BS EN 62321-3-1:2014

BSI Standards Publication

Determination of certain substances in electrotechnical products

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

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National foreword

This British Standard is the UK implementation of EN 62321-3-1:2014. It is identical to IEC 62321-3-1:2013.
Together with BS EN 62321-1:2013, BS EN 62321-2:2014, BS EN 62321-3-2:2014, BS EN 62321-4:2014, BS EN 62321-5:2014, BS EN 62321-7-1, BS EN 62321-7-2 and BS EN 62321-8 it supersedes BS EN 62321:2009, which will be withdrawn upon publication of all parts of the BS EN 62321 series.

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A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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

(IEC 62321-3-1:2013)

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
(CEI 62321-3-1:2013)

Verfahren zur Bestimmung von bestimmten Substanzen in Produkten der Elektrotechnik -
Teil 3-1: Screening -
Blei, Quecksilber, Cadmium, Gesamtchrom und Gesamtbrom durch Röntgenfluoreszenz-Spektrometrie
(IEC 62321-3-1:2013)

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European Committee for Electrotechnical Standardization
Comité Européen de Normalisation Electrotechnique
Europäisches Komitee für Elektrotechnische Normung

CEN-CENELEC Management Centre: Avenue Marnix 17, B - 1000 Brussels

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Foreword

The text of document 111/298/FDIS, future edition 1 of IEC 62321-3-1, prepared by IEC/TC 111 "Environmental standardization for electrical and electronic products and systems" was submitted to the IEC-CENELEC parallel vote and approved by CENELEC as EN 62321-3-1:2014.

The following dates are fixed:

• latest date by which the document has to be implemented at national level by publication of an identical national standard or by endorsement	(dop)	2014-10-25
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EN 62321-3-1:2014 is a partial replacement of EN 62321:2009, forming a structural revision and generally replacing Clauses 6 and Annex D.

Future parts in the EN 62321 series will gradually replace the corresponding clauses in EN 62321:2009. Until such time as all parts are published, however, EN 62321:2009 remains valid for those clauses not yet re-published as a separate part.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CENELEC [and/or CEN] shall not be held responsible for identifying any or all such patent rights.

Endorsement notice

The text of the International Standard IEC 62321-3-1:2013 was approved by CENELEC as a European Standard without any modification.

Annex ZA
(normative)

Normative references to international publications with their corresponding European publications

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.

NOTE When an international publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

Publication	Year	Title	EN/HD	Year
IEC 62321-1	-	Determination of certain substances in electrotechnical products - Part 1: Introduction and overview	EN 62321-1	-
IEC 62321-2	-	Determination of certain substances in electrotechnical products - Part 2: Disassembly, disjunction and mechanical sample preparation	EN 62321-2	-
ISO/IEC Guide 98-1	-	Uncertainty of measurement - Part 1: Introduction to the expression of uncertainty in measurement	-	-

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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].

Table 6 – Recommended X-ray lines for individual analytes[a]

Analyte	Preferred line	Secondary line
Lead (Pb)	L_2–M_4 ($L\beta_1$)	L_3–$M_{4,5}$ ($L\alpha_{1,2}$)
Mercury (Hg)	L_3–$M_{4,5}$ ($L\alpha_{1,2}$)	
Cadmium (Cd)	K–$L_{2,3}$ ($K\alpha_{1,2}$)[b]	
Chromium (Cr)	K–$L_{2,3}$ ($K\alpha_{1,2}$)	
Bromine (Br)	K–$L_{2,3}$ ($K\alpha_{1,2}$)	K–$M_{2,3}$ ($K\beta_{1,3}$)

[a] Other X-ray line choices may provide adequate performance. However, when deciding on alternative analytical lines one should be aware of possible spectral interferences from other elements present in the sample (e.g. BrKα on PbLα or AsKα on PbLα lines; see Clause A.2 b) for more typical examples).

[b] K–$L_{2,3}$ ($K\alpha_{1,2}$) means that there are actually two transitions to the K shell, i.e. one from the L_2 shell which generates $K\alpha_2$ X-rays and another from the L_3 shell that generates $K\alpha_1$ X- rays. However, since both energies are very close, energy dispersive spectrometers cannot distinguish them and so they are analysed as one combined K $\alpha_{1,2}$ energy.

8.3 Test portion

The creation of a test portion is described in Clause 7.

In the case of destructive sample preparation, measure the mass and dimensions of the test portion as required by the calibration method and the work instruction established by the laboratory to ensure repeatable sampling. The location of the test portion shall also be documented in relation to its origin on the electrotechnical product.

8.4 Verification of spectrometer performance

Spectrometer performance shall be verified as follows:

a) Users shall provide objective evidence of the performance of the method as implemented in their laboratories. This is necessary to enable the users and their customers to understand the limitations of the method and to make decisions using the results of analyses. Critical aspects regarding the performance of the method are as follows:
   - sensitivity for each analyte;
   - spectral resolution;
   - limit of detection;
   - demonstration of measured area;
   - repeatability of sample preparation and measurement;
   - accuracy of calibration, which will be checked according to Clause 10.

   Given the variety of spectrometers and the associated software operating systems, it is acceptable for the users to obtain this information in their own laboratory using their own procedures or as a service provided by the manufacturer. It is important to obtain verification of spectrometer and method performance when the method is implemented. Evidence of the maintenance of performance may be obtained through the use of control charts or by repeating the measurements and calculations made at the time of implementation;

b) Spectrometer sensitivity is used as a figure of merit to compare spectrometers and to ensure that a meaningful calibration is possible.

c) Spectral resolution is important to ensure that the analyte and interfering spectral lines are handled correctly in the collection of data and in the calibration. For the purposes of this standard, the correction of line overlaps is considered as part of the spectrometer calibration.

d) The limit of detection, LOD, shall be estimated for each set of operating conditions employed in the test method using Equation (1) below:

$$\mathrm{LOD} = 3\sigma \qquad (1)$$

where

LOD is the limit of detection (LOD) for given analyte expressed in units of concentration;

σ is the standard deviation of the results of multiple determinations using a blank material. Standard deviation is usually estimated using a small (but not less than seven) number of determinations, in which case the symbol, *s* (the unbiased estimate of standard deviation, σ) is substituted for σ.

The limit of detection is a critical parameter that tells the user whether the spectrometer is being operated under conditions that allow the detection of an analyte at levels sufficiently below the allowed substance limits to be useful for making decisions [7, 8 and 9]. Limit of detection is a function of the measurement process of which the material is a significant part. If the measurement process changes when the material is changed, the limits of detection may also change. For optimum performance, the limit of detection should be equal to or less than 30 % of the laboratory's own action limits, established to provide maximum acceptable risk of non-compliance.

e) Demonstration of the measured area is important to ensure that the viewed area is known for the spectrometer equipped with any accessories that define X-ray beam size, shape and location. In many cases, the beam size, shape and location define the test portion. The laboratory or the manufacturer shall provide a means to define the beam size and shape and identify its location on the test portion.

f) Repeatability of sample preparation and measurement is an important parameter to demonstrate that the test method has statistical control. If destructive sample preparation precedes the measurement, the repeatability shall be tested, including sample preparation, otherwise repeatability of the measurement shall be tested on the same sample. Repeatability is expressed as the standard deviation of at least seven measurements of a prepared sample using the optimum spectrometer operating conditions. Repeatability shall be measured for each analyte in a test portion containing a concentration of the analyte greater than five times the limit of detection estimated in 8.4 d).

g) A quality control standard is used to verify that the method is in control. If the chosen quality control standard's repeatability varies by more than the repeatability value expected for that concentration (acceptance value obtained from Clause 10) then the procedure is deemed to be out of control and the instrument should be recalibrated before running any further analysis.

NOTE Not all matrices currently have appropriate reference samples available for use as QC samples. For such cases, it is common practice to use in-house well characterized samples.

8.5 Tests

Place the test portion in the correct position for measurement with the XRF spectrometer. If necessary, establish the required atmosphere in the chamber of the spectrometer and allow it to stabilize.

Measurements are typically made in an air atmosphere. However, should there be a need to measure light elements such as S, Al, etc., it may be advantageous to measure in a vacuum or helium atmosphere.

Measure the test portion by collecting sufficient numbers of X-ray counts to attain a counting statistical uncertainty less than the established relative standard deviation for measurement repeatability (see 8.4). The settings of the XRF spectrometer for analysis of the test portion shall be identical to those used for calibration measurements.

8.6 Calibration

The analytical method shall be calibrated taking into account matrix effects and other effects that influence the determination of the intensity of the fluorescence radiation. These effects are discussed in detail in Clause A.2.

There are two principal calibration options in XRF spectrometry:

- Fundamental parameters approaches which employ as calibrants pure elements, pure compounds, mixtures of compounds or reference materials with well defined matrix compositions. As with all XRF calibrations, accuracy can be expected to improve when the calibrants are increasingly similar to the samples to be analysed.
- Empirical (traditional) calibration using a model based on influence coefficients derived either from empirical data obtained with a suite of calibrants similar to the unknowns, or derived using a fundamental parameters approach.

