU: antimony (Sb), baryte, bismuth (Bi), cobalt (Co), fluorspar, gallium (Ga), germanium (Ge), hafnium (Hf), indium (In), magnesium (Mg), niobium (Nb), phosphorus (P), scandium (Sc), tantalum (Ta), tungsten (W), vanadium (V), platinum group metals, heavy rare earth elements, light rare earth elements).

NOTE From EU information on critical raw materials [1]¹ raw materials are crucial to Europe's economy. They form a strong industrial base, producing a broad range of goods and applications used in everyday life and modern technologies. Reliable and unhindered access to certain raw materials is a growing concern within the EU and across the globe. To address this challenge, the European Commission has created a list of critical raw materials (CRMs) for the EU, which is subject to a regular review and update. CRMs combine raw materials of high importance to the EU economy and of high risk associated with their supply.

The method is applicable to ~~polymers~~ plastics, metals and ceramic materials. The test method ~~may~~ can be applied to raw materials, individual materials taken from products and "homogenized" mixtures of more than one material. Screening of a sample is performed using any type of XRF spectrometer, provided it has the performance characteristics specified in this test method. Not all types of XRF spectrometers are suitable for all sizes and shapes of sample. ~~Care should be taken to select~~ The appropriate spectrometer design will be selected with care for the task concerned.

The performance of this test method has been tested for the following substances in various media and within the concentration ranges as specified in Table 1 to Table 5. During an IIS (international interlaboratory study) the feasibility of the test method to use for the added elements was tested. The results are listed in Table 6 to Table 10.

Table 1 – Tested concentration ranges for lead in materials

Substance/ element	Lead									
	Parameter	Unit of measure	Medium/material tested							
ABS ^a			PE ^b	Low- alloy steel	Al, Al-Si alloy	Lead- free solder	Ground PWB ^c	Crystal glass	PVC ^d	
Concentration or concentration range tested	mg/kg	15,7 to 954	14 to 108	30 ^e	190 to 930	174	22 000 to 23 000	240 000	390 to 665	380 to 640
^a Acrylonitrile butadiene styrene. ^b Polyethylene. ^c Printed wiring board. ^d Polyvinyl chloride. ^e This lead concentration was not detectable by instruments participating in tests.										

¹ Numbers in square brackets refer to the Bibliography.

Table 2 – Tested concentration ranges for mercury in materials

Substance/element	Mercury		
Parameter	Unit of measure	Medium/material tested	
		ABS ^a	PE ^b
Concentration or concentration range tested	mg/kg	100 to 942	4 to 25
^a Acrylonitrile butadiene styrene. ^b Polyethylene.			

Table 3 – Tested concentration ranges for cadmium in materials

Substance/element	Cadmium			
Parameter	Unit of measure	Medium/material tested		
		Lead-free solder	ABS ^a	PE ^b
Concentration or concentration range tested	mg/kg	3 ^c	10 to 183	19,6 to 141
^a Acrylonitrile butadiene styrene. ^b Polyethylene. ^c This cadmium concentration was not detectable by instruments participating in tests.				

Table 4 – Tested concentration ranges for total chromium in materials

Substance/element	Chromium					
Parameter	Unit of measure	Medium/material tested				
		ABS ^a	PE ^b	Low-alloy steel	Al, Al-Si alloy	Glass
Concentration or concentration range tested	mg/kg	16 to 944	16 to 115	240	130 to 1 100	94
^a Acrylonitrile butadiene styrene. ^b Polyethylene.						

Table 5 – Tested concentration ranges for total bromine in materials

Substance/element	Bromine			
Parameter	Unit of measure	Medium/material tested		
		HIPS ^c , ABS ^a	PC/ABS ^d	PE ^b
Concentration or concentration range tested	mg/kg	25 to 118 400	800 to 2 400	96 to 808
^a Acrylonitrile butadiene styrene. ^b Polyethylene. ^c High impact polystyrene. ^d Polycarbonate and ABS blend.				

