DD IEC/PAS 62596:2009

BSI British Standards

Electrotechnical products – Determination of restricted substances – Sampling procedure – Guidelines

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British Standards

National foreword

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A review of this Draft for Development will be carried out not later than three years after its publication.

Notification of the start of the review period, with a request for the submission of comments from users of this Draft for Development, will be made in an announcement in the appropriate issue of Update Standards. According to the replies received, the responsible BSI Committee will judge whether the validity of the PAS should be extended for a further three years or what other action should be taken and pass their comments on to the relevant international committee.

Observations which it is felt should receive attention before the official call for comments will be welcomed. These should be sent to the Secretary of the responsible BSI Technical Committee at British Standards House, 389 Chiswick High Road, London W4 4AL.

The UK participation in its preparation was entrusted to Technical Committee GEL/111, Electrotechnical environment committee.

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

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Electrotechnical products – Determination of restricted substances – Sampling procedure – Guidelines

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

ELECTROTECHNICAL PRODUCTS – DETERMINATION OF RESTRICTED SUBSTANCES – SAMPLING PROCEDURE – GUIDELINES

FOREWORD

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IEC-PAS 62596 has been processed by IEC technical committee 111: Environmental standardization for electrotechnical products and systems.

The text of this PAS is based on the following document:	This PAS was approved for publication by the P-members of the committee concerned as indicated in the following document
Draft PAS	**Report on voting**
111/112/PAS	111/126/RVD

Following publication of this PAS, which is a pre-standard publication, the technical committee or subcommittee concerned may transform it into an International Standard.

This PAS shall remain valid for an initial maximum period of 3 years starting from the publication date. The validity may be extended for a single 3-year period, following which it shall be revised to become another type of normative document, or shall be withdrawn.

INTRODUCTION

In the electrotechnical industry, much emphasis has been placed on minimizing the environmental burden of its products. Waste handling, recycling, chemicals and energy consumption are covered by regulations. Specifically, the use of materials containing lead (Pb), mercury (Hg), cadmium (Cd) and hexavalent chromium (Cr VI), as well as two types of brominated flame retardants (polybrominated biphenyls, PBBs, and polybrominated diphenyl ethers, PBDEs) in electrotechnical equipment is restricted in current and proposed regional legislation.

To demonstrate compliance with these requirements, it may be necessary to analyse electrotechnical products for a variety of reasons:

- to supplement supply chain material declarations (companies may choose to test products directly to determine compliance);
- companies may require their suppliers to perform analysis to support material declarations;
- companies may perform "spot checks" of their suppliers to assess compliance
- enforcement authorities may perform testing as part of their market surveillance activities.

IEC 62321 already provides test methods for the determination of six regulated substances in electrotechnical products. However, the preparatory steps before the analysis are critically important in obtaining accurate, reproducible results. Prior to this PAS, there was virtually no guidance or consensus as to how electrotechnical products should be sampled.

The purpose of this PAS is primarily to complement IEC 62321 by providing agreed guidelines on how electrotechnical products, assemblies and components should be sampled to determine the levels of restricted substances present.

Please note sampling and analytical testing is not the only way to obtain relevant information on the levels of substances in an electrotechnical product or component. Experience and knowledge of the materials used could remove the need for sampling and testing; for example, flame retardants are never used in metals. Furthermore, analytical test reports and material declarations received can be used to demonstrate that the levels of restricted substances are below the required limits.

ELECTROTECHNICAL PRODUCTS – DETERMINATION OF RESTRICTED SUBSTANCES – SAMPLING PROCEDURE – GUIDELINES

1 Scope

This PAS provides general sampling guidelines and strategies of sampling for electrotechnical products, electronic assemblies, electronic components. In order to obtain samples that can be used for analytical testing to determine the levels of restricted substances as described in the test methods of IEC 62321. Restrictions for substances will vary between geographic regions and from time to time. This PAS describes a generic process for the sampling of any substance which could be restricted.

This PAS does not provide:

- Full guidance on each and every product that could be classified as electrotechnical equipment. Since there is a huge variety of electrotechnical components, with various structures and processes, along with the continuous innovations in the industry, it is unrealistic to attempt to provide procedures for the disjointment of every type of component.
- Analysis procedures to measure the levels of restricted substances. This is covered by other standards (for example the future IEC 62321), which are referred to as the "test standard" in this PAS.
- Guidelines for assessment of compliance.
- Guidance regarding other routes to gather additional information on restricted substances in a product, although the information collected has relevance to the sampling strategies in this PAS.
- Sampling procedures for packaging and packaging materials.
- Safe disassembly and mechanical disjointment instructions related to electrotechnical products (e.g. Hg containing switches) and the recycling industry (e.g. how to handle CRTs or the safe removal of batteries).

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 62321, *Electrotechnical products – Determination of levels of six restricted substances (lead, mercury, cadmium, hexavalent chromium, polybrominated biphenyls, polybrominated diphenyl ethers)*

3 Terms, definitions and abbreviations

3.1 Terms and definitions

For the purpose of this document, the following terms and definitions apply.

NOTE As this PAS is closely related to IEC 62321, terms and definitions from that standard have not been duplicated here.

3.1.1
electronic assembly

group of components, at least one of which is an electronic device, but in which individual parts may be replaced without damage to the assembly

[Definition H.2.5.9, IEC 60730-1:1999]

EXAMPLE Group of components mounted on a printed wiring board.

3.1.2
electronic components

electrical or electronic devices that are not subject to disassembly without destruction or impairment of design use. They are sometimes called electronic parts, or piece parts

[Definition 3.1.5, IEC 62239:2008]

EXAMPLE Resistors, capacitors, diodes, integrated circuits, hybrids, application specific integrated circuits, wound components and relays.

3.1.3
composite testing

testing two or more materials as a single sample that could be mechanically disjointed if necessary

3.1.3
electronics

electronic assembly and/or electronic component and/or field replaceable unit

3.1.4
field replaceable unit
FRU

part, component or subassembly that is easily removed (mechanically disjointed) using ordinary tools

NOTE "Easily removed" consists of using ordinary tools to perform such functions as screwing or disconnecting, and only without irreversibly destroying the unit.

[Definition 3.7, IEC Guide 114:2005]

3.1.5
disassembly

process of taking apart an electrotechnical product; possibly using simple hand tools such as a screwdriver, pliers and wrenches

NOTE A disassembled unit can, in theory, be re-assembled and be made operational.

3.1.6
disjointment

process of, in principle, separating the materials by mechanical actions such as: unscrewing, cutting, grinding, scratching and abrasive processes

NOTE A disjointed part or assembly cannot be re-assembled into an operational unit.

3.1.7
homogeneous material

material that cannot be mechanically disjoined into different materials

NOTE 1 The term "homogeneous" means "of uniform composition throughout". Examples of "homogeneous materials" are individual types of: plastics, ceramics, glass, metals, alloys, paper, board, resins and coatings.

NOTE 2 The term "mechanically disjointed" means that the materials can, in principle, be separated by mechanical actions such as: unscrewing, cutting, crushing, grinding and abrasive processes

3.1.8
infinite thickness
critical thickness
thickness of the specimen which, if increased, yields no increase in intensity of X-rays measured from the sample due to their absorption by the sample matrix. This thickness varies with the energy of X-rays

3.1.9
sampling
process of selecting a representative part or section of a product (any electrotechnical device) for the purpose of determining by means of analysis the concentrations of restricted substances present. Sampling can be carried out by selecting a section on an object or by disassembly and disjointment

NOTE Representative part or section could be the lead-frame of an integrated circuit or the plastic jacket of an electrical wire.

3.1.10
screening
analytical procedure to determine the presence or absence of substances or compounds in the representative part or section of a product, relative to the value or values accepted as the criterion for this decision

NOTE If the screening method produces values that are not conclusive, then additional analysis or other follow-up actions may be necessary to make a final presence/absence decision

3.2 Abbreviations

AAS	Atomic absorption spectroscopy
ABS	Acrylonitrile butadiene styrene
AFS	Atomic fluorescence spectroscopy
ASTM	American Society for Testing and Materials
BGA	Ball grid array (electronic component)
CV-AAS	Cold vapour atomic absorption spectrometry
CRT	Cathode ray rube (television)
DIP	Dual-in-line package (electronic component)
DVD	Digital versatile disc
ED XRF	Energy dispersive X-ray fluorescence
EDX	Energy dispersive X-ray spectroscopy
FRU	Field replaceable unit
GC-MS	Gas chromatography – mass spectrometry
GLP	Good laboratory practice
HPLC-UV	High-performance liquid chromatography – Ultraviolet
IC	Integrated circuit
ICP-OES	Inductively coupled plasma optical emission spectrometry
ICP-MS	Inductively coupled plasma mass spectrometry
MDL	Minimum detection level
LCD	Liquid crystal display
MQCA	Minimal quantity for chemical analysis
OEM	Original equipment manufacturer
PAS	Publicly Available Specification
PBB	Polybrominated biphenyl

PBDE	Polybrominated diphenyl ether
PC	Polycarbonate
PDA	Personal digital assistant
PE-HD	High-density polyethylene
PS-HI	High-impact polystyrene
PVC	Polyvinyl chloride
PWB	Printed wiring board
QA	Quality assurance
SEM-EDX	Scanning electron microscopy – energy dispersive XRF
SMD	Surface mounted device
TFT	Thin film transistor
TV	Television
WD XRF	Wavelength dispersive X-ray fluorescence
XRF	X-ray fluorescence

4 Introduction to sampling

4.1 Introductory remark

Obtaining a sample (i.e. sampling) is the first step in analyzing electrotechnical products for the presence of restricted substances. The strategy and process of sampling is often as important as the analytical measurement itself. Hence an effective sampling strategy requires a clear understanding of the electrotechnical product, reasons for the analysis, and the requirements that are to be met.

Sampling and testing for restricted substances is performed for many reasons including:

- business-to-business for commercial release (e.g. contractual agreement between the OEM and component manufacturer).
- compliance with regulatory limits.
- forensic/impact assessment (why the product does not satisfy contractual or legal requirements, when did this happen, and how many products are affected?).

4.2 Requirements and concerns for restricted substances

While many governments, industry partners and other stakeholders have their own requirements, it is not the intention of this PAS to discuss fully all of these differences. However, awareness of different limits for regulated substances is an important step in preparing the sampling strategy. This clause highlights the main areas of concern regarding the requirements for restricted substances.

