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Part 3-1: Elemental Screening by X-ray fluorescence spectrometry

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Introduction

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For international standards, comments will be reviewed by the relevant UK national committee before sending the consensus UK vote and comments to the international committee, which will then decide appropriate action. If the international standard is approved, it is usual for the text to be published as a British Standard.

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Template for comments and secretariat observations

Date: xx/xx/20xx	Document: ISO/DIS xxxx
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1	2	(3)	4	5	(6)	7
MB	Clause No./ Subclause No./Annex (e.g. 3.1)	Paragraph/Figure/ Table/Note	Type of comment	Commend (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	



111/697/CD

COMMITTEE DRAFT (CD)

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DATE OF CIRCULATION: 2023-03-10	CLOSING DATE FOR COMMENTS: 2023-06-02
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IEC TC 111 : ENVIRONMENTAL STANDARDIZATION FOR ELECTRICAL AND ELECTRONIC PRODUCTS AND SYSTEMS	
SECRETARIAT: Italy	SECRETARY: Mr Alfonso Sturchio
OF INTEREST TO THE FOLLOWING COMMITTEES:	PROPOSED HORIZONTAL STANDARD: ☒ Other TC/SCs are requested to indicate their interest, if any, in this CD to the secretary.
FUNCTIONS CONCERNED: ☐ EMC ☒ ENVIRONMENT ☐ QUALITY ASSURANCE ☐ SAFETY	

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- any relevant patent rights of which they are aware and to provide supporting documentation,
- any local regulations or technical reasons that may exist and should be considered should this proposal proceed, recognizing that failure to address such requirements could result in the need for new or existing "in some countries" clauses. See AC/22/2007.

TITLE:

**DETERMINATION OF CERTAIN SUBSTANCES IN ELECTROTECHNICAL PRODUCTS – Part 3-1:
Elemental Screening 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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- International Standard IEC 62321-3-1 has been prepared by IEC technical committee 111: Environmental standardization for electrical and electronic products and systems.

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

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)

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

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

FDIS	Report on voting

160
161 Full information on the voting for the approval of this standard can be found in the report on
162 voting indicated in the above table.

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

164 A list of all parts in the IEC 62321 series can be found on the IEC website under the general
165 title: *Determination of certain substances in electrotechnical products*

166 The committee has decided that the contents of this publication will remain unchanged until the
167 stability date indicated on the IEC web site under "<http://webstore.iec.ch>" in the data related to
168 the specific publication. At this date, the publication will be

- 169 • reconfirmed,
- 170 • withdrawn,
- 171 • replaced by a revised edition, or
- 172 • amended.

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INTRODUCTION

177 The widespread use of electrotechnical products has drawn increased attention to their impact
178 on the environment. In many countries this has resulted in the adaptation of regulations
179 affecting wastes, substances and energy use of electrotechnical products.

180 The use of certain substances (e.g. lead (Pb), cadmium (Cd) and polybrominated diphenyl
181 ethers (PBDEs)) in electrotechnical products, is a source of concern in current and proposed
182 regional legislation. With the actual revision the following elements are added: phosphorus (P),
183 assume the source of P is related to TCEP, Trixylyl-phosphate, chlorine (Cl), assume the source
184 of Cl is related to SCCP, TCEP, TBTC, tin (Sn), assume the source of Sn is related to restricted
185 organo-tin compounds, antimony (Sb), assume the source of Sb is related to Pyrochlore,
186 antimony lead yellow.

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

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

195

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

1 Scope

Part 3-1 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), assume the source of P is related to TCEP (CAS 115-96-8), Trixylyl-phosphate (CAS 25155-23-1), total chlorine (Cl), assume 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), assume the source of Sn is related to restricted organo-tin compounds, total antimony (Sb), assume the source of Sb is related to Pyrochlore, 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] 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, 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. During a Pre-IIS the feasibility of the test method to be used for the added elements was tested. The results are listed in the Tables 6-10.

NOTE The text of this document will be reviewed and will be used for a full IIS, which is planned for 2023.

237

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.										

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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.			

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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.				

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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 8300

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 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,

¹ To be published.

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.

For the screening analysis of critical raw materials only the analysis results is 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 TCEP, Trixylyl-phosphate, SCCP, 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 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:

- 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 [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 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