Follow the guidelines in the manufacturer's manual when selecting the calibration options available in the operating system software.

Depending on the instrument, the user may or may not be required to perform the calibration. There are a number of commercially available instruments which are already optimized, calibrated and preset for specific applications. These instruments do not require calibration by the analyst.

The choice of calibrants depends in part on the choice of calibration model. For empirical options, the calibrants shall be similar in matrix composition to the materials to be analysed. In this scenario the minimum number of calibrants for an empirical method is $2(n+2)$, where n is the number of analytes. In the set of calibrants, element concentrations shall cover the range of concentrations expected in the samples and they shall vary independently of one another. If the calibration covers many elements in a wide range of concentrations, a large number of calibration samples may be necessary.

A fundamental parameters calibration approach can significantly reduce the number of calibration samples. Fundamental parameters software allows the user to calibrate the sensitivity of each element using pure elements and compounds. As an alternative to using pure elements or substances as calibrants, the software will typically allow the use of a small number of reference materials which more closely resemble actual samples. Enhancements of the method include the use of scattered radiation to correct for certain matrix or sample morphology effects.

9 Calculations

The following calculations shall be performed as necessary when using this test method:

a) In contemporary instruments the calculations are typically performed automatically by the spectrometer operating system software. If calculations are to be done by hand, the algorithms and all the parameters shall be specified in the work instructions for the test method. Calculate the result for each analyte, in per cent by mass, in each test portion using the calibration model established for the sample type.

b) If the test portion has been prepared by dilution, calculate the result on the basis of the original test sample using the appropriate dilution factor.

   Estimate the uncertainty of the results using one of the following methods and compare the result to the maximum allowed concentration of the analyte in the material.

c) The preferred method is to create an uncertainty budget for each calibration implemented in the test method. The uncertainty budget shall be compliant with ISO/IEC Guide 98-1. Express the expanded uncertainty estimate at the 95 % confidence level.

   It is an oversimplification to assign the uncertainty as some multiple of the repeatability standard deviation of replicate determinations. Under certain circumstances, XRF measurements can be far too precise, leading to an estimated uncertainty that is too small to cover all sources of error. This approach ignores important contributions from the calibrants, the mathematical model used to fit the calibration curve and the potential for the introduction of bias during sample preparation. Moreover, the definition of an uncertainty budget is beyond the scope of this standard.

d) If it is impractical or impossible to perform a proper uncertainty budget, prepare for each analyte, *i*, the estimate of the expanded uncertainty, U_i, which shall include a safety factor expressed as the fraction of the maximum allowed concentration of the analyte, *i*. In practice, this amounts to defining a confidence interval around the maximum allowed concentration value of the analyte, which can be used for the purpose of making decisions regarding the need for additional testing. The concept of safety factor and guidance on its selection are discussed in detail in Clause A.3.

10 Precision

10.1 General

The detailed summary of results obtained in the course of international interlaboratory studies 2 and 4 (IIS2 and IIS4) for each substance and material tested using XRF are listed in Tables A.3 to A.7. Only these results shall be a basis for any conclusions about the method performance.

The following general conclusions can be made, based on the results summarized in the tables and the analysis of data from IIS2 and IIS4.

a) Evaluation of the results and method performance can only be fragmentary because of the shortage of certified reference material (CRM) to fully cover the required ranges of concentrations and types of materials.
b) Due to the limited amounts of available CRM, not all laboratories tested all samples; consequently, the results are not always directly comparable. Additionally, some samples of the same material were in granular or chip form while other were in solid form such as plates.
c) The samples were analysed “as received”, i.e. no sample preparation was involved.
d) Precisions reported by individual laboratories for individual results were typically at much less than 5 % relative standard deviation (RSD).
e) The participating laboratories used various calibration methods, such as empirical, Compton normalization and methods based on fundamental parameters.
f) It is imperative that the method performance be further researched and tested during interlaboratory studies.

10.2 Lead

The average inaccuracy of Pb determination in polymers above a level of 100 mg/kg was better than ± 13 % relative and the imprecision was better than ± 19 % relative. At a Pb concentration of 10 mg/kg, the inaccuracy and imprecision were ± 30 % relative and ± 70 % relative, respectively. In Al alloys, the inaccuracy and imprecision were less than ± 10 % relative and ± 25 % relative, respectively. A Pb concentration of 174 mg/kg in tin-based alloy (an example of lead-free solder) produced results ranging from 60 mg/kg to 380 mg/kg. 30 mg/kg of Pb in an alloy steel was not detected.

The results for ground PWBs point to possible non-homogeneity of the material as the source of great imprecision and inaccuracy of the results.

10.3 Mercury

The average inaccuracy of Hg determination in polymers at or below 1 000 mg/kg was better than ± 10 % relative, while the imprecision was better than ± 25 % relative. No alloy material was tested for Hg.

10.4 Cadmium

The average inaccuracy of Cd determination in polymers at or above 100 mg/kg was ± 10 % relative, and the imprecision was better than ± 15 % relative. At a level of 20 mg/kg Cd, the inaccuracy varied from ± 10 % to ± 50 % relative, and the imprecision varied from 20 % to 100 % relative. A level of 3,3 mg/kg of Cd in tin-based alloy was not detected by any instrument.

10.5 Chromium

The average inaccuracy of total Cr determination in polymers at or below 115 mg/kg was observed to be better than 17 % relative while the imprecision was about ± 30 % relative. For a similar concentration level in glass, the inaccuracy and imprecision for total Cr were better than ± 20 % relative and 35 % relative respectively. In aluminium alloys at 1 100 mg/kg Cr, the inaccuracy and imprecision were ± 10 % relative and better than ± 41 % relative, respectively.

10.6 Bromine

Based on the CRMs, the average inaccuracy of determination of total Br concentration in polymers at or below 1 000 mg/kg was better than ± 10 % relative, and the standard deviation was better than ± 13 % relative. At elevated Br concentrations of 10 %, inaccuracy was better than ± 25 % relative and imprecision was about ± 30 % relative. These latter results reflect the inadequacy of empirical calibrations for high Br concentrations. This also confirms the fact that the instrument calibration optimized for low concentrations of analyte (such as from 0 mg/kg to 1 500 mg/kg) may not be accurate for concentrations larger by one or two orders of magnitude. However, all instruments flagged Br concentrations larger than 1 000 mg/kg as non-compliant.

Generally, the inaccuracy and imprecision of analysis for all of the five elements were better than ± 20 % relative for concentrations above 100 mg/kg in polymers and aluminium alloys.

10.7 Repeatability statement for five tested substances sorted by type of tested material

10.7.1 General

When the values of two independent single test results, obtained using the same method, on identical test material, in the same laboratory, by the same operator, using the same equipment, within a short interval of time, lie within the range of the mean values cited below, the absolute difference between the two test results obtained shall not exceed the repeatability limit *r* deduced by linear interpolation from the following data in more than 5 % of cases.

10.7.2 Material: ABS (acrylonitrile butadiene styrene), as granules and plates

Parameter	**Material**			
	1	2	3	4
Bromine content (mg/kg)	25	938	116 800	118 400
r, (mg/kg)	2,5	44,54	9 093	11 876
Cadmium content (mg/kg)	10	94	100	183
r, (mg/kg)	5	19	7,3	14,25
Chromium content (mg/kg)	16	47	100	944
r, (mg/kg)	4,92	6,95	68	127
Mercury content (mg/kg)	33	63	100	942
r, (mg/kg)	3,56	3,47	17	72
Lead content (mg/kg)	15,7	100	954,3	945

Parameter	Material			
	1	2	3	4
r, (mg/kg)	1,96	18	35,66	192

10.7.3 Material: PE (low density polyethtylene), as granules

Parameter	Material			
	1	2	3	4
Bromine content (mg/kg)	96	98	770	808
r, (mg/kg)	5,46	12	11,32	42
Cadmium content (mg/kg)	19,6	22	137	141
r, (mg/kg)	3,42	8	5,6	33
Chromium content (mg/kg)	18	20	100	115
r, (mg/kg)	7	9,53	2,8	25
Mercury content (mg/kg)	5	5	24	25
r, (mg/kg)	0,81	2	0	11
Lead content (mg/kg)	14	14	98	108
r, (mg/kg)	1,02	4	3,23	16

10.7.4 Material: PC/ABS (polycarbonate and ABS blend), as granules

Parameter	Material	
	1	2
Bromine content (mg/kg)	800	2 400
r, (mg/kg)	30	100

10.7.5 Material: HIPS (high impact polystyrene)

Parameter	Material	
	1	2
Bromine content (mg/kg)	99 138	100 050
r, (mg/kg)	20 766	12 629

10.7.6 Material: PVC (polyvinyl chloride), as granules

Lead content (mg/kg)	390-665
r, (mg/kg)	67

10.7.7 Material: Polyolefin, as granules

Lead content, (mg/kg)	380-640
r, (mg/kg)	16

10.7.8 Material: Crystal glass

Lead content, (mg/kg)	240 000
r, (mg/kg)	12 070

10.7.9 Material: Glass

Chromium content, (mg/kg)	94
r, (mg/kg)	11

10.7.10 Material: Lead-free solder, chips

Lead content (mg/kg)	174
r , (mg/kg)	39

10.7.11 Material: Si/Al Alloy, chips

Lead content (mg/kg)	930
r, (mg/kg)	204

Chromium content (mg/kg)	1 100
r, (mg/kg)	242

10.7.12 Material: Aluminum casting alloy, chips

Lead content (mg/kg)	190
r, (mg/kg)	60

Chromium content (mg/kg)	130
r, (mg/kg)	40

10.7.13 Material: PCB – Printed circuit board ground to less than 250 µm

Lead content (mg/kg)	23 000
r, (mg/kg)	2 562

10.8 Reproducibility statement for five tested substances sorted by type of tested material

10.8.1 General

When the values of two single test results, obtained using the same method on identical test material, in different laboratories, by different operators, using different equipment, lie within the range of the values cited below, the absolute difference between the two results will not be greater than the reproducibility limit *R* deduced by linear interpolation from the following data in more than 5 % of cases.

