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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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FOREWORD

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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.

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 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 plastics, metals and ceramic materials. The test method 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. 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]	Polyolefin
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 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*

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

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

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 and IEC 62321-2 apply.

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

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

3.2 Abbreviated terms

CRM	Certified reference material
CRMs	Critical raw materials
HBCDD	Hexabromocyclododecane
MCCP	Medium chain chlorinated paraffins
PBB	Polybrominated biphenyl
PBDE	Polybrominated diphenyl ether
SCCP	Short chain chlorinated paraffins
TBBPA	Tetrabromobisphenol A
TBTC	Tributyltin chloride
TCEP	Tris(2-chloroethyl) phosphate

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 can be deemed unacceptable;

– contains a certain substance at a concentration significantly lower than its value or values chosen as criterion, and therefore can 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 can be required, including further analysis using verification testing procedures.

For the screening analysis of critical raw materials only the analysis results are important, there is no interpretation with regards to a maximum threshold value required.

This test method is designed specifically to screen for lead, mercury, cadmium, chromium, bromine, phosphorus, chlorine, tin and antimony (Pb, Hg, Cd, Cr, Br, P, Cl, Sn, Sb) plus elements required for screening for content of critical raw materials in uniform materials, which occur in most electrotechnical products. Under typical circumstances, XRF spectrometry provides information on the total quantity of each element present in the sample but does not identify compounds or valence states of the elements. Therefore, special attention shall be paid when screening for chromium, bromine, phosphorus, chlorine, tin and antimony, where the result will reflect only the total chromium, total bromine, total phosphorus, total chlorine, total tin and total antimony present. The presence of Cr(VI) or the brominated flame retardants PBB or PBDE or TBBPA or HBCDD, or TCEP, Trixylyl-phosphate, red phosphorus, SCCP or MCCP, TBTC, restricted organo-tin compounds, Pyrochlore, or antimony lead yellow shall be confirmed by a verification test procedure that identify compounds or valence states of the elements. 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. Similarly, the analysis of Cr in conversion coatings can be difficult due to the presence of Cr in substrate material. It also can be difficult because of insufficient sensitivity for Cr in typically very thin (typically 200 nm to 600 nm) conversion coating layers.

Screening analysis can be carried out by one of two means:

– non-destructively - by directly analysing the sample "as received";

– destructively - by applying one or more sample preparation steps prior to analysis.

In the latter case, the user shall apply the procedure for sample preparation as described in IEC 62321-2. This test method will guide the user in choosing the proper approach to sample presentation.

4.2 Principle of test

The representative specimen of the object tested is placed in the measuring chamber or over the measuring aperture of the X-ray fluorescence spectrometer. Alternatively, a measuring window or aperture of a handheld, portable XRF analyser is placed flush against the surface of the object tested. The analyser illuminates the specimen for a preselected measurement time with a beam of X-rays which in turn excite characteristic X-rays of elements in the specimen. The intensities of these characteristic X-rays are measured and converted to mass fractions or concentrations of the elements in the tested sample using a calibration implemented in the analyser.

The fundamentals of XRF spectrometry, as well as practical aspects of sampling for XRF, are covered in detail in [2], [3], and [4].

4.3 Explanatory comments

To achieve its purpose, this test method shall provide rapid, unambiguous identification of the elements of interest. The test method shall provide at least a level of accuracy that is sometimes described as semi-quantitative, i.e. the relative uncertainty of a result is typically 30 % or better at a defined level of confidence of 68 %. Some users can tolerate higher relative uncertainty, depending on their needs. This level of performance allows the user to sort materials for additional testing. The overall goal is to obtain information for risk management purposes.

This test method is designed to allow XRF spectrometers of all designs, complexity and capability to contribute screening analyses. However, the capabilities of different XRF spectrometers cover such a wide range that some will be relatively inadequate in their selectivity and sensitivity while others will be more than adequate. Some spectrometers will allow easy measurement of a wide range of sample shapes and sizes, while others, especially research-grade WDXRF units, will be very inflexible in terms of test portions.