1. Restricted substances: Not all geographic regions or industrial partners restrict the same substances. For example, some regions have chosen to restrict the use of only a few specific PBDE compounds, while others have a broader restriction regarding this class of flame-retardants. When sampling a product, component etc. it is critical to keep in mind what are the applicable legal requirements.
2. Allowable limits for restricted substances: Generally speaking, the allowable levels of most restricted substances are below 1 000 mg/kg. Some geographic regions and industrial partners have limits below 1 000 mg/kg. For some product types, limits for restricted substance are above 1 000 mg/kg, e.g. lead in copper and aluminum alloys.
3. Application of the allowable level: The manner in which the allowable level of a restricted substance is applied to an electrotechnical product determines the sampling strategy and how the test results are interpreted. Many geographic regions apply their allowable limits

to "homogeneous materials". In this PAS an “homogeneous material” (3.1.8) is defined as a material that cannot be mechanically disjointed into different materials. However, the interpretation of "homogeneous material" is not consistent across the different regions. Some regions have defined the smallest possible amount of material to be homogeneous (e.g. bonding wires in semiconductor chips only several microns thick), while other regions try to apply a more pragmatic approach.

4. Applicable exemptions: Some types of electrotechnical products are exempt from restricted substances requirements. These exemptions may be based on different rationales including the scope of the restrictions (e.g. for military purposes), the application of the material (e.g high melting temperature solder), size of the sample, or the electrical properties of the product.

4.3 Complexity of electrotechnical products and related challenges

The complex characteristics of electrotechnical products are another important consideration when preparing a sampling strategy. These characteristics have a bearing on the practical execution of sampling and analysis. The following elements are identified as relevant to analysis and sampling:

1. Miniaturization: Miniaturization is one of the key trends in the electrotechnical industry. This implies that more functionality is provided within a smaller volume. More and more components and materials are used per cm² of printed wiring board (PWB) every year. Taking samples for measurement from these small amounts of material is difficult. For example, the size of surface mounted devices (SMDs) is too small for regular tools to further disjoint or separate and the quantity of the remaining sample is often too small after disjointment to satisfy the requirements of adequate analysis.
2. Number of homogeneous materials: Many components have complex structures and are constructed of multiple layers of different materials. In a typical case, one single component has more than 10 to 20 material layers, whereas many electrotechnical products or assemblies contain hundreds or thousands of components. This means one electrotechnical product can have more than 1 000 to more than 10 000 homogeneous materials. Often homogeneous materials adhere too tightly together for a clean separation in a practical manner (see Figure 15). Experience has shown that the composition often changes due to molecular diffusion between materials (e.g. the composition of a plating is affected by a base material containing Pb). Similarly, current electrotechnical products are made of many components and parts. A typical TV or laptop computer for example, contains thousands of parts/components. Hence the design database for an OEM may include several tens of thousands of components. In Clause 5 this point is further illustrated in the disassembly of a mobile phone.
3. "Invisible" substances: Another complicating factor in sampling and analysis is that generally restricted substances are not visibly apparent. A component containing a restricted substance looks and performs in an identical manner to one that is "clean". The presence or absence of restricted substances can vary from lot to lot in the manufacturing process without any readily observable clues. While there are some visible indications (e.g. a yellow coating on steel products suggests the presence of hexavalent chromium) as to the presence of restricted substances, visual detection is not practical.
4. Batch–to-batch variations: Most product assembly manufacturers use commodity components from several suppliers simultaneously, e.g. cables, resistors and capacitors. Commodity components are mixed during production, because technically they are fully interchangeable as long as they fit the umbrella specification. However in most cases they are not chemically identical. Furthermore experience has shown that base materials can be changed by commodity manufacturers (e.g. in times of shortage) which leads to a change in the chemical composition as well. Notifications of these changes do not always occur if the component still meets its technical specification.
5. Depth of the supply chain: Producing electronic components/parts involves a complex supply chain. Relatively simple products such as an external cable, can utilize supply chains at least seven tiers deep. The supply chain for a more complex component like an LCD screen or IC is considerably deeper.

These characteristics of the electrotechnical industry show that the management of restricted substances, along with sampling and analysis, is not straightforward. The size and number of components, and complexity of the supply chain make it challenging to fully grasp the locations of restricted substances in an electrotechnical product. The prospect of implementing homogeneous material level sampling and testing at the upper regions of the supply chain (towards finished products) is not practical for complex products.

4.4 Strategies for sampling

While different sampling approaches may be utilized as appropriate for the broad range of electrotechnical products, it is possible to describe a generic procedure that will be applicable in the majority of cases. This is shown in Figure 1.

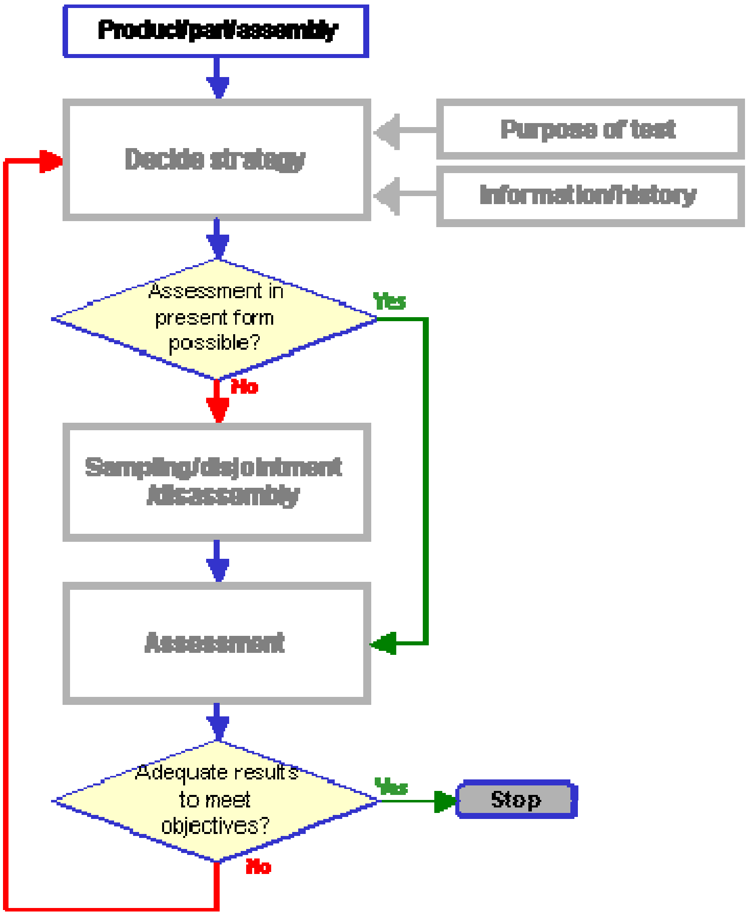


Figure 1 – Generic iterative procedure for sampling

The process depicted in Figure 1 can have several iterative loops including:

- 1st iteration: partial disassembly (see 5.2)
- 2nd iteration: complete disassembly (see 5.3)
- 3rd iteration: partial disjointment (see 5.4).
- 4th – nth iteration: complete disjointment (see 5.5)

These iterative steps are described further in Clause 5.

Development of the sampling strategy for a particular electrotechnical product/part/assembly begins with an information gathering stage. Some basic questions to be considered include:

- What is the complexity of the product/part/assembly and is it practical to consider sampling and testing at the homogeneous material level?

- Which substances are restricted?
- What are the allowable limits for these restricted substances?
- Are there appropriate exemptions for the restricted substance?
- Is a bill of materials available for the components/assemblies/materials in the product?
- Are specifications/drawings of the components available?
- What is the depth of the supply chain for the components and materials in this product?
- Are material declarations for this product available?
- Is there any previous experience evaluating this product or similar products that could be helpful?
- Are there any published probability of presence matrices for the materials or parts used in this product?
- Was any screening (e.g. XRF) previously performed on this product or similar products that could be helpful?
- Is there any information regarding the manufacturing process of materials/components (metal making or IC production) used in this product or similar products that could be helpful?
- Are there any perceived process controls present at the component or material suppliers (e.g. level of trust in the manufacturer)
- Is there any history of concern with the component or material supplier?

The answers to these questions and other characteristics will influence the sampling strategy. The organization's position in the supply chain will determine what extent of sampling is appropriate. Release for production of products/components etc. requires a more in-depth sampling strategy than an occasional verification check on specifications. In order to optimize costs and efficiency the desired outcome of the testing needs to be understood. As previously stated, it is often impractical to sample and test all components/materials. An organization is left to determine the optimum balance of effort/costs against effectiveness of the sampling strategy. Some considerations to minimize sampling/testing efforts and costs are listed below:

- homogeneous materials with a low probability of containing restricted substances (see Annex B);
- applicable exemptions for restricted substances;
- material declarations;
- historical test data;
- composite sampling and testing (see 5.6 and Annex C);
- minimum sample size necessary to run analytical tests and the number of samples necessary to determine whether or not it's practical to test.

The sampling strategy will depend very much on the ultimate objective of analysis. One strategy (perhaps used by enforcement authorities) could be an analysis to verify if the product contains at least one restricted substance at the level exceeding the allowable limit. This approach involves gradual, selective sampling, targeting deliberately those parts of the product that are either known to, or are likely to, contain restricted substances. Each sampling phase could be followed by analysis. If the results show no restricted substances above the allowable limit, a further stage of sampling and analysis could be performed. Once the test results exceed the allowable limit for at least one restricted substance in any part of the product, it is deemed non-compliant and no further sampling and analysis is necessary. Annex B provides a list of components where there is currently a probability of the presence of one or more of six restricted substances.

Another strategy may be to prove total compliance of the product, as far as possible down to the homogeneous material level. This approach would be typical of the product or component manufacturer. Samples would be prepared from each individual material or component. As the objective is to cover all components and materials in a product/assembly, other routes may be used to gather information on a product level. In the downstream supply chain process documentation and/or analysis reports may exist that would reduce the effort required in sampling and analysis.

Once the objective of the analysis has been defined, an assessment is performed as to the feasibility of testing (e.g. is the sample mass/size/volume sufficient?). Further sampling and disjointment may be necessary, in which a choice can be made to either completely disjoint or only select materials with a high probability of containing restricted substances. Table B.1 can be used to assist in the identification of these components and materials.