10.8.2 Material: ABS (Acrylonitrile butadiene styrene), as granules and plates

Parameter	**Material**			
	1	2	3	4
Bromine content (mg/kg)	25	938	116 800	118 400
R, (mg/kg)	20,53	203,74	83 409	94 258
Cadmium content (mg/kg)	10	94	100	183
R, (mg/kg)	6	83	53,32	41,57
Chromium content (mg/kg)	16	47	100	944
R, (mg/kg)	10,06	25,69	120	405
Mercury content (mg/kg)	33	63	100	942
R, (mg/kg)	15,06	27,23	44	314
Lead content (mg/kg)	15,7	100	954,3	945
R, (mg/kg)	6,25	56	284,76	475

10.8.3 Material: PE (low density polyethylene), as granules

Parameter	**Material**			
	1	2	3	4
Bromine content (mg/kg)	96	98	770	808
R, (mg/kg)	5,62	40	--	340
Cadmium content (mg/kg)	19,6	22	137	141
R, (mg/kg)	9,34	13	--	57
Chromium content (mg/kg)	18	20	100	115
R, (mg/kg)	15	10,64	--	80
Mercury content (mg/kg)	5	5	24	25
R, (mg/kg)	0,69	4	--	14
Lead content (mg/kg)	14	14	98	108
R, (mg/kg)	5,08	28	--	59

10.8.4 Material: PC/ABS (Polycarbonate and ABS blend), as granules

Parameter	**Material**	
	1	2
Bromine content (mg/kg)	800	2 400
R, (mg/kg)	253	1 309

10.8.5 Material: HIPS (high impact polystyrene)

Parameter	**Material**	
	1	2
Bromine content (mg/kg)	99 138	100 050
R, (mg/kg)	106 216	102 804

10.8.6 Material: PVC (polyvinyl chloride), as granules

Lead content (mg/kg)	390-665
R, (mg/kg)	443

10.8.7 Material: Polyolefin, as granules

Lead content (mg/kg)	380-640
R, (mg/kg)	209

10.8.8 Material: Crystal glass

Lead content (mg/kg)	240 000
R, (mg/kg)	182 314

10.8.9 Material: Glass

Chromium content (mg/kg)	94
R, (mg/kg)	83

10.8.10 Material: Lead-free solder, chips

Lead content (mg/kg)	174
R, (mg/kg)	155

10.8.11 Material: Si/Al alloy, chips

Lead content (mg/kg)	930
R, (mg/kg)	790

Chromium content (mg/kg)	1 100
R, (mg/kg)	1963

10.8.12 Material: Aluminum casting alloy, chips

Lead content, (mg/kg)	190
R, (mg/kg)	153

Chromium content, (mg/kg)	130
R, (mg/kg)	107

10.8.13 Material: PCB – Printed circuit board ground to less than 250 μm

Lead content (mg/kg)	23 000
R, (mg/kg)	14 173

11 Quality control

11.1 Accuracy of calibration

The following steps shall be taken to validate the accuracy of calibration:

a) The accuracy of each calibration shall be validated by analysing one or more reference materials representative of each material used in the implementation of this test method. Analyte concentration levels in the reference materials shall be within one order of magnitude of the maximum allowed values for the analyte in the material. Ideally, reference materials will be available to bracket the maximum allowed values.

b) Results of measurements of the reference materials shall be calculated and expressed according to Clause 9, including an estimate of uncertainty.

c) Apply a bias test to the results and the certified or reference values assigned to the reference materials. The bias test shall take into account the uncertainty of an assigned value.

   For guidance on bias tests, refer to the National Institute of Standards and Technology Special Publication 829 [10] or similar documents.

d) If a bias is detected, the calibration shall be corrected and the validation repeated.

11.2 Control samples

Control samples shall be prepared and used as follows:

a) Designate a quantity of stable material as the control sample for each calibration. Preferably, this shall be a solid in the form of a disc (pellet).

b) Prepare a test portion of the control sample and subject it to testing using each of the calibrations as soon as they have been validated. Do this at least four times. Calculate the average and standard deviation and use these values to establish a control chart for each analyte in each calibration. Control samples may be created by the analysts. Some instrument manufacturers provide control sample(s) with their equipment.

c) At appropriate time intervals, prepare a test portion of the control sample and subject it to testing, using each of the calibrations implemented in the test method. Compare the results to the control chart limits. If the results violate accepted rules for control, troubleshoot the test methods, correct the problem and perform a test on a new control sample.

12 Special cases

The precision described in this test method may not be achieved in any of the following cases:

a) analysis of samples which are not flat or large enough to cover the measuring aperture of the spectrometer;

b) thin samples or multi-layered samples;

c) non uniform samples.

13 Test report

Results of all tests performed on analysed materials shall be recorded in the report which shall include following components:

- information necessary for unambiguous identification of the sample tested;
- date, time and location of the test;
- reference to this standard (IEC 62321-3-1);
- test procedure used;

- results of the test and uncertainty estimate for each analyte;
- any deviations from the specified procedure;
- any anomalies observed during the test.

Annex A
(informative)

Practical aspects of screening by X-ray fluorescence spectrometry (XRF) and interpretation of the results

A.1 Introductory remark

This annex provides general information to aid in the practical application of the method described above. Some manufacturers may provide a standard operating procedure (SOP) with the instrument. Following the recommendation contained in such a document assures the operator of the best possible quality of analytical results.

A.2 Matrix and interference effects

As a general guide, the user of this method is advised that limitations in corrections for spectral interference and matrix variations from material to material may significantly affect the sensitivity, detection limit or accuracy of determination for each analyte. The following list covers the most common issues:

a) The intensity of characteristic radiation of the element in the sample is adversely influenced by the process of scattering of the excitation radiation, which contributes to the spectral background. In addition, two major effects occur:
   1) absorption of excitation radiation and fluorescence radiation by the analyte and by the other elements (matrix) in the sample;
   2) secondary excitation (enhancement) of the analyte by other elements in the sample:
      - polymers: In polymer samples the matrix influence on the analyte characteristic X-ray intensity comes from:
        - the scattering (mainly incoherent) of the primary radiation, which contributes heavily to the spectral background;
        - the absorption of the fluorescence radiation mainly by Cl in PVC, by additive elements such as Ca, Ti, Zn, Sn, and by such elements as Br and Sb, which originate in flame retardants;
        - the secondary excitation by elements such as Sb, Sn, and Br;
        - some high-powered WDXRF (>500 W) spectrometers can irreversibly alter the surface of a polymer sample if exposed to the X-ray beam for long periods of time. A newly prepared sample shall always be used in this case.
      - metals: In metal samples the scattering of the primary radiation, while still present, does not play an important role. The matrix effects are mainly caused by absorption and secondary excitation. These will be different for each metal matrix. The following list shows some typical elements in the various matrices:
        - Fe alloys: Fe, Cr, Ni, Nb, Mo, W;
        - Al alloys: Al, Mg, Si, Cu, Zn;
        - Cu alloys: Cu, Zn, Sn, Pb, Mn, Ni, Co;
        - Solder alloys: Pb, Cu, Zn, Sn, Sb, Bi, Ag;
        - Zn alloys: Zn, Al;
        - Precious metals alloys: Rh, Pd, Ag, Ir, Pt, Au, Cu, Zn;
        - Other metals such as Ti, Mg.
      - electronics: In principle all effects that are described for polymers and metals.

b) In addition, the intensity of characteristic radiation of the element in the sample can be influenced by interfering lines from other elements in the sample. For the target elements, these can typically be the following:
   - Cd: interferences possible from Br, Pb, Sn, Ag and Sb;
   - Pb: interferences possible from Br, As, Bi;
   - Hg: interferences possible from Br, Pb, Bi, Au and from Ca and Fe if the samples contain Ca and Fe in high concentrations;
   - Cr: interferences possible from Cl;
   - Br: interferences possible from Fe, Pb and Hg. On rare occasions an interference from Al might be experienced if a BrL_α line is selected to analyse Br.

c) Influence of matrix effects on LOD.