NOTE One technical parameter for ED-XRF instruments can be for example the detector resolution. A resolution of better than 250 eV (at Mn K_α) has been found suitable.

Given the above level of required performance and the wide variety of XRF spectrometers capable of contributing useful measurements, the requirements for the specification of procedures must be defined carefully. As guidance the information listed in Table A.2 can be used.

This test method is based on the concept of a performance-based measurement system. Apparatus, sample preparation and calibration are specified in this document in relatively general terms. It is the responsibility of the user to document all procedures developed in the laboratory that uses the test method. The user shall establish a written procedure for all cases denoted in this method by the term "work instructions".

The user of this test method shall document all relevant spectrometer and method performance parameters.

For additional practical aspects of screening by X-ray fluorescence spectrometry (XRF) and interpretation of the results please also refer to Annex A. For practical examples of screening with XRF refer to Annex B.

WARNING 1 Persons using the XRF test method shall be trained in the use of XRF spectrometers and the related sampling requirements.

WARNING 2 X-rays are hazardous to humans. Care shall be taken to operate the equipment in accordance with both the safety instructions provided by the manufacturer and the applicable local health and occupational safety regulations.

5 Apparatus, equipment and materials

5.1 XRF spectrometer

An XRF spectrometer consists of an X-ray excitation source, a means of reproducible sample presentation, an X-ray detector, a data processor and a control system [5], [6] and [7]:

a) source of X-ray excitation - X-ray tube or radio-isotope sources are commonly used;

b) X-ray detector (detection subsystem) - device used to convert the energy of an X-ray photon to a corresponding electric pulse of amplitude proportional to the photon energy.

5.2 Materials and tools

All materials used in the preparation of samples for XRF measurements shall be shown to be free of contamination, specifically by the analytes of this test method. This means that all grinding materials, solvents, fluxes, etc. shall not contain detectable quantities of Pb, Hg, Cd, Cr, Br, P, Cl, Sn, Sb, or any other critical raw material.

Tools used in the handling of samples shall be chosen to minimize contamination by the analytes of this test method as well as by any other elements. Any procedures used to clean the tools shall not introduce contaminants.

6 Reagents

Reagents, if any, shall be of recognized analytical grade and shall not contain detectable quantities of Pb, Hg, Cd, Cr, Br, P, Cl, Sn, Sb or any critical raw materials.

7 Sampling

7.1 General

It is the responsibility of the user of this test method to define the test sample using documented work instructions. The user can choose to define the test sample in a number of ways, either via a non-destructive approach in which the portion to be measured is defined by the viewing area of the spectrometer, or by a destructive approach in which the portion to be measured is removed from the larger body of material and either measured as is, or destroyed and prepared using a defined procedure.

7.2 Non-destructive approach

The user of this test method shall:

a) establish the area viewed by the spectrometer and place the test sample within that area, taking care to ascertain that no fluorescent X-rays will be detected from materials other than the defined test specimen. Usually, the area viewed by the spectrometer is a section of a plane delineated by the shape and boundary of the measuring window of the instrument. The area of the test sample viewed by the spectrometer shall be flat. Any deviation from the flat area requirement shall be documented;

b) make sure that a repeatable measurement geometry with a repeatable distance between the spectrometer and the test portion is established;

c) document the steps taken to disassemble a larger object to obtain a test portion.

7.3 Destructive approach

The following points shall be taken into account in the destructive approach:

a) the user shall create and follow a documented work instruction for the means of destruction applied to obtain the test portion, as this information is critical for correct interpretation of the measurement results;

b) a procedure that results in a powder shall produce a material with a known or controlled particle size. In cases where the particles have different chemical, phase or mineralogical compositions, it is critical to reduce their size sufficiently to minimize differential absorption effects;