If testing is appropriate, the relevant testing procedure should be followed. Where restricted substances are present in the product/part there may be an applicable exemption (some examples are given in Table B.1).

Following the flowchart in Figure 1 is an iterative process, retrieving samples at an ever deeper level. How far this process is pursued will be dependent on the objective of the sampling strategy. After the screening steps further analytical testing may be undertaken.

5 Sampling

5.1 Introductory remark

This PAS only provides general sampling guidelines, which are intended to form the basis of the sampling strategy appropriate to the electrotechnical product.

Whenever possible, sampling should be performed by stages of minimal disassembly and disjointment. Each stage is followed by an assessment of its effectiveness (see the flowchart in Figure 1), typically by screening analysis. Depending on the results of the assessment and objectives of the analysis, further disassembly and sampling may be required, especially for verification analysis of the product’s components/materials. This approach to sampling and assessment offers the least expensive, fastest and the most efficient means of analysis, especially when undertaken on the finished product.

The enormous number of types and diversity of electrotechnical products make it impractical to provide detailed sampling strategies for each product. Instead, sampling procedures covering four levels of disjointment and sampling are described for two products:

- cell phone;
- printed wiring board (PWB).

The cell phone is a compact and complex product containing large number of small components. Therefore, sampling procedure of such product should be a good example of how sampling strategies can be developed for virtually any other electrotechnical product.

Annex A provides generic sampling flowcharts based on Figure 1 for some characteristic electrotechnical products: DVD player, CRT tube, LCD TV Set, PDA/phone and a desk fan, along with two components, a thick film resistor and SMD potentiometer. Annex D lists some commonly used disassembly and disjointment tools, and Annex E describes the use of XRF screening techniques in sampling.

NOTE During disjointment, several tools are used. In restricted substances assessment is highly recommended to ensure that the tools are free of the restricted substances to avoid possible contamination.

5.2 Partial disassembly

Sampling of the complete product is the first step in the iterative sampling strategy. It infers that representative parts of the product can be analysed without disassembly or disjointment;

that is in a non-destructive manner. Even if we decide to disassemble the product to gain access to its subassemblies, we may still be able to test them non-destructively.

However, "complete product" is a relative term. For example, an AC power cord is a complete product for its manufacturer, but it may be just a component in a television set. Complete products may be evaluated without disassembly if they have a very simple construction, or if the locations of expected restricted substances are known and it is possible to test for them without disassembly. Examples of such products are power cords, printer or other peripheral cables, equipment housings, etc. Please note even a seemingly simple product such as a power cord may be composed of between 10 to 20 different homogeneous materials.

Before sampling, the following questions need to be asked:

1. Based on knowledge of the product, can any sections/parts be identified that are likely to contain restricted substances (see Annex B)?
2. Is it practical to analyse any of these sections/parts without its disassembly?
3. Can the section/part selected be regarded as homogeneous material for the purpose of the analysis?
4. Is the section/part selected for analysis representative?
5. Does the sample selected fulfil the criteria of minimum mass, area, thickness or volume required by the chosen analytical methods?

If the answer to all of these questions is "yes" then the sampling strategy can begin with disassembly and analysis of the complete product.

5.2.1 Example 1: Cell phone type A – Disassembly without tools

Figure 2 – Cell phone with battery charger and camera lens cap

This cell phone offers limited opportunities for analysis as a complete product. The charger includes at least six individual areas that are made of different materials, and may be screened without disassembly. The phone back cover may be removed and phone battery taken out. As the back cover is metallized on the outside it should be analysed (screening) on both sides. This partial disassembly of the cell phone is the limit of disassembly without the use of tools, as shown in Figure 3.

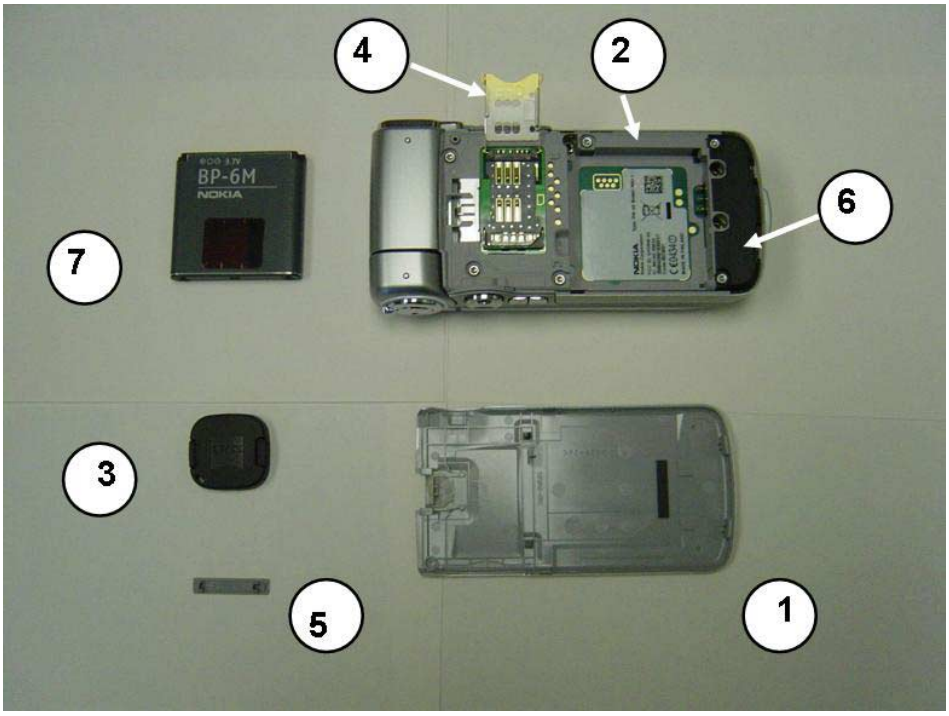


Figure 3 – Cell phone with battery and back cover removed

Table 1 – Possible restricted or screening substances from a cell phone

Sample number	Component/assembly	Materials	Probability of presence[a]	Elements related to restricted substances[b]	Select for analysis
1	Plastic back cover	Polymer	Moderate	Pb, Br	See 4.3
2	Plastic housing of the phone	Polymer	Moderate	Pb, Br	See 4.3
3	Plastic cover for lens	Polymer	Moderate	Pb	See 4.3
4	Metal clip for SIM card	Polymer	Low	Cr, Cd	See 4.3
5	ComPort cover	Polymer	Moderate	Pb, Br	See 4.3
6	Plastic part in body	Polymer	Moderate	Pb, Br	See 4.3
7	Battery	Complex	High ?	(Cd, Pb, Hg)	See 4.3

The samples marked in Figure 3 can be screened directly.

Subclause 4.3 shows considerations for further actions.

[a] Probability of presence indicates the likelihood of finding the listed restricted substances.

[b] Presence of Br (bromine) could indicate the usage of a restricted brominated flame retardant.

5.2.2 Example 2: Cell phone type B – Partial disassembly

This subclause describes the disassembly process of another cell phone using only simple tools such as screwdrivers. Please note after disassembly the parts of the phone can be easily put back together and the phone will be functional.

Firstly, the major parts and components can be separated as shown in Figure 4. Here the parts are the TFT display, keypad, bottom housing, main PWB, housing /frame etc as listed in Table 2.

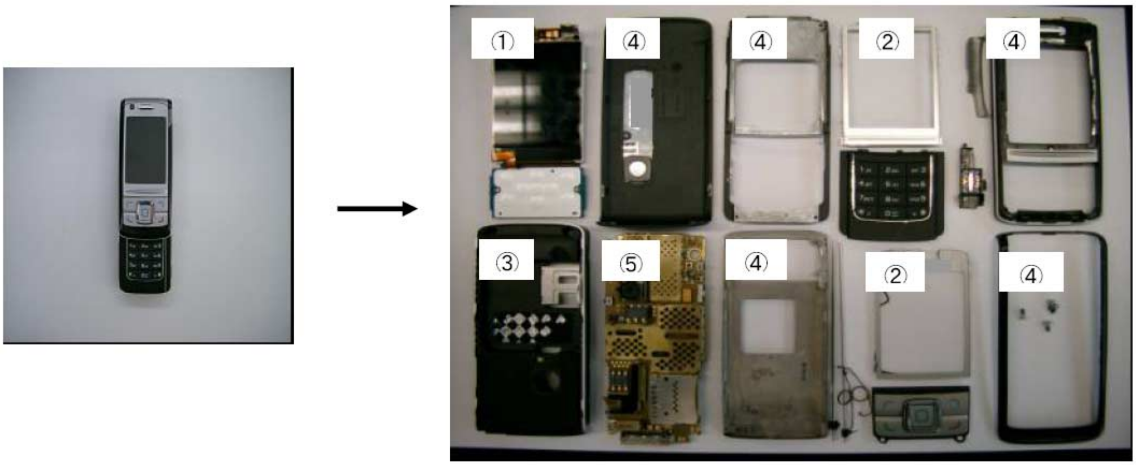


Figure 4 – Partial disassembly of a cell phone (type B) into its major components

Table 2 – Possible restricted substances in major components of the cell phone

Sample number	Component/assembly	Materials	Probability of presence[a]	Elements related to restricted substances[b]	Select for analysis
1	TFT display	Polymer/glass/metal	Moderate	Pb	After further disassembly or disjointment
2	Key pad	Polymer	High	Cd, Hg	Needs further disassembly
3	Bottom housing	Polymer	High	Cd, Br	Yes
4	Other housing/frame	Polymer	High	Cd, Br	Yes
5	Main PWBs	Many different (fibre glass, copper)	High	Pb, Br, Hg	After further disassembly or disjointment

[a] Probability of presence indicates the likelihood of finding the listed restricted substances.

[b] Presence of Br (bromine) could indicate the usage of a restricted brominated flame retardant

Not all components of the cell phone separated after this partial disassembly are suitable for direct analysis. The TFT display and PWB are complex structures, containing many different materials. Their construction and size make it difficult to analyse the materials without further disassembly/disjointment steps, regardless of the analytical method used. However, if the first step in the analysis is XRF screening, the elements present relating to restricted substances within these complex assemblies can still be detected. A decision would then have to be made on the need for confirmatory testing. Samples 3 and 4 seem to be a homogeneous materials that may be tested directly with XRF. Sample 2, keyboard assembly, may be screened at this stage, but it may still undergo further disassembly.