Table A.1 – Effect of matrix composition on limits of detection of some controlled elements

Element/analyte	Pure polymer	Polymer with ≥ 2 % Sb, without Br	Polymer with ≥ 2 % Br, without Sb
Cadmium	A	~ A → 2A	≥2A
Lead	B	~ 2B	≥3B

NOTE 1 If A and B are limits of detection (LOD) for Cd and Pb, respectively, in a pure polymer, then the LODs to be expected for more complex matrices are expressed as multiples of A and B as in Table A.1.

NOTE 2 The information in Table A.1 is provided as guidance only; the actual LODs for the target analytes are specific for each instrument and analytical conditions/parameters employed.

A.3 Interpretation of results

For each analyte, the analyst shall prepare an uncertainty budget with an estimate of the expanded uncertainty, U, expressed at a chosen confidence level. Using the value for U and the maximum allowed level, L, of the substance, the analyst shall categorize each sample as:

a) “BELOW LIMIT” – If the results, C_i, of the quantitative analysis for all analytes are lower than the pass values, P_i, calculated by Equation (A.1), the result for the sample is “BELOW LIMIT”.

$$P_i = L_i - U_i \tag{A.1}$$

where “i” indicates each analyte.

b) “OVER LIMIT” – If the results, C_i, of the quantitative analysis for any individual analyte are higher than the fail values, F_i, calculated from Equation (A.2), the result for the sample is “OVER LIMIT”.

$$F_i = L_i + U_i \tag{A.2}$$

c) “INCONCLUSIVE” – If the result, C_i, of the quantitative analysis for any individual analyte in a sample is intermediate between P_i and F_i, the test is “INCONCLUSIVE” for that sample.

NOTE 1 If the maximum allowed level restricts PBB/PBDE and Cr(VI) rather than Br and Cr, the exceptions are the XRF determinations of Br and Cr. If the quantitative results for the elements Br and/or Cr are higher than the limit (for Br calculated based on the stoichiometry of Br in the most common congeners of PBB/PBDE), the sample is “inconclusive”, and even if the quantitative results for all other analytes are “below limit”.

- The value L is defined by the restrictions being used to judge the acceptability of the material in the product. If the material listed in the governing restrictions is in the elemental form, L shall be used directly from the governing restrictions. If the material listed in the governing restrictions is in compound form, the value for L shall be calculated using the gravimetric factor for the element being determined using XRF in the target chemical compound.

- The value U above denotes an estimate of the expanded uncertainty associated with the XRF determination of each analyte. That is, U is different for each combination of analyte, sample preparation procedure, calibration and spectrometer. Guidance on the estimation of uncertainty may be obtained from ISO/IEC Guide 98-1.

d) If it is impractical or impossible to perform a proper uncertainty budget, the value of expanded uncertainty, U_i, can be estimated as a sum of the repeatability of the analyser and relative uncertainty expressed as a safety factor equal to 30 % of the maximum allowed value of analyte concentration (50 % for composite material). For the purpose of this test method, the safety factor value of 30 % (50 % for composite materials) has been agreed to and recommended through consensus between the experts in the field. However, the user of this test method may select different safety factor(s) based on experience and knowledge of the materials tested.

e) Table A.2 gives an example of a scheme for interpreting results at sample limits and given safety factors.

Table A.2 – Screening limits in mg/kg for regulated elements in various matrices

Element	Polymers	Metals	Composite material
Cd	BL ≤(70-3σ) <X <(130+3σ) ≤OL	BL ≤(70-3σ) <X <(130+3σ) ≤OL	LOD <X <(150+3σ) ≤OL
Pb	BL ≤(700-3σ) <X <(1 300+3σ) ≤OL	BL ≤(700-3σ) <X <(1 300+3σ) ≤OL	BL ≤(500-3σ) <X <(1 500+3σ) ≤OL
Hg	BL ≤(700-3σ) <X <(1 300+3σ) ≤OL	BL ≤(700-3σ) <X <(1 300+3σ) ≤OL	BL ≤(500-3σ) <X <(1 500+3σ) ≤OL
Br	BL ≤(300-3σ) <X	Not applicable	BL ≤(250-3σ) <X
Cr	BL ≤(700-3σ) <X	BL ≤(700-3σ) <X	BL ≤(500-3σ) <X

- A common set of limits for the substances of interest have been assumed for the purposes of this example. The limits are 100 mg/kg for Cd and 1 000 mg/kg for Pb, Hg and Cr. The limit for Br is calculated based on the stoichiometry of Br in the most common congeners of PBB/PBDE and their limit of 1 000 mg/kg. The “action levels” for this method have been set for the purpose of this screening procedure with a 30 % margin of safety (50 % for composite materials).
- A “BELOW LIMIT” (BL) or “OVER LIMIT” (OL) determination will be set at 30 % (50 % for composite materials) less than or greater than the limit, respectively.The symbol “X” marks the region where further investigation is necessary.
- The term “3σ” expresses the repeatability of the analyser at the action level, where σ is determined as the standard deviation of the result obtained on a typical sample with the content of the regulated substances near the limits of interest (see spectrometer performance verification test 8.4 d). The repeatability is expressed in terms of “3σ” 99,7 % confidence level rather than the more common “2σ” 95 % confidence level. The 99,7 % confidence level will allow the method to produce fewer “false negative errors”.

  Accordingly, and in agreement with Clause A.3, the expanded uncertainty, U_i, for the above three examples can be written as follows:

$$U_i = 0{,}3 * L_i + 3\sigma \qquad \text{(A.3)}$$

or

$$U_i = 0{,}5 * L_i + 3\sigma \qquad \text{(A.4)}$$

where 0,3 and 0,5 are the “30 % and 50 % safety factors, respectively”. Therefore, if for example the maximum allowed level, L, of the substance is set at 100 mg/kg, then Equation (A.1) will be:

$$P_i = L_i - U_i = 100 - 0{,}3*100 - 3\sigma = 70 - 3\ \sigma \qquad \text{(A.5)}$$

and Equation (A.2) will be:

$$F_i = L_i + U_i = 100 + 0{,}3*100 + 3\sigma = 130 + 3\ \sigma \qquad \text{(A.6)}$$

These are the exact expressions for acceptance limits for Cd in polymers and metals listed in Table A.2. Other acceptance limits were obtained in similar manner.

The limit of detection of the instrument should be below the “action level” and should be applied in accordance with 8.4 d).

The use of a safety factor is an over-simplification due in part to the fact that, in most cases, relative uncertainty is a function of concentration. Typically, relative uncertainty increases rapidly as the analyte concentration decreases. The analyst is cautioned not to interpret the 30 % safety factor as a relative uncertainty of results of determinations. The analyst is also cautioned to re-evaluate the safety factor if the detection limit is greater than 20 % relative to the maximum allowed concentration, or if the maximum allowed concentration is reduced.

A.4 Statistical data of the IIS2 and IIS4 for the XRF method

Volunteer laboratories participated in an international inter-laboratory studies IIS2 and IIS4 to determine the performance of this test method. The samples used in studies were CRMs (certified reference materials) that were donated, research samples of known composition, and real samples which were analysed according to the procedures described in this test method. The equipment used in these tests ranged from laboratory ED-XRF or WD-XRF, through bench-top to portable and hand-held XRF analysers. Samples were analysed “as is”. All samples were assumed to be homogeneous, although this assumption has been validated only for CRM samples. The most questionable was homogeneity of samples of ground printed wiring board (F20 and F21).

Statistical data were calculated according to ISO 5725-2 [7]. Some reproducibility values are not provided due to the low number of accepted results.

