IEC 62321-3-1

Edition 1.0 2013-06

INTERNATIONAL STANDARD

NORME INTERNATIONALE

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HORIZONTAL STANDARD

NORME HORIZONTALE

Determination of certain substances in electrotechnical products – Part 3-1: Screening – Lead, mercury, cadmium, total chromium and total bromine by X-ray fluorescence spectrometry

Détermination de certaines substances dans les produits électrotechniques – Partie 3-1: Méthodes d'essai – Plomb, du mercure, du cadmium, du chrome total et du brome total par la spectrométrie par fluorescence X

IEC 62321-3-1:2013

IEC Central Office
3, rue de Varembé
CH-1211 Geneva 20
Switzerland

Tel.: +41 22 919 02 11
Fax: +41 22 919 03 00
info@iec.ch
www.iec.ch

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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International Standard IEC 62321-3-1 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 62321-3-1 is a partial replacement of IEC 62321:2008, forming a structural revision and generally replacing Clauses 6 and Annex D.

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
111/298/FDIS	111/308/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

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- withdrawn,
- replaced by a revised edition, or
- amended.

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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.

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 3-1: Screening – Lead, mercury, cadmium, total chromium and total bromine by X-ray fluorescence spectrometry

1 Scope

Part 3-1 of IEC 62321 describes the screening analysis of five substances, specifically lead (Pb), mercury (Hg), cadmium (Cd), total chromium (Cr) and total bromine (Br) in uniform materials found in electrotechnical products, using the analytical technique of X-ray fluorescence (XRF) spectrometry.

It is applicable to polymers, metals and ceramic materials. The test method may 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 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 Tables 1 to 5.

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]	Poly-olefine
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.

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.				

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

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 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]

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

3 Terms, definitions and abbreviations

For the purposes of this document, the terms, definitions and abbreviations given in IEC 62321-1 and IEC 62321-2 apply.

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 lead, mercury, cadmium, chromium and bromine (Pb, Hg, Cd, Cr, Br) 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 and bromine, where the result will reflect only the total chromium and total bromine present. The presence of Cr(VI) or the 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. Similarly, the analysis of Cr in conversion coatings may be difficult due to the presence of Cr in substrate material and/or because of insufficient sensitivity for Cr in typically very thin (several hundred nm) conversion coating layers.

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

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[1] To be published.

- 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/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 [1, 2 and 3].

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 may 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.

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 are considerably lower than for a high-performance test method for quantitative determinations with low estimates of uncertainty.

This test method is based on the concept of a performance based measurement system. Apparatus, sample preparation and calibration are specified in this standard 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.

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

WARNING 2 Xrays 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 [4, 5 and 6]:

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 and/or Br.

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 and/or Br.

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 may 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 portion. 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;

c) in a procedure that results in a material being dissolved in a liquid matrix, the quantity and physical characteristics of the material to be dissolved shall be controlled and documented. The resulting solution shall be completely homogeneous. Instructions shall be provided to deal with undissolved portions to ensure proper interpretation of the measured results. Instructions shall be provided for presentation of the test portion of the solution to the X-ray spectrometer in a repeatable manner, i.e. in a liquid cell of specified construction and dimensions;

d) in a procedure that results in a sample material being fused or pressed in a solid matrix, the quantity and physical characteristics of the sample material shall be controlled and documented. The resulting solid (fused or pressed pellet) shall be completely uniform. Instructions shall be provided to deal with unmixed portions to ensure proper interpretation of the measured results.

8 Test procedure

8.1 General

The test procedure covers preparation of the X-ray spectrometer, preparation and mounting of test portions and calibration. Certain instructions are presented in general terms due to the wide range of XRF equipment and the even greater variety of laboratory and test samples to which this test method will be applied. However, a cardinal rule that applies without exception to all spectrometers and analytical methods shall be followed; that is that the calibration and sample measurements be performed under the same conditions and using the same sample preparation procedures.

In view of the wide range of XRF spectrometer designs and the concomitant range of detection capabilities, it is important to understand the limitation of the chosen instrument. Certain designs may be incapable of detecting or accurately determining the composition of a very small area or very thin samples. As a consequence, it is imperative that users carefully establish and clearly document the performance of the test method as implemented in their laboratories. One goal is to prevent false negative test results.

8.2 Preparation of the spectrometer

Prepare the spectrometer as follows:

a) switch on the instrument and prepare it for operation according to the manufacturer's manual. Allow the instrument to stabilize as per guidelines established by the manufacturer or laboratory work instructions;

b) set the measurement conditions to the optimum conditions previously established by the manufacturer or the laboratory.

Many instruments available on the market are already optimized and preset for a particular application, and therefore this step might not be necessary. Otherwise, the laboratory should establish optimum operating conditions for each calibration. Choices should be made to optimize sensitivity and minimize spectral interferences. Excitation conditions may vary by material, analyte and X-ray line energy. A list of recommended analytical X-ray lines is given in Table 6. Detection system settings should optimize the compromise between sensitivity and energy resolution. Guidance can usually be found in the instrument manual and in literature on X-ray spectrometry [1, 2 and 3].