5.3 Complete disassembly

As illustrated in the previous clauses, very few parts of the cell phone are simple enough in construction and composition for direct, meaningful analysis. Almost all parts require further disassembly, which may render them nonfunctional.

This subclause describes the complete disassembly of the cell phone type B. Figures 5 to 7. show the disassembly process of the key pad (Figure 5), bottom housing (Figure 6) and the other housing/frame materials (Figure 7). These parts of the phone can be easily disassembled by using the usual tools (see Annex D) to obtain almost single materials such as plastics or metals. Again, sample selection can be based on the presence of restricted substance guidance in Table B.1.

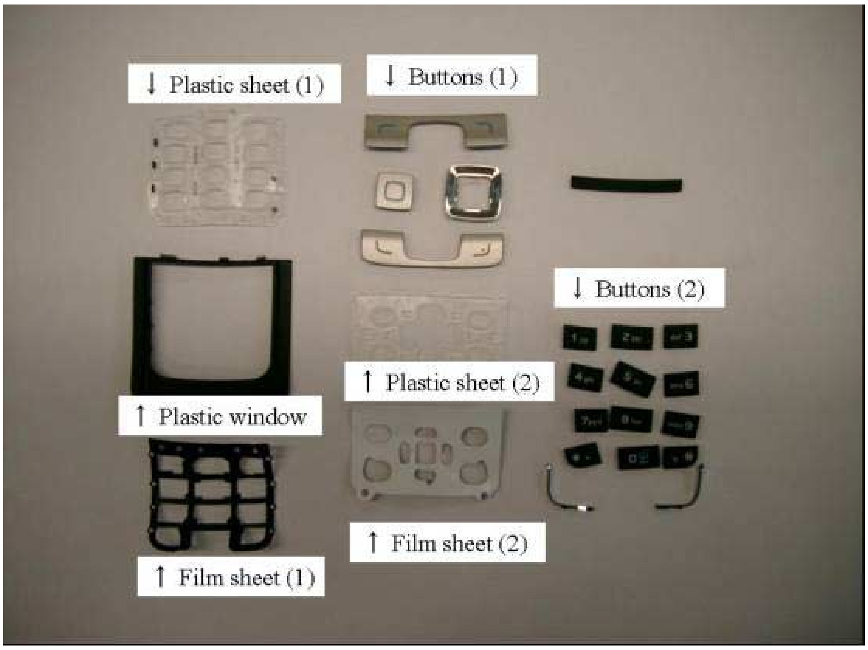


Figure 5 – Complete disassembly of the key pad

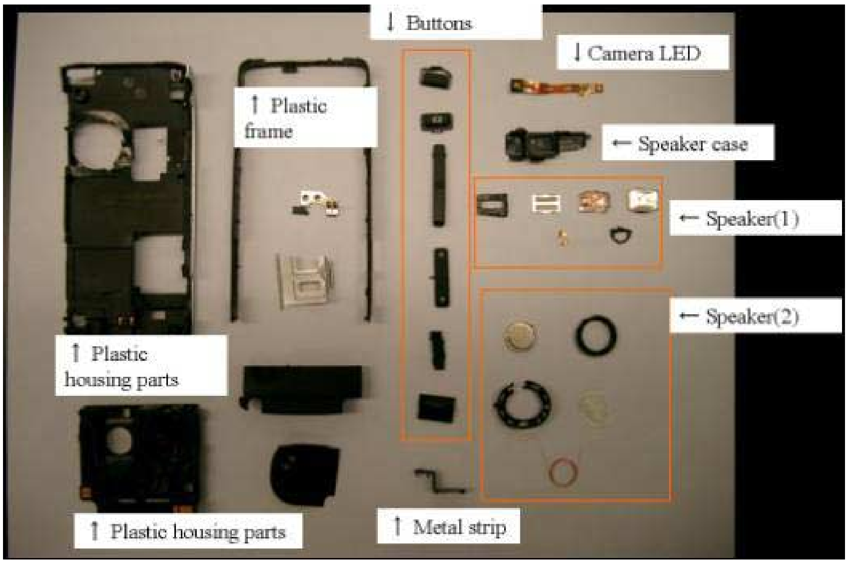


Figure 6 – Complete disassembly of the bottom housing

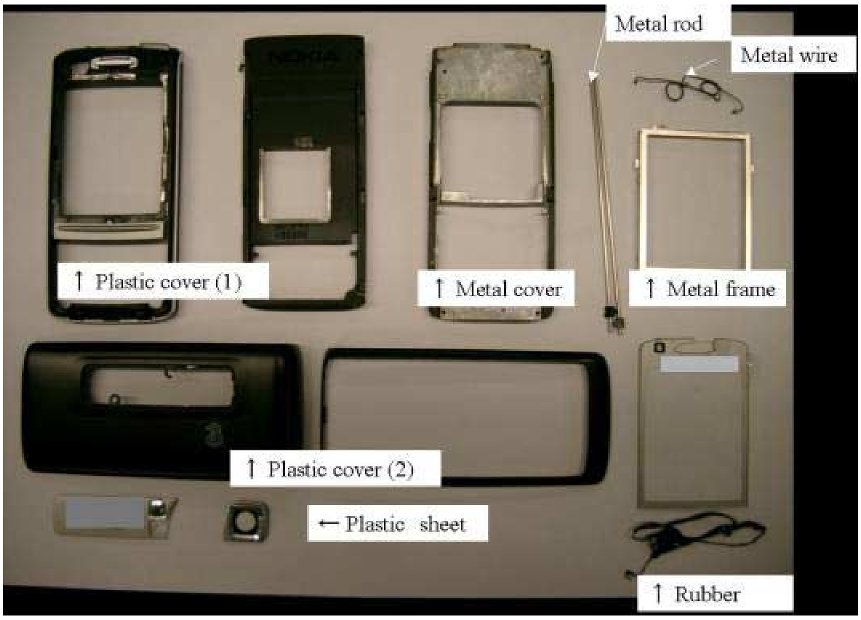


Figure 7 – Complete disassembly of the other housing/frame

5.4 Partial disjointment

5.4.1 Introductory remark

Frequently, the detailed analysis of a complete electrotechnical product for restricted substances requires disassembled internal subassemblies and components to be further separated by disjointment However, it is often impractical for such products to be disjointed completely into their constituent homogeneous materials. Hence sampling of subassemblies and components should focus on those parts with a high probability of containing restricted substances (see Table B.1). When considering the locations from which samples can be obtained for analysis it is important to understand the structure and materials of these items and the possible locations of restricted substances within them.

Note that after disjointment it may be impossible to reassemble the separated parts.

Subassemblies and components form discrete parts within complete electrotechnical products such as cell phones, televisions, personal computers, refrigerators, etc, of which typical examples are

- populated printed wiring boards (PWBs),
- resistors,
- capacitors,
- semiconductor packages (ICs),
- transformers,
- molded connectors,
- heat sinks,
- connecting cables, etc.

Figure 8 shows the partial disjointment of the TFT subassembly of that the type B cell phone (previously disassembled in 5.2 and 5.3). Separation of certain components of the display such as casing, insulation and filter could only be achieved destructively. Similarly, partial disjointment of the main PWB of this cell phone into its components is shown in Figure 9. The tools required for this disjointment were screwdrivers, a soldering iron and small knife (see Annex D for other tools).

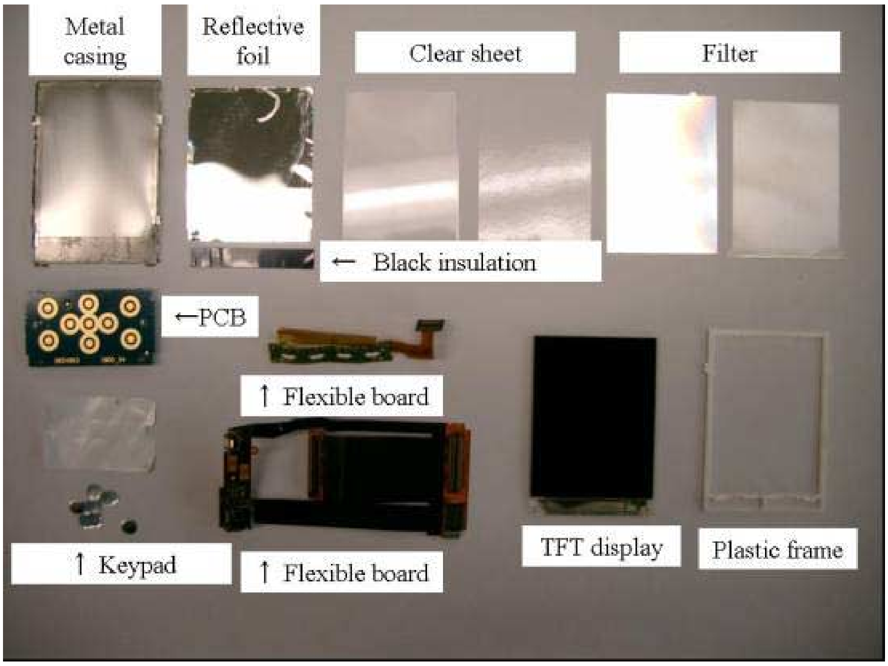


Figure 8 – Components of the TFT display of the cell phone after partial disjointment

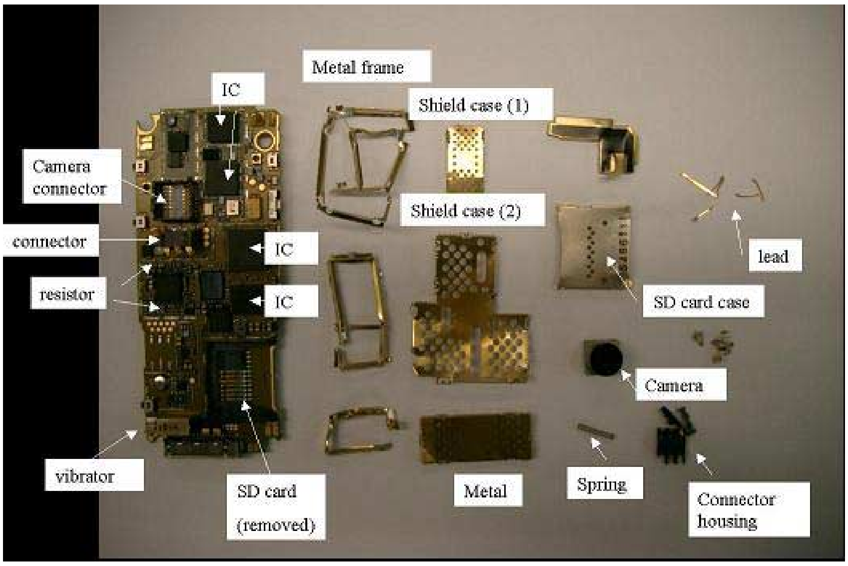


Figure 9 – Components of the main PWB of the cell phone after partial disjointment

5.5 Complete disjointment

5.5.1 Introductory remark

The objective of complete disjointment is the complete separation of all components into their homogeneous materials. Generally this is not practical, such that “complete disjointment” becomes the separation of components into their homogeneous materials only, as far as the available tools and techniques permit.