Table A.3 – Statistical data from IIS2

Sample	**Parameter**	**m**[a] mg/kg	**v**[b] mg/kg	**N**[c]	**s(r)**[d] mg/kg	**r**[e] mg/kg	**s(R)**[f] mg/kg	**R**[g] mg/kg
IIS2-A01	Br	109 137	99 138	21	7 416	20 766	37 934	106 216
IIS2-A02	Br	118 099	100 050	21	4 510	12 629	36 716	102 804
IIS2-A03	Br	115 038	116 800	13	3 247	9 093	29 789	83 409
IIS2-A04	Br	124 408	118 400	13	4 242	11 876	33 663	94 258
IIS2-A05	Br	995	800	9	11	30	90	253
IIS2-A06	Br	3 034	2 400	9	36	100	468	1 309
IIS2-C10	Br	771	808	22	15	42	122	340
IIS2-C11	Br	90	98	19	4	12	14	40
IIS2-B08	Pb	492	390-665	16	24	67	158	443
IIS2-B09	Pb	552	380-640	5	6	16	74	209
IIS2-C10	Pb	115	108	26	6	16	21	59
IIS2-C11	Pb	18	14	19	1	4	10	28
IIS2-C12	Pb	97	100	35	6	18	20	56
IIS2-C13	Pb	950	945	34	69	192	169	475
IIS2-D15	Pb	187	190	10	21	60	55	153
IIS2-D16	Pb	1 021	930	21	73	204	282	790
IIS2-E19	Pb	191	174	8	14	39	55	155
IIS2-F20	Pb	17 252	23 000	10	915	2 562	5 062	14 173
IIS2-F22	Pb	232 192	240 000	10	4 311	12 070	65 112	182 314
IIS2-C10	Cd	131	141	23	12	33	21	57
IIS2-C11	Cd	20	22	25	3	8	5	13
IIS2-C12	Cd	10	10	10	2	5	2	6
IIS2-C13	Cd	96	94	31	7	19	30	83
IIS2-C10	Hg	29	25	19	4	11	5	14
IIS2-C11	Hg	5	5	10	1	2	2	4
IIS2-C12	Hg	92	100	32	6	17	16	44
IIS2-C13	Hg	893	942	32	26	72	112	314
IIS2-B07	Cr	77	94	6	4	11	42	116
IIS2-C10	Cr	124	115	23	9	25	29	80
IIS2-C11	Cr	19	18	16	3	7	5	15
IIS2-C12	Cr	125	100	29	24	68	43	120
IIS2-C13	Cr	1 037	944	25	45	127	145	405
IIS2-D15	Cr	114	130	6	14	40	38	107
IIS2-D16	Cr	1 365	1 100	15	86	242	701	1 963

[a] m is the arithmetic mean of test results.

[b] v is the expected value.

[c] N is the number of accepted results.

[d] s(r) is the repeatability standard deviation.

[e] r is the repeatability limit.

[f] s(R) is the reproducibility standard deviation.

[g] R is the reproducibility limit.

Table A.4 – Statistical data from IIS4

Sample	**Parameter**	**m**[a] mg/kg	**v**[b] mg/kg	**N**[c]	**s(r)**[d] mg/kg	**r**[e] mg/kg	**s(R)**[f] mg/kg	**R**[g] mg/kg
IIS4A-04	Cd	176,1	183	21	5,09	14,25	14,85	41,57
IIS4A-05	Cd	104,4	100	15	2,61	7,30	19,04	53,32
IIS4A-07	Cd	21,8	19,6	6	1,22	3,42	3,33	9,34
IIS4A-08	Cd	105,0	137	3	2,00	5,60	–	–
IIS4A-04	Pb	15,3	15,7	18	0,70	1,96	2,23	6,25
IIS4A-05	Pb	1 033,5	954,3	15	12,74	35,66	101,70	284,76
IIS4A-07	Pb	15,0	14	6	0,36	1,02	1,82	5,08
IIS4A-08	Pb	77,3	98	3	1,15	3,23	–	–
IIS4A-04	Hg	31,3	33	21	1,27	3,56	5,38	15,06
IIS4A-05	Hg	63,5	63	15	1,24	3,47	9,72	27,23
IIS4A-07	Hg	4,8	5	6	0,29	0,81	0,25	0,69
IIS4A-08	Hg	10,0	24	3	0,00	0,00	–	–
IIS4A-04	Cr	42,0	47	18	2,48	6,95	9,18	25,69
IIS4A-05	Cr	16,3	16	12	1,76	4,92	3,59	10,06
IIS4A-07	Cr	18,5	20	6	3,40	9,53	3,80	10,64
IIS4A-08	Cr	102,0	100	3	1,00	2,80	–	–
IIS4A-04	Br	996,2	938	21	15,91	44,54	72,76	203,74
IIS4A-05	Br	24,1	25	15	0,89	2,50	7,33	20,53
IIS4A-07	Br	97,1	96	9	1,95	5,46	2,01	5,62
IIS4A-08	Br	670,7	770	3	4,04	11,32	–	–

[a] m is the arithmetic mean of test results.

[b] v is the expected value.

[c] N is the number of accepted results.

[d] s(r) is the repeatability standard deviation.

[e] r is the repeatability limit.

[f] s(R) is the reproducibility standard deviation.

[g] R is reproducibility limit.

Annex B
(informative)

Practical examples of screening with XRF

B.1 Introductory remark

This Part 3-1 of IEC 62321 outlines XRF (X-ray fluorescence) screening as a method to determine the presence or absence of restricted substances in electrotechnical products. XRF is a useful technique to study the chemical content of electrotechnical products and in deciding which parts should be disjointed further and which do not require further disjointment and testing.

B.2 XRF instrumentation

XRF instrumentation is available in many different configurations, from those which can analyse large, bulk samples in a defined measurement position to those that have the ability to isolate and analyse small objects within a complex sample, such as a surface mounted component on an assembled PWB.

Laboratory XRF instrumentation (both energy dispersive, ED-XRF, and wavelength dispersive, WD-XRF) typically offers the highest excitation power, but not the ability to measure small objects in complex samples. Generally, samples are ground into a homogeneous powder and transferred to a special sample cup prior to measurement. This class of instrumentation is very useful for screening and quantifying raw materials such as polymers before moulding.

Another class of XRF instruments is characterized by a collimated excitation X-ray beam, the so-called small-spot and micro-spot XRF analysers, that allow screening of much smaller samples than the typical laboratory XRF equipment. The size of the area analysed on a sample may vary from 0,1 mm^2 to approximately 10 mm^2. Some of these instruments have the ability to measure both the composition and thickness of multi-layer samples if their structure is known.

Finally, portable hand-held XRF instrumentation exists that offers the highest versatility of sampling and therefore can be used for in-situ screening and analysis under different circumstances. These instruments allow measurement of samples of any size and shape, since the analyser is placed on the sample rather than the sample being extracted from the object and placed on the instrument. The typical spot size of hand-held portable XRF instruments ranges in diameter from about 3 mm to 10 mm, which in some instances may be too large for the analysis of small objects.

All three configurations of XRF analysers discussed here offer detection limits acceptable for screening.

B.3 Factors affecting XRF results

B.3.1 General

When using XRF analytical techniques there are several factors that may affect the quality of the results, some of which are listed below:

- it is essential that the sample being analysed is homogeneous for quantitative results to be reliable;
- it is necessary to ensure that only the area of interest on the sample is confined within the measurement area (window) of the analyser;
- it is essential to understand

a) the depth of penetration of excitation X-rays, and

b) the depth from which fluorescence X-rays can be observed in the analysed material in order to correctly interpret the results obtained;

- when analysing multilayer samples, dedicated software should be used that will properly account for both thickness and composition of each layer.

B.3.2 Examples of screening with XRF

The following examples illustrate how XRF screening can be used to determine the compliance status of various samples and how the results of screening may affect further sampling decisions.

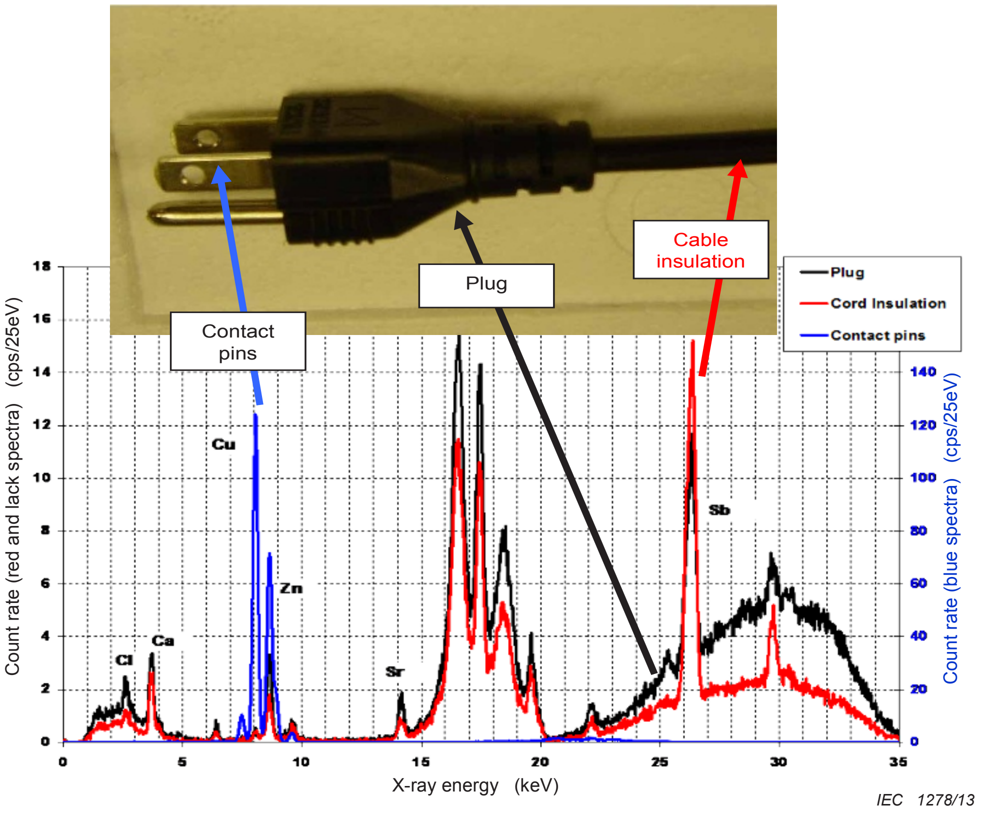
B.3.2.1 AC power cord

Figure B.1 below shows one end of the AC power cord. On visual inspection of the cord, three separate sections can be distinguished which are marked with arrows. These sections were also selected as samples (locations to be tested) for screening with XRF. Table B.1 summarizes the screening of the product.