Table 6 – Tested concentration ranges for total phosphorus in materials

Substance/element	Phosphorus	
Parameter	Unit of measure	Medium/material tested
		plastics
Concentration or concentration range tested	mg/kg	90 to 8 300

Table 7 – Tested concentration ranges for total chlorine in materials

Substance/element	Chlorine	
Parameter	Unit of measure	Medium/material tested
		plastics
Concentration or concentration range tested	mg/kg	100 to 380

Table 8 – Tested concentration ranges for total tin in materials

Substance/element	Tin	
Parameter	Unit of measure	Medium/material tested
		plastics
Concentration or concentration range tested	mg/kg	30 to 110

Table 9 – Tested concentration ranges for total antimony in materials

Substance/element	Antimony	
Parameter	Unit of measure	Medium/material tested
		plastics
Concentration or concentration range tested	mg/kg	190 to 380

These substances in similar media outside of the specified concentration ranges ~~may~~ can be analysed according to this test method; however, the performance has not been established for this document.

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.

This document is a basic environment horizontal publication focusing on test methods and is primarily intended for use by committees in the preparation of publications within the area of environment in accordance with the principles laid down in IEC Guide 123. Wherever applicable, it is the responsibility of committees to make use of environment basic publications in the preparation of their environment group and product publications. Committees can apply this document directly to products when they do not develop a product publication in the area of environment.

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 62321-1, *Determination of certain substances in electrotechnical products - Part 1: Introduction and overview*²

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

~~IEC/ISO Guide 98-1, *Uncertainty of measurement — Part 1: Introduction to the expression of uncertainty in measurement*~~

ISO/IEC Guide 98-1, *Guide to the expression of uncertainty in measurement - Part 1: Introduction*

Bibliography

- [1] EU information on Critical Raw Materials: https://ec.europa.eu/growth/sectors/raw-materials/specific-interest/critical_en
- [2] BERTIN, E.P., *Principles and practices of X-ray spectrometric analysis*, 2nd Edition Plenum Press, N.Y.
- [3] BUHRKE, V.E., JENKINS, R., SMITH, DK., *A practical guide for the preparation of specimens for X-ray fluorescence and X-ray diffraction analysis*, Wiley-VCHR
- [4] VAN GRIEKEN, R. and MARKOWICZ, A. *Handbook of X ray spectrometry*, 2nd Edition, Marcel Dekker Inc.
- [5] ASTM C 982-03, *Guide for selecting components for energy-dispersive X-ray fluorescence systems* (withdrawn in 2008)
- [6] ASTM C 1118-07, *Guide for selecting components for wavelength-dispersive X-ray fluorescence systems* (withdrawn in 2011)
- [7] ASTM E 1172-87, *Standard practice for describing and specifying a wavelength-dispersive X-ray spectrometer*
- [8] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results - Basic method for the determination of repeatability and reproducibility of a standard measurement method*
- [9] International Union of Pure and Applied Chemistry, *Harmonized guidelines for single laboratory validation of methods of analysis* (IUPAC Technical Report), Pure Appl. Chem., 2002, vol. 74, no. 5, p. 835–855
- [10] International Union of Pure and Applied Chemistry, *Nomenclature in Evaluation of analytical methods including detection and quantification limits*, Pure Appl. Chem., 1995, vol. 67, no. 10, p.1699-1723
- [11] BECKER, D. et al., *Use of NIST standard reference materials for decisions on performance of analytical chemical methods and laboratories*, National Institute of Standards and Technology (NIST) Special Publication 829, 1992
- [12] GB/T 33352-2016, *General rules of screening application of restricted substances in electrical and electronic products - X-Ray fluorescence spectrometry*
- [13] 111/812/INF, Report of the International Interlaboratory Study (IIS) on the "IEC 62321-3-1: Screening - Lead, mercury, cadmium, total chromium, total bromine, total phosphorus, total chlorine, total tin and total antimony content by X-ray fluorescence spectrometry"

- [14] ASTM E 1361-02, *Guide for correction of inter-element effects in X-ray spectrometric analysis*
 - [15] ASTM E 1621-05, *Standard guide for X-ray emission spectrometric analysis*
 - [16] ASTM E 1622-94, *Standard practice for correction of spectral line overlap in wavelength-dispersive X-ray spectrometry*
(withdrawn in 2006)
 - [17] ASTM F 2617-08, *Standard test method for identification and quantification of chromium, bromine, cadmium, mercury and lead in polymeric material using energy dispersive X-ray spectrometry*
-