When dealing with complex components, before attempting their disjointment into homogeneous materials, professional experience, skill, and non-destructive analysis can help to determine their internal structure and materials. This can be achieved by:

- using one's professional knowledge;
- access to technical documentation;
- analysis by means of X-ray fluorescence (XRF) screening.

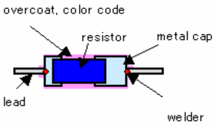
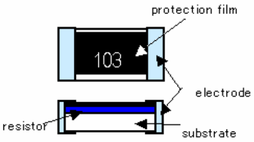
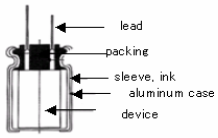
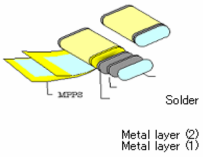
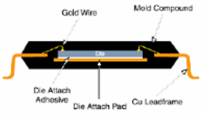
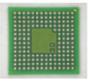
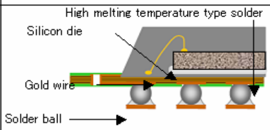
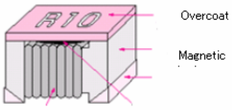
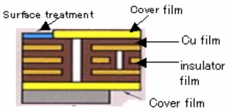
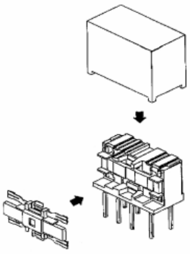
The XRF method is very helpful in identifying the materials present in advance of disjointment, especially when the structure of the sample is unknown. As always one should exercise caution when interpreting the results.

5.5.2 Typical examples of disjointment at the component level

Table 3 shows examples of the structure and materials of typical components used in electrotechnical products. These components may be obtained from the supplier or after their disassembly from the PWB. The analytical sample of a particular material can be sampled from the component using appropriate tools (see Annex D) while at the same time focusing on those materials with the highest probabilty of containing restricted substances (see Table B.1).

The examples in Table 3 reflect the current and historical use of components (for example, BGA integrated circuits were not in use 15 years ago). Clearly, even apparently simple components, such as through hole resistors, contain about ten different materials. This underlines the need for careful planning and the development of an individual sampling strategy for each component.

Table 3 – Examples of disjointment for typical small electronic components

Assemblies and components*		Structure	Sampling point	Possible hazardous materials	Exemption part	Sampling procedure for IEC 62321 Test Method Analysis	Sampling Considerations or Limitations
Resistor	IMD-type 		lead	Pb		Cutting lead	# samples to get adequate sample mass; sample prep time
			overcoat	Pb, Cr6+		Scraping	# samples to get adequate sample mass; sample prep time
			color code (ink)	Pb, Cr6+		No practical mechanical disjointment method	cross-contamination, # of samples to get adequate sample size, sample prep time
			resistor	Pb	Resistor: Pb (glass	No practical mechanical disjointment method	cross-contamination, # of samples to get adequate sample size, sample prep time
	SMD-type 		electrode	Pb		No practical mechanical disjointment method	cross-contamination, # of samples to get adequate sample size, sample prep time
			Protection film	Pb	Protection film: Pb (	No practical mechanical disjointment method	# samples to get adequate sample mass; sample prep time
Capacitor	Electrolyte 		lead	Pb		cutting lead	# samples to get adequate sample mass; sample prep time
			sleeve (PVC)	Pb		Scraping	# samples to get adequate sample mass; sample prep time
			ink	Pb, Cr6+		No practical mechanical disjointment method	cross-contamination, # of samples to get adequate sample size, sample prep time
	Chip-type 		solder	Pb		No practical mechanical disjointment method	cross-contamination, # of samples to get adequate sample size, sample prep time
			ink	Pb, Cr6+		No practical mechanical disjointment method	cross-contamination, # of samples to get adequate sample size, sample prep time
IC Chips	leadframe-type 		lead-frame	Pb		Cutting Lead	cross-contamination, # of samples to get adequate sample size, sample prep time
			ink	Pb, Cr6+		No practical mechanical disjointment method	cross-contamination, # of samples to get adequate sample size, sample prep time
	BGA type 		Solder Ball	Pb	Solder: Pb (high melting temperature type)	No practical mechanical disjointment method	cross-contamination, # of samples to get adequate sample size, sample prep time
Molded Connectors			Housing	PBB/ PBDE		Cutting or scraping	# samples to get adequate sample mass; sample prep time
			Fasting Nail	Pb		No practical mechanical disjointment method	cross-contamination, # of samples to get adequate sample size, sample prep time
			Contact	Pb		Cutting or scraping	# samples to get adequate sample mass; sample prep time
Coil			Electrode	Pb	Magnetic body: Pb (ceramics)	Cutting or scraping	# samples to get adequate sample mass; sample prep time
			Overcoat	Pb, Cr6+, Cd		Scraping	# samples to get adequate sample mass; sample prep time
Flexible board			Surface treatment	Pb		Scraping	# samples to get adequate sample mass; sample prep time
Relays			Case cover	Pb, Cr6+, Cd		Cutting	# samples to get adequate sample mass; sample prep time
			Armature	Pb		Scraping	# samples to get adequate sample mass; sample prep time
			Fixed contact (	Pb		Cutting or scraping	# samples to get adequate sample mass; sample prep time
			Terminal	Pb		Cutting or scraping	# samples to get adequate sample mass; sample prep time
			Body	Pb, Cr6+, Cd		Scraping	# samples to get adequate sample mass; sample prep time
					Fixed contact: Cd		
					Movable contaact: Cd		

5.5.3 Examples of disjointment at the base materials level – Disjointment of integrated circuit (IC) chips

This subclause shows examples of the disjointment of integrated circuit (IC) chips (in which Pb is likely to be present in the tin (Sn) plated termination coating).

Example 1 – Disjointment of lead frame component

Step 1 – Metal leads removal from IC package

Leads were removed from the package by cutting off the metal parts which extended beyond the rest of the package. The leads removal procedure took approximately 2 h for 36 individual ICs and resulted in 1,2 g of metal sample.

If proper care is taken during cutting of the leads, and the samples are not taken too close to the IC body, then the lead sample collected should not be contaminated with other materials. The preparation time and number of samples needed to obtain enough mass for the analysis of metals make this lead frame disjointment procedure possible on an industry-wide basis Figure 10b shows the lead sample after disjointment.

Step 2 – Package disjointment

Figure 10c shows the IC package after disjointment. This is assuming that the disjointment could be accomplished in a manner that does not cause cross-contamination with other material layers.

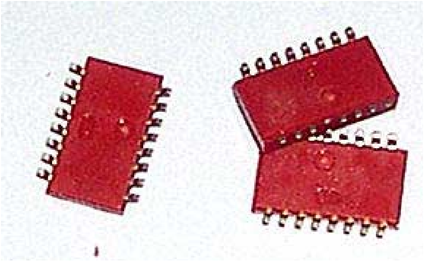


Figure 10a – Lead frame package (IC)

Figure 10b – Metal leads after disjointment

Figure 10c – IC package after disjointment

Figure 10 – Disjointment of lead frame component

Example 2 – Disjointment of BGA component

A typical BGA component package is made up of several different material layers: substrate, underfill, silicon die, C4 solder bumps, solder paste and solder balls. Figure 11 shows an example of BGA package prior to disjointment.

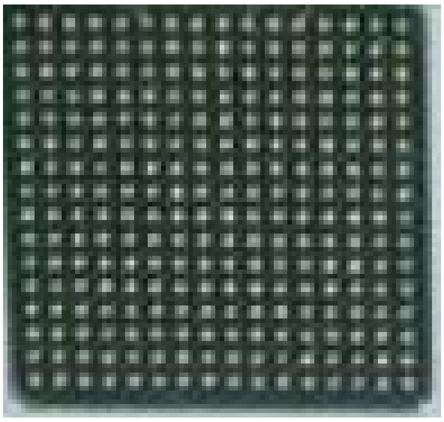


Figure 11 – BGA package prior to disjointment

Step 1 – Solder ball removal from BGA package

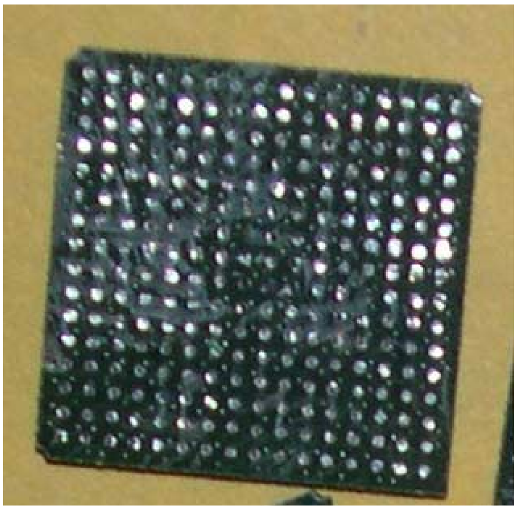


Figure 12 – BGA package disjointed by the hand removal procedure

The solder balls seen in Figures 11 and 12 were disjointed from the BGA package using a hand removal procedure during using a sharp utensil, to shave or cut the solder balls away from the substrate. While care was taken not to include any substrate, flux or solder paste with the detached balls of solder, there was inconsistency in the solder ball collection using this method. Some solder balls could only be cut away, while other solder balls simply "popped" out of the package instead of being cut or scraped away. As can be seen in Figure 12 the balls of solder did not disjoint from the package in a manner that guaranteed only solder ball material was collected.