Table B.1 – Selection of samples for analysis of AC power cord

Section identified	Material	Elements monitored	Probability of presence	Select for analysis
Plastic insulation of cable	Polymer	Pb, Br, Sb[a]	High	Yes
Plastic body of plug	Polymer	Pb, Br, Sb[a]	High	Yes
Metal prongs	Metal alloy	Cu, Zn,(Pb)	Moderate	Yes

[a] Presence of bromine (Br) and antimony (Sb) could indicate the use of a restricted brominated flame retardant.



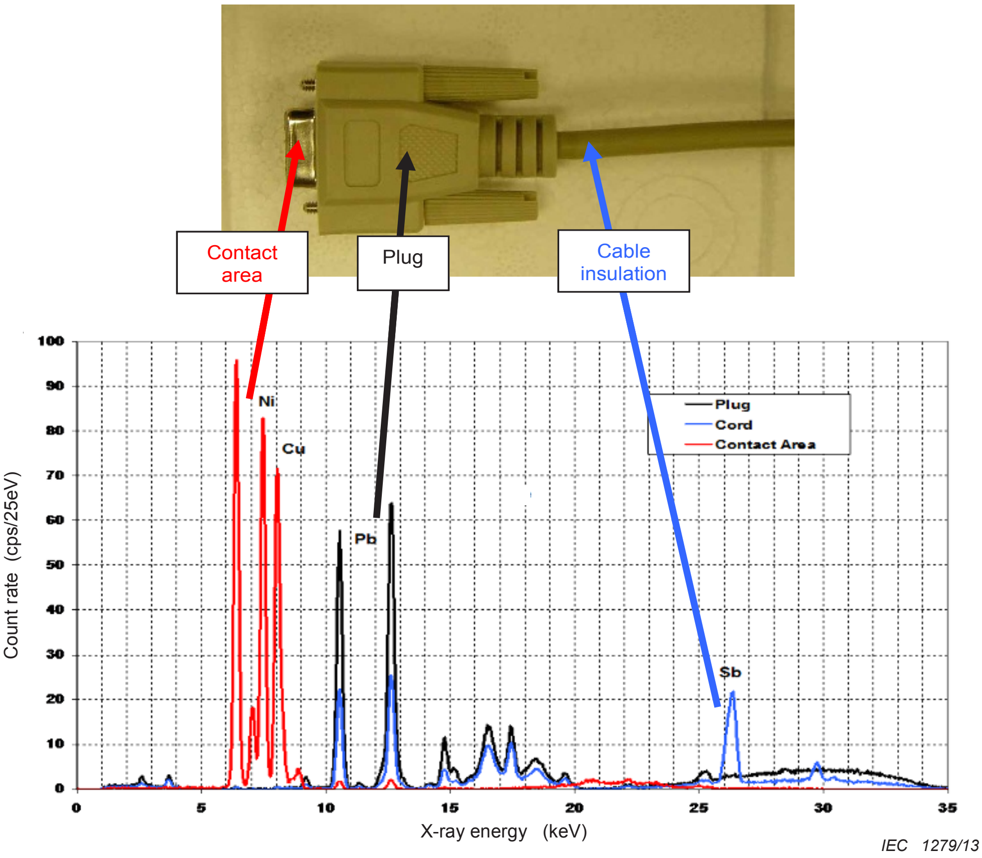
IEC 1278/13

Figure B.1 – AC power cord, X-ray spectra of sampled sections

The three sampling areas were selected, based on the probability of presence of the restricted substance supported by the knowledge of product construction. For example, polymers used for plugs tend to contain high concentrations (in the per cent range) of Pb. The X-ray spectra excited in each "sample" are shown in Figure B.1. Neither in cord insulation nor in plug polymer were any of the certain substances of interest found during the test. There are calcium (Ca), strontium (Sr), zinc (Zn), and antimony (Sb) present in both the cable insulation and plug. The plug also shows the presence of chlorine (Cl), which may suggest PVC as a plug material. However, in neither of these two parts were Pb or Br detected. The connecting pins are made of nickel-plated brass. Up to this point in the sampling and screening process the cable is regarded as being “below level”. The cord therefore needs to be disassembled (in this case destructively) and its parts tested for the presence of Pb on internal solder points of the wires to the connecting pins. The insulation of each individual wire in the cable should also be tested.

B.3.2.2 Serial RS232 cable

This example, illustrated in Figure B.2, shows a printer cable that contains a restricted substance at the level exceeding the allowable limit. In this case the cable insulation contained 2 500 mg/kg of Pb, while the plug contained 7 600 mg/kg Pb. These results, obtained without any disassembly of the product, rendered it non-compliant because of the excessive Pb content, thus effectively eliminating the need for further analysis. For forensic reasons, e.g. to determine the root cause of the contamination in the manufacturing process, it could be advantageous to further sample and analyse the cable.

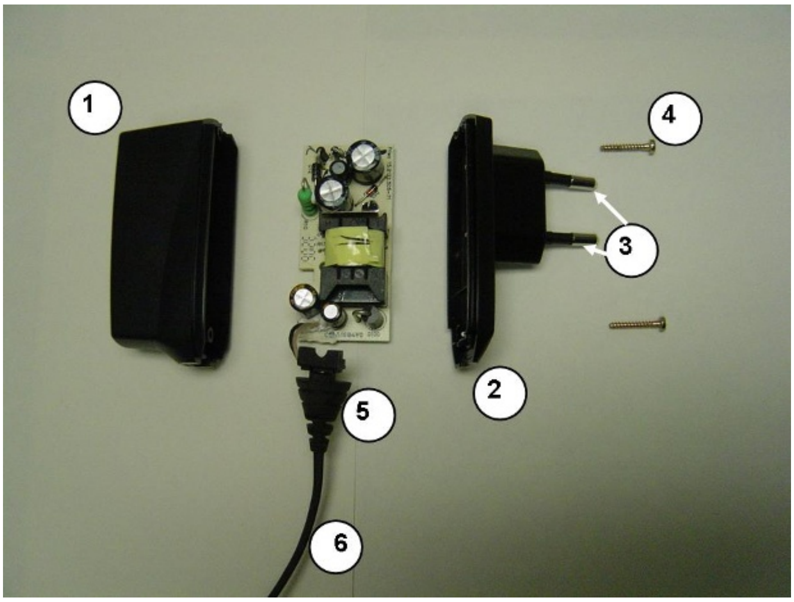


IEC 1279/13

Figure B.2 – RS232 cable and its X-ray spectra

B.3.2.3 Cell phone charger

Figures B.3 and B.4 show a partially disassembled AC charger for a cell phone. As is shown in Table B.2, there are at least ten different areas (parts) available for direct sampling.



IEC 1280/13

Figure B.3 – Cell phone charger shown partially disassembled

Table B.2 – Selection of samples (testing locations) for analysis after visual inspection – Cell phone charger

Sample number	Section identified	Material	Monitored elements	Probability of presence	Select for test?
1	Plastic black cover	Polymer	Pb, Br, Sb[a]	Moderate	Yes
2	Plastic plug base	Polymer	Pb, Br, Sb[a]	Moderate	Yes
3	Contact pins	Metal	Br, Cu, Zn, (Pb)	Low	Yes
4	Screws	Metal	Cr[b], Cd	High	Yes
5	Grommet	Polyurethane rubber (?)	Pb, Br, Sb[a]	Moderate	Yes
6	Cable insulation	Polyurethane rubber (?)	Pb, Br, Sb[a]	Moderate	Yes
7	PWB	Composite	Br	High	Yes
8	Contact tip	Metal	Pb, Cr[b]	Llow	Yes
9	Plug insulation	Polyurethane rubber (?)	Pb, Br, Sb[a]	Moderate	Yes
10	Touch-and-close strap	Synthetic fibre	Cr[b], Sb[a]	?	Yes

[a] Presence of bromine (Br) and antimony (Sb) could indicate the use of a restricted brominated flame retardant.

[b] Presence of chromium (Cr) could indicate the usage of restricted hexavalent chromium (Cr^{6+}).

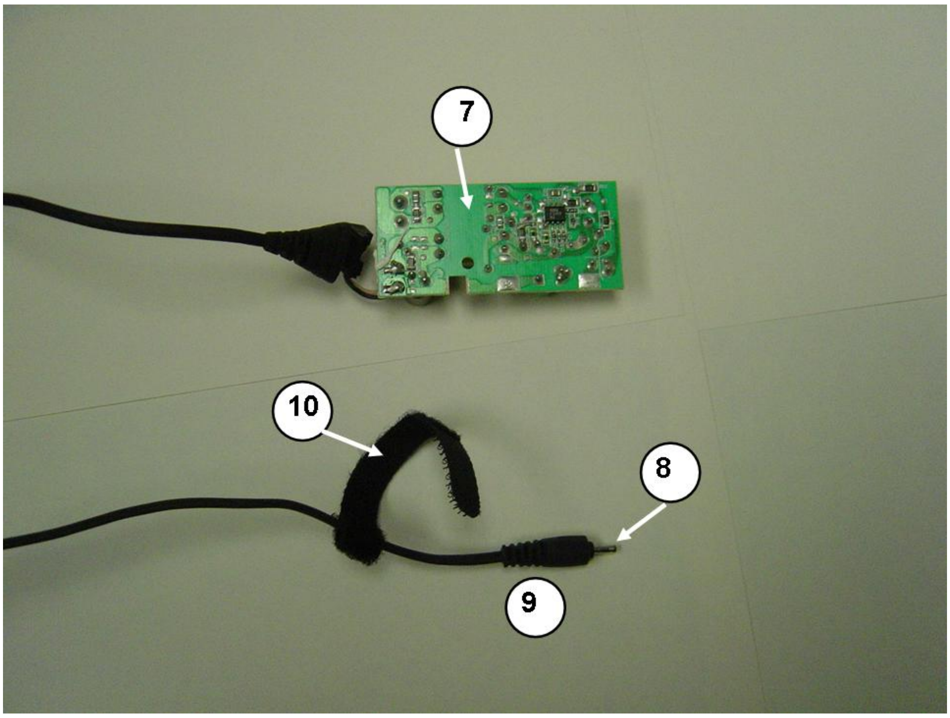


Figure B.4 – PWB and cable of cell phone charger

The case of the cell phone charger is very educational. Firstly, the charger can be sampled and analysed without disassembly. When its case was analysed (sample 1 in Figure B.3) prior to disassembly, it showed, depending on location, between 2 600 mg/kg to 7 000 mg/kg of Br. If analysis was stopped there, it might be concluded that confirmatory analysis of charger case for flame retardants was required. However, only two screws need to be removed to open this device, so the first step of disassembly is very easy. When sample 1 was measured after disassembly it showed no Br content. Sample 7 was then analysed. It is a section of the PWB board with no components, which can therefore be directly analysed with the XRF analyser. Actual analysis of this sample showed 5,5 % Br, which necessitates further analysis for flame retardants. Similarly, the transformer located on the other side of PWB pictured in Figure B.4, showed 8,9 % bromine. This example illustrates how after simple disassembly, it was possible to determine that it is not the plastic case of the charger but the PWB board and transformer that contain Br compounds. Note that even when analysing without disassembly, it was possible to determine elevated levels of Br in the whole product.

B.3.2.4 Testing a printed wiring board

Testing the printed wiring board presents the challenge of analysing a small electronic surface mount component on a PWB populated with a number of other small, but different parts.

Normally, the excitation X-ray beam is collimated within the instrument and this collimation defines the area of the sample which is measured by the system. Figures B.5a and B.5b show the measurement area resulting from two different collimators when attempting to analyse a single solder joint on a PWB. In the case of a large diameter collimator (Figure B.5a) the measurement spot is larger than the sample itself and the results of this measurement will include some content of the solder, the PWB, the metal track on the board and the component itself. In the case of the small diameter collimator (Figure B.5b) the measurement area is small enough that only the solder will contribute to the measurement.

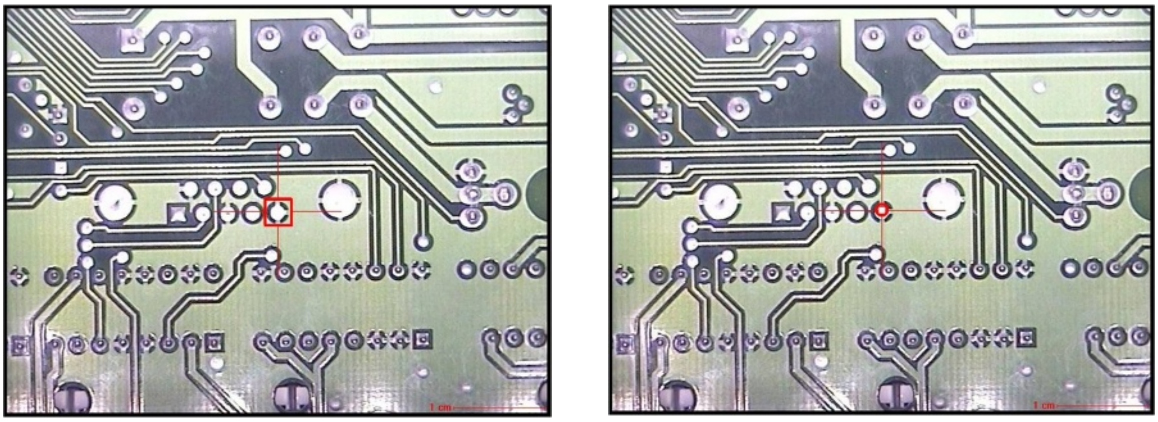


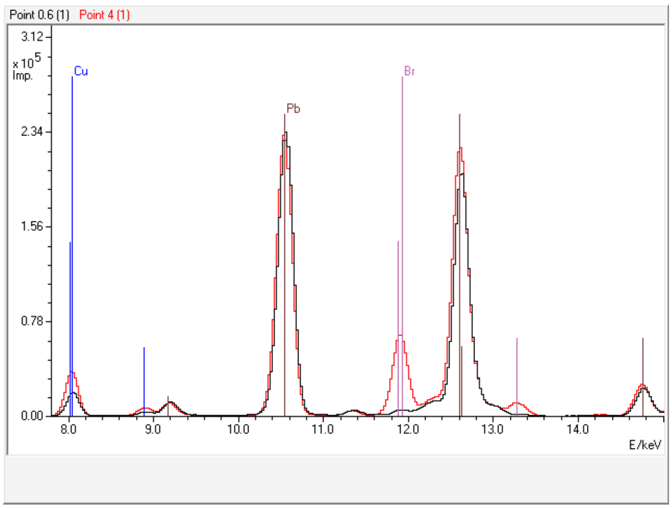
Figure B.5a – Spot from large collimator **Figure B.5b – Spot from small collimator**

IEC 1282/13

Figure B.5 – Analysis of a single solder joint on a PWB

This example illustrates the importance of matching the size of the measuring area of the instrument with the size of the analysed object (sample).

Note that, in the case of large diameter collimation (Figure B.5a), the instrument analysed part of the PWB, which highlights the problem of the influence of sample thickness on the measured results. Since the material of the PWB is less absorbing for X-rays of Pb, for example, than for solder, the PWB thickness will affect the measured results for Pb. Usually, it would take at least 5 mm of PWB material so that its thickness does not affect the assay for Pb. On the other hand, when using small diameter collimator, the whole measuring area is confined to only the solder joint. Since solder is usually much thicker than the so-called "infinite thickness" for Pb and Sn, the measured result for Pb will be accurate. This is further illustrated in Figure B.6 by spectra of the two measurements and their respective elemental results.



Assay	Small spot	Large spot
Cu in %	1,5	2,5
Br in %		3,4
Sn in %	65,2	43,5
Pb in %	31,9	22,6

Small spot: black spectrum
Large spot: red spectrum

IEC 1283/13

Figure B.6 – Spectra and results obtained on printed circuit board with two collimators

B.3.2.5 XRF mapping of elements

Some XRF instruments are equipped with an option which allows the collection of elemental maps. These instruments can capture and record the photographic image of the sample such as for example a PWB, and then create X-ray intensity maps which show the presence and concentration of measured substances (elements) at each scanned point on the sample. By merging the original photographic image of the sample with the intensity map or maps, it is possible to understand the distribution of particular substances (elements) within the sample. Such information, when combined with the structure of the sample, is extremely useful to determine whether the restricted substances are present in an exempt application or not.

In the case of PWBs, the part with the highest probability of presence of restricted substances is the solder. When using substance mapping, the results are shown in Figure B.7. The main concern is whether the Pb found on the board is exempt or not. The bottom part of Figure B.7 shows the combined map of Pb and Sn. Pb is marked in green while Sn is red.