Figure 13 – Solder ball material collected from BGA using a hand removal procedure

Using the hand removal procedure required approximately 2 h to remove the balls from 15 BGAs. The mass of solder ball material collected was only 1,5 g and contained some flux, solder paste and substrate materials. Thus, about 45 BGAs and about 6 h work would be required to collect enough sample mass for analysis.

Figure 13 shows the collected solder ball material. However, the sample collected using this hand removal procedure could not be considered as a "homogeneous" solder ball material as it contained other materials (such as flux, fragments of substrate) collected during the disjointment procedure.

Since the hand removal procedure was not able to provide a homogeneous solder ball sample, another solder ball removal technique was needed, namely the solder ball shear procedure JEDEC JESD22-B117. Although this JEDEC standard was not developed for removing solder balls for material analysis, it is an industry procedure used by some companies in their Quality Assurance/Quality Control (QA/QC) labs.

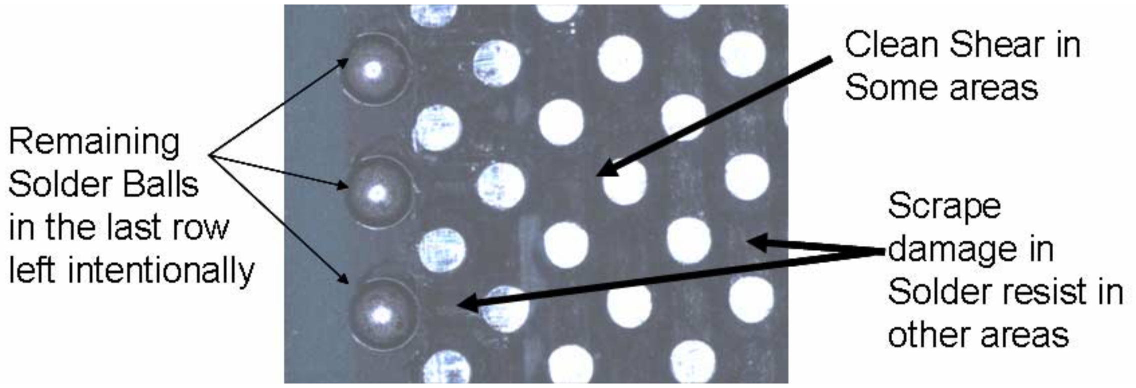


Figure 14 – BGA solder ball removal using the ball shear procedure

Once again, to obtain enough sample mass for analysis, approximately 45 BGAs would be required, which means approximately 6 h for sample preparation.

As can be seen in Figure 14 above, the ball shear method also created contamination through the scraping of solder resist along with the solder ball material in some areas. Nevertheless, the amount of contamination using the ball shear procedure was significantly less than the amount of contamination using the hand removal procedure.

5.6 Considerations of sampling and disjointment

5.6.1 Introductory remark

In the previous subclause, disassembly and disjointment were discussed from the high level down to the detailed level. When arriving at this detailed level some difficulties become apparent. In theory, sampling to the homogeneous level could continue down to the nano-scale. In practice however, such sampling is at best very difficult or unacceptably time consuming. Test method detection limits are dependent on sample masses, sizes, or volumes. Testing below these masses, sizes, or volumes can render the test method unsuitable to confirm the presence or absence of a given substance below the allowable level.

5.6.2 Sample size required

IEC 62321 testing methodology has defined the following minimum sample sizes for analysis:

- Clause 6 – Analysis by XRF
  – Strongly instrument and geometry dependent, but in the mm range in thickness.
- Clause 7 – Mercury
  – 0,5 g for a < 5 mg/kg MDL
- Clauses 8 to 10 – Lead and cadmium (run together)

– – 0,5 g for < 50 mg/kg minimum detection limit (MDL)

– – 1 g for < 5 mg/kg MDL

- Annex A – PBBs and PBDEs
  – 100 mg for <100 mg/kg MDL per congener group
- Annexes B and C – Hexavalent chromium
  – 2,5 g for <15 mg/kg MDL for polymers and electronics
  – 50 cm2 for <0,02 mg/kg MDL for chromate coatings on metals

NOTE Certain materials have matrices which may fall outside these parameters. Samples with high concentrations will require dilutions which can increase MDLs.

For small components, it is often impractical to obtain enough sample mass to conduct analytical testing. For illustrative purposes, Table 4 summarizes how weights in a typical industry lead frame material declaration report and the IEC 62321 test method minimum sample sizes can be used to calculate a minimum number of samples that would be required to collect sufficient sample mass for analytical testing. The technical assumptions for the calculations are also shown in the table.

Table 4 – Minimum number of lead frame samples required for analytical testing

Material layer	Weight of material per unit /mg	Number of units required for Pb/Hg/Cd analysis[a]	Number of units required for Cr^{6+} analysis	Number of units required for PBB and PBDE analysis
Bond wire [b,c,d,e,f]	1,7	365	NA	NA
Die attach [b,c,d,e,f]	0,7	893	NA	1 786
Lead frame [b,c,d,e,f]	42,0	15	74	NA
Lead frame plating [b,c,d,e,f]	0,6	1 008	5 040	NA
Mould compound [b,c,d,e,f]	52,9	12	NA	24
Si die [b,c,d,e,f]	6,0	104	NA	NA

Assumptions

[a] The same test method is used for the 3 elements and the concentrations can be obtained from one sample. This is used for illustrative purposes to demonstrate sample sizes. However, per Table B.1, Hg and Cd would not be expected in lead frame component.

If more than one substance is required for analysis, then the number of units required is the sum of the number of samples required for the individual substance.

[b] Minimum sample weights are specified in IEC 62321

[c] 20 % volume (weight) loss due to sample preparation

[d] It is technically possible to separate each material layer without contamination

[e] 100 % of each material can be collected from each layer

[f] Not applicable to substances that are not expected to be present in the material layer (see Table B.1).

Table 4 shows that thoretically 15 samples are required for Pb testing of the lead frame material layer. However, as discussed in 5.4.2, the actual number of samples required to obtain sufficient mass from the lead frame layer in a lab environment was closer to 30-35 samples. This was because the entire lead frame layer could not be collected from each unit as the mechanical disjointment process had to prevent cross-contamination of other material layers. (There is no need to analyse for hexavalent chromium as the legs of an IC are at very low risk of containing hexavalent chromium.) Therefore, the numbers presented in Table 4 should be viewed as "best case" or "theoretical minimum".

Based on the requirements specified above, to run all six of the restricted substance tests per IEC 62321 requires a minimum sample mass of 4,5 g. However, depending on the material, there may be as much as a 20 % loss during the sample preparation techniques (see Clause 5 of IEC 62631) before testing. Therefore, 4,5/(1-0,2) = 5,6 g could be the actual minimum sample size to collect from the parts or components to analyse for all six restricted substances. Flame retardants are not typically used for metal material layers, so for metal analysis only, the minimum sample size could be reduced to 3,5/(1-0,2) = 4,4 g.

As the number of samples required for testing increases, so does the risk that the component population will not be identical. The components may come from different lots, and/or their materials may differ, even if the part number and/or performance remain the same. Consequently the analytical result could be misleading (diluting high levels of a restricted substance in only a few of the samples) or inconclusive.

Hence sampling large numbers of small components, such as those shown in Table 3 and the lead frame example in Table 4, can make analytical testing and disjointment very difficult. For example, can the material collected for analysis be considered a homogeneous material? Are the samples used identical to each other? What criteria are used to assess the uniformity of the component's lot?

5.6.3 Sample size v. detection limit

There is an inverse relationship between sample size and detection limit. As the amount of material available to analyse decreases, the detection limit of a given substance increases.

The disjointment process becomes progressively more difficult as the size of the components decreases. However, size is not always the limiting factor. For example it is more difficult to disjoint a 10 mm^3 IC than a 4 mm^3 varistor. Due to the difficulty of disjointing small parts, it could be helpful to specify a minimum size beyond which disjointment is no longer required. Components smaller than this minimum size may be pulverized and the resultant material analysed as a homogeneous material. In this case the detection limit issues discussed in Table 5 become important. It should be noted that setting a minimum size at a certain level does not solve some basic issues related to sampling and analytical testing.

In cases where the disjointment is continued to the smallest homogeneous material, the amount of material which is required for analysis is determined by the minimum sample quantity required for a particular analytical method, which is related to the sample type, sample preparation technique (see Clause 5 of IEC 62321) and the analytical method. For example, when testing for Pb in polymers by ICP-MS using dry ashing as the sample preparation procedure a 0,2 g sample is required for a 0,1 mg/kg reporting limit. In general every such combination of matrix, sample preparation procedure and analytical method will have a minimum sample size required to achieve a particular reporting limit. As the size of the sample is reduced the reporting limit increases until at 0,02 g sample size the reporting limit has increased to 50 mg/kg. It is important to understand the relationship of sample size and reporting limit for the matrix, sample preparation method and measurement technique being used to determine the restricted substance.

5.6.4 Composite testing of disjointable samples

When testable material is limited and difficult to obtain, it is particularly important to be aware of the probability of presence of a given restricted substance to avoid unnecessary testing (see Table B.1). Ignoring these considerations could lead to false positives. For example, metals do not need to be tested for flame retardants. However when collecting leads or balls of ICs, a small amount of moulding compound could remain attached to the metal, providing a source of flame retardant. Such cross-contamination is virtually unavoidable and needs to be taken into consideration.

Therefore, sampling that results in a test specimen consisting of multiple homogeneous materials ("a composite") may test positive for one or more restricted substances. When results are expressed in mg/kg of the mixed composite sample they may be found to be under the allowable limit. This mechanism is illustrated in the hypothetical case of Table 5. When expressing the concentration of the total composite material this could be below the tolerated level (e.g. 1 000 mg/kg), while the results on the basis of the homogeneous material may well exceed the tolerated level. An example of such a situation is depicted in Table 2, where material A contains a significant level of Pb which is diluted in the overall Pb result of the mixed composite sample. Further considerations for composite samples are covered in Annex C.

Table 5 – Levels of a restricted substance in a composite sample

Material	**Mass contribution** %	**Pb concentration** mg/kg	**Pb concentration in the composite sample** mg/kg
Homogeneous material A	25	1 200	300
Homogeneous material B	25	600	150
Homogeneous material C	20	0	0
Homogeneous material D	15	0	0
Homogeneous material E	15	40	6
Total for the composite sample	100		456

As this PAS shows, sampling and analysis of restricted substances at the homogeneous material level is often difficult and a proper understanding of the basis of interpretation, particularly when dealing with composite samples, is crucial.

5.6.5 Non-uniform "homogeneous materials"

There is an inherent conflict within the definition of homogeneous material for those parts or components that cannot be mechanically disjointed, but do not have a uniform chemical composition. As mentioned above, it is sometimes permissible to homogenize such samples and treat them as one material. However much more knowledge of the component's structure and materials is needed.

When the component is prepared (e.g. ground into a powder) and tested as a single sample the analaytical result will be the average level of the restricted substance over the entire mass of the sample tested. Difficulties arise when both restricted and non-restricted forms of the same substance co-exist in the sample. This is shown in Figure 15 for a resistor that cannot be mechanically disjointed, but contains Pb in restricted (Pb-based plating, upper cross-section) and exempt (PbO glass, lower cross-section) applications. The composite sample from this non-disjointable component cannot distinguish between the different sources of Pb. Hence it is not possible to determine the Pb concentrations in each of the homogeneous materials by testing the resistor as a composite sample. This holds true in essence for several sizes of components that cannot be mechanically disjointed.

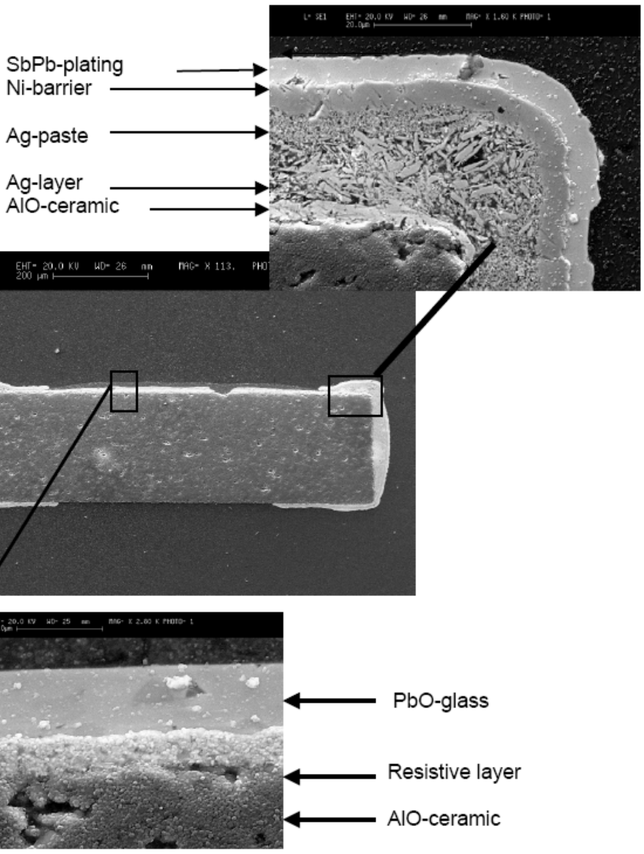


Figure 15 – Cross-section of a 900 µm wide lead oxide-based resistor (SMD)

Where several substance restrictions apply to a single component at the homogeneous material level, perhaps based on the specific application, the individual homogeneous materials should be analysed separately, which can be challenging in practice.

Strategies need to be developed to address these challenges. Especially as electrotechnical products continue both to shrink and evolve in complexity, while the number of restricted substances grows and the allowable limits fall. Screening techniques can be and are used to gather more information on the restricted substances, and it may be possible to analyse materials in-situ as a cross-section, per Figure 15, as discussed in Annex E. However it remains difficult to distinguish between exempt and restricted forms of the same substance.

5.6.6 Determination of sampling position of homogeneous materials

Although commercial “homogeneous” materials share the same physical or chemical properties, their compositions are not always completely uniform. If the sample mass required for testing is not more than half of the available mass of the material, sampling should be made at several locations. For example, sampling positions should be selected from different areas, including at least one geometric centre and the ends of two diagonals.

6 Conclusions and recommendations

The strategy and process of sampling are critical steps during the analysis of restricted substances in electrotechnical products. The quality of analytical results and specifically their representativeness of the analysed object/product, are directly affected by the sampling procedure. Selection of the sampling strategy depends on the substance(s) to be determined, their allowable limits, the basis by which the restrictions are applied and possible exemptions. Other important factors are the complex structure of electrotechnical products, the small size of their components, diverse material content, batch-to-batch variations, depth of the supply chain and the analytical test method. These considerations are discussed in depth in Clause 4, concluding that the selection of the sampling strategy is driven by the objectives of the analysis, which should be kept in mind.

A generic, iterative procedure for sampling. is described in Figure 1. Given its iterative nature, there is no single, universal approach. The entity performing the sampling should determine which and how many iterative levels of disassembly and disjointment are necessary to meet the objectives of the analysis. Each iteration is followed by an assessment of the extent to which these objectives have been met. The many examples in Clause 5, Annexes A and E show that the proper sampling strategy and intelligent use of analytical techniques can substantially reduce the amount of work required.

This PAS illustrates that sampling for the analysis of restricted substances in electrotechnical products is often complicated and even impractical when evaluating small complex parts and components. The difficulty stems from the restriction of substances at the ”homogeneous material” level, which therefore becomes a key element of sampling. Problems arise when evaluating a sample that is not uniform in composition throughout, yet for practical reasons cannot be mechanically disjointed (e.g. the resistor in Figure 15). This scenario illustrates the deficiency of the definition of homogeneous material. At the point where no further mechanical disjointment is possible, analytical testing alone cannot determine whether the sample complies with the restricted substance requirements at the homogeneous material level.

It is often impractical to disjoint and test all homogeneous materials in a product or component. Even when it is physically possible, constraints such as time, resources and the amount of material available maybe insufficient for a meaningful analysis. To alleviate this difficulty sampling strategies should prudently utilize information from material declarations and knowledge of the probability of presence for the restricted substance in a specific material type (see Table B.1). Subclause 5.6 highlights the importance of understanding the limitations of intelligent testing strategies (e.g. testing of composite materials) in order to be able to make effective decisions. These strategies should also be aligned with the objectives of the analysis.

Annex A
(informative)

Examples of procedures for sampling and disjointment

A.1 Introductory remark

This Annex provides a detailed generic procedure for sampling and disjointment along with several worked examples:

- Example 1 – Sampling of a DVD player
- Example 2 – Sampling of a CRT tube
- Example 3 – Sampling of a LCD TV set
- Example 4 – Sampling of a PDA/phone
- Example 5 – Sampling of a desk fan
- Components – Example 1 – Thick film resistor
- Components – Example 2 – SMD potentiometer

The following points should be considered in conjunction with the flowcharts:

a) Analysis may be being undertaken for a number of reasons
  - Analysis of a specific material or location (e.g. a manufacturer carrying out an internal audit, a distributor checking areas of the electrotechnical product which have a high probablity of presence for restricted substances, an enforcement authority pursuing particular intelligence).
  - Screening as part of a incoming goods inspection.
  - The initial stage of a comprehensive review of the whole product.

b) See Table B.1 for the probability of presence for restricted substances

c) Exemptions for some substances in certain applications only apply under particular regional legislation.