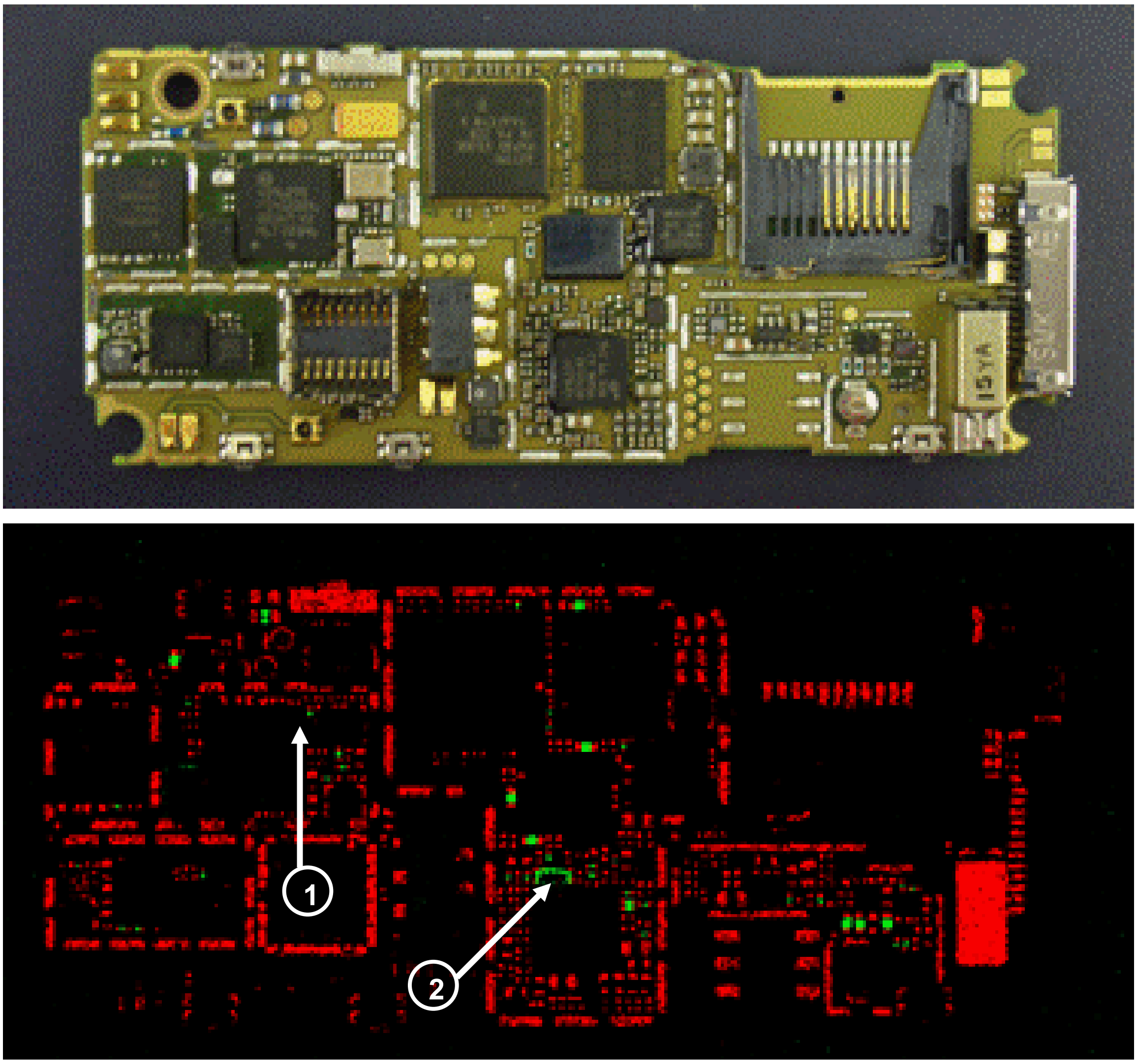
The quantitative results of the analysis of the PWB at spots (1) and (2) are reported in Table B.3. At spot (1), Pb is present with Sn (Pb/(Sn + Pb) = 85 %) which suggests that Pb is contained in a high temperature solder which may be exempt from restrictions. At spot (2), Pb is present not with Sn, but with other elements such as silicon (Si) and titanium (Ti) which, when combined with the photograph, may suggest that Pb is contained in glass or a ceramic. It should be noted that when a restricted substance is identified on the map, it could point to the presence of a restricted use, an exempt use or even both restricted and exempt uses in one component, as is sometimes found with Pb. Further assessment is needed to determine the actual situation.

While very useful, XRF mapping is not a rapid procedure. The maps presented in this example were obtained with an instrument featuring an X-ray beam of 50 kV and diameter of 100 µm. The scan of one side of the board of 100 mm by 50 mm took 1 500 s.

Meaningful results can be obtained with this procedure only if the optimum geometry of measurement can be assured. If the sample cannot be moved into the “focus” of the analyser, the sample may have to be disjointed to perform a meaningful analysis.

Table B.3 – Results of XRF analysis at spots (1) and (2) as shown in Figure B.6

Spot	**Si** %	**Cu** %	**Zn** %	**Sn** %	**Pb** %	**Ti** %	**Fe** %
1	5,2	18,6	43	6,25	35,98	–	–
2	6,5	1,7	3,9	–	–	3,9	1,2

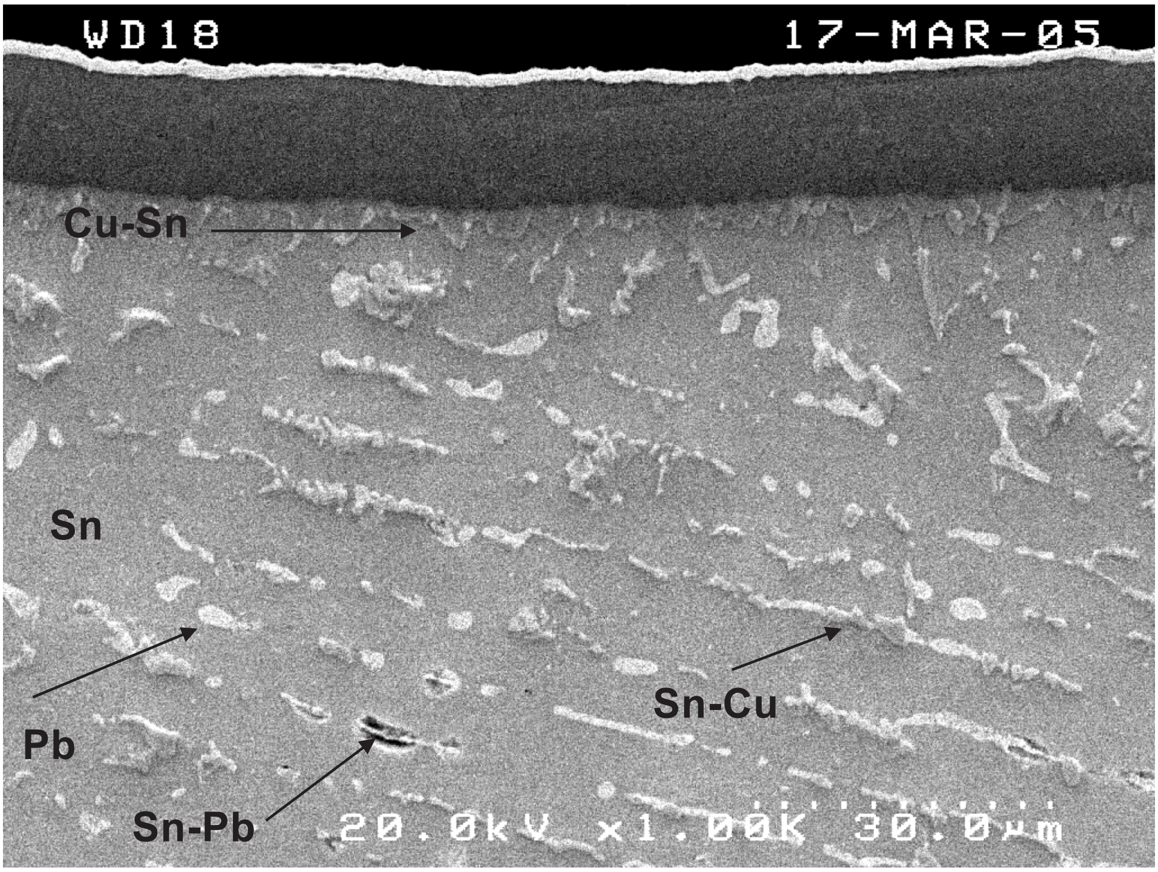


IEC 1284/13

Figure B.7 – Examples of substance mapping on PWBs

NOTE on the SEM-EDX method

This method is mentioned here only for completeness and to draw attention to the existence of this tool. Scanning electron microscopy – energy dispersive XRF (SEM-EDX) makes use of the characteristic X-rays generated by the electron beam in an electron microscope. Since electrons have a very short penetration depth into solid matter, the SEM-EDX is typically a qualitative tool at best. This technique analyses the material only on the very surface of the sample. The principal advantage of SEM-EDX is that it can be used to screen very small (micrometre size) samples and determine the presence of substances in very small volumes. Figure B.8 shows a cross-section of a SAC alloy (tin-silver-copper, Sn-Ag-Cu) solder ball contaminated by Pb solder. The Pb is clustered in small intermetallic domains in a bulk of Sn alloy. SEM-EDX is a very sophisticated method which may only be used by very well trained and experienced personnel, typically an XRF scientist.



IEC 1285/13

Figure B.8 – SEM-EDX image of Pb free solder with small intrusions of Pb (size = 30 μm)

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