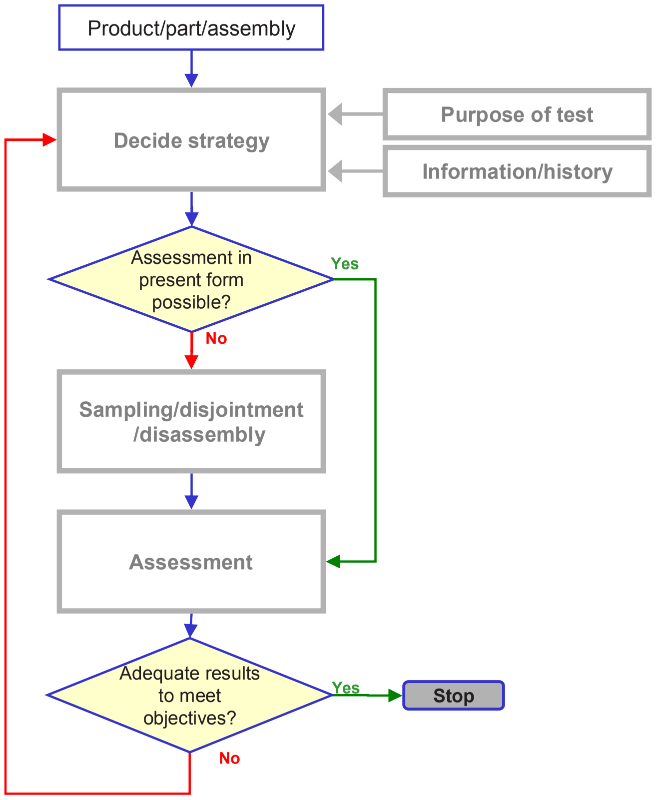


Figure A.1 – Example of methodology for sampling and disjointment

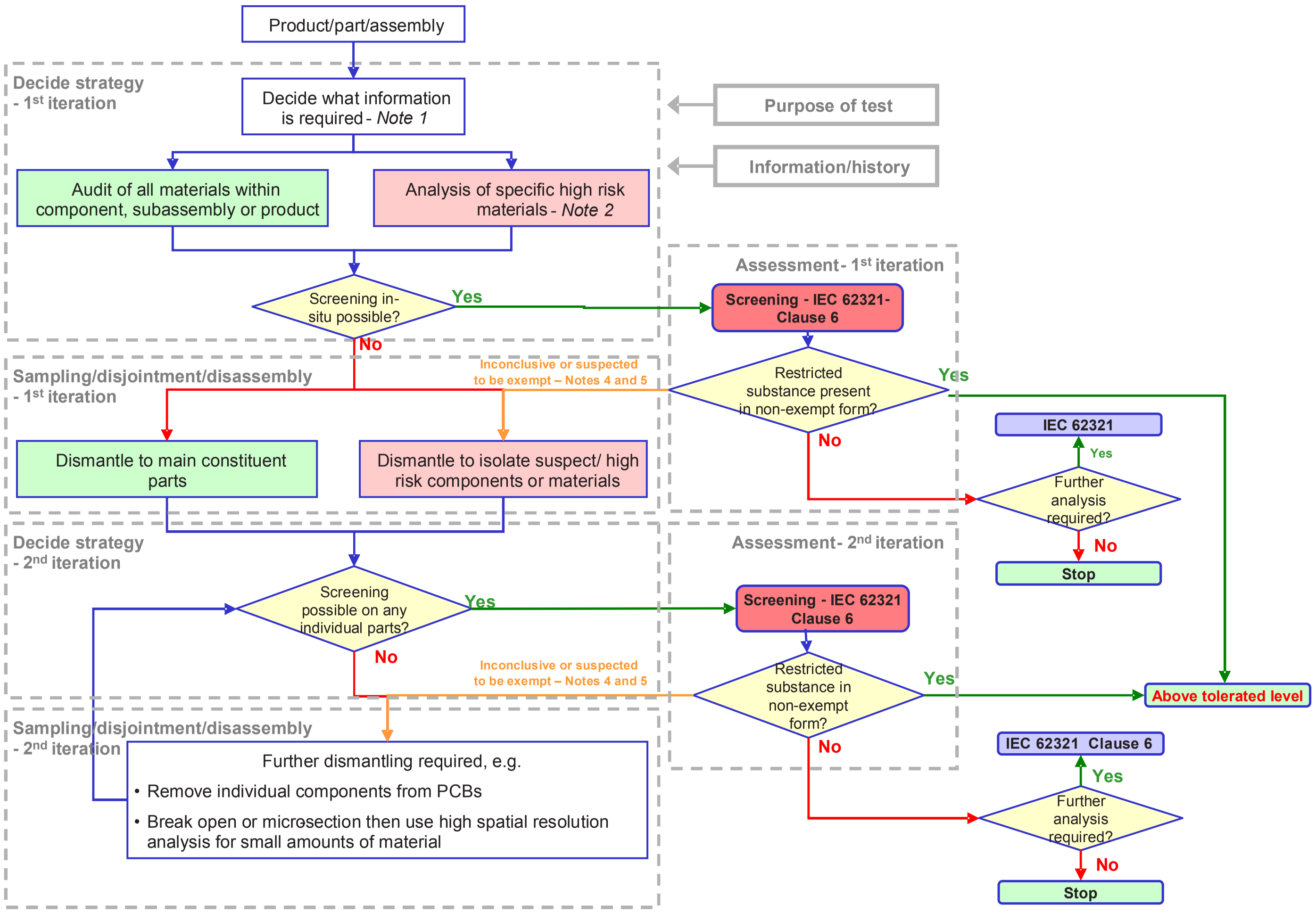


Figure A.2 – Methodology for sampling and disjointment

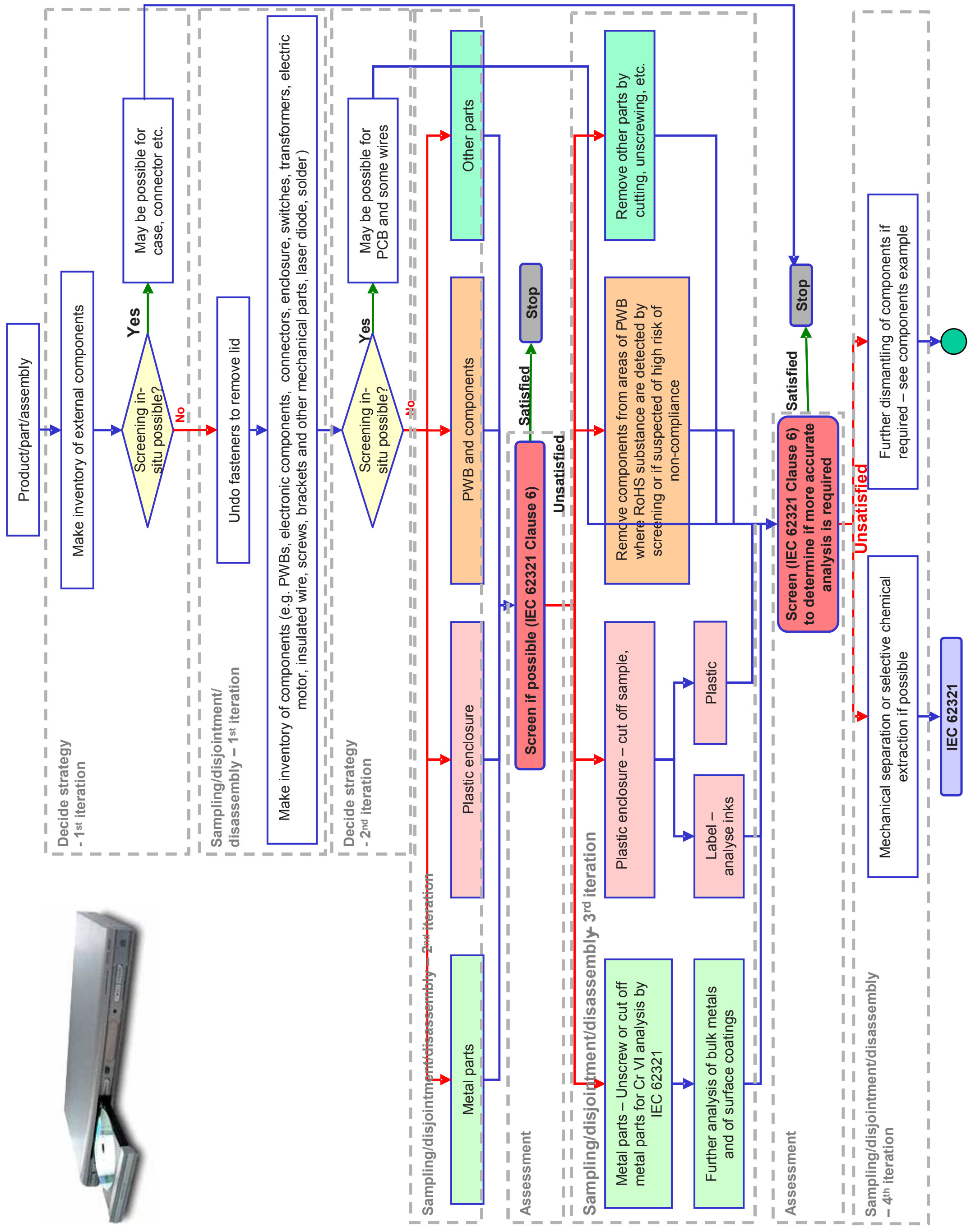


Figure A.3 – Sampling of DVD player

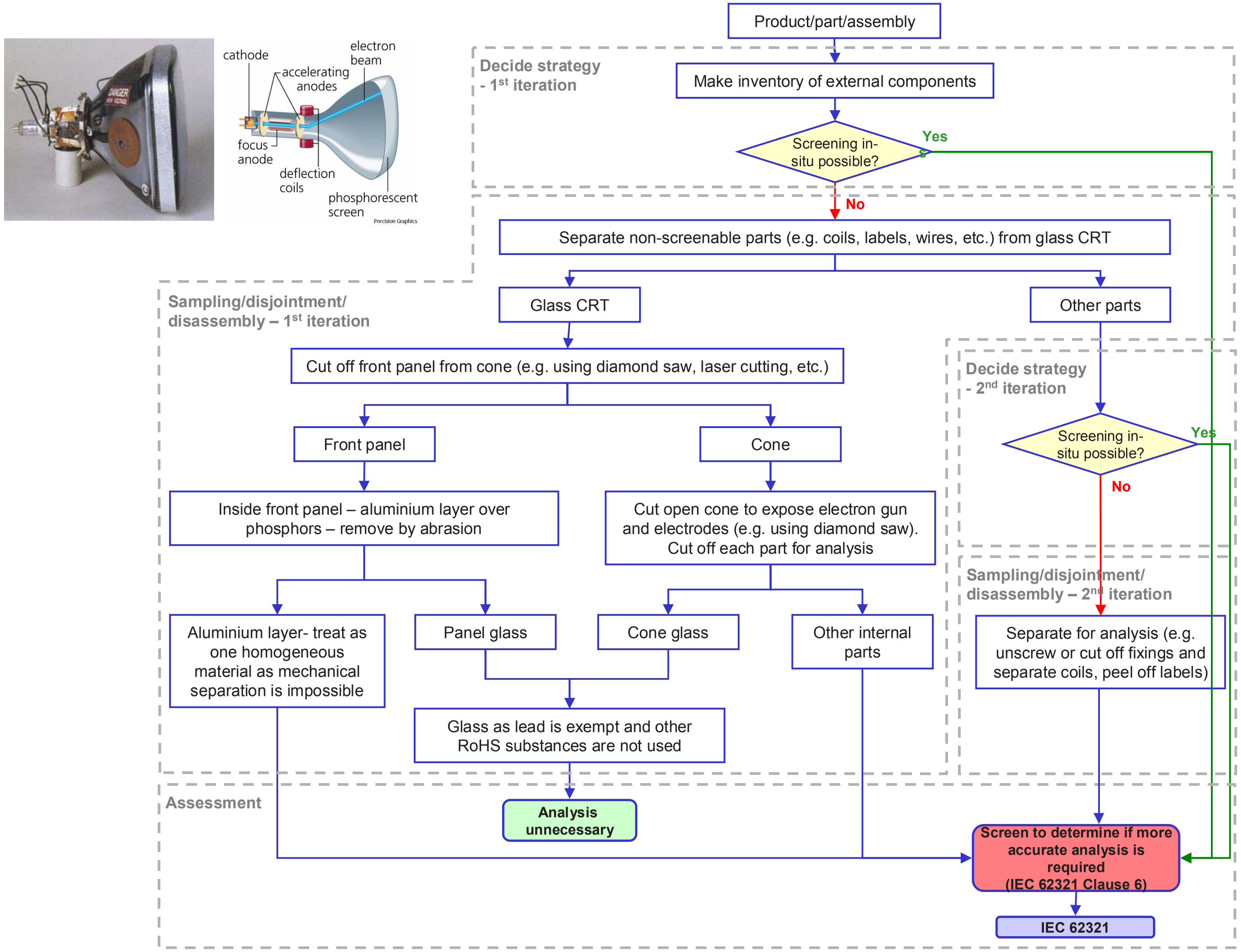


Figure A.4 – Sampling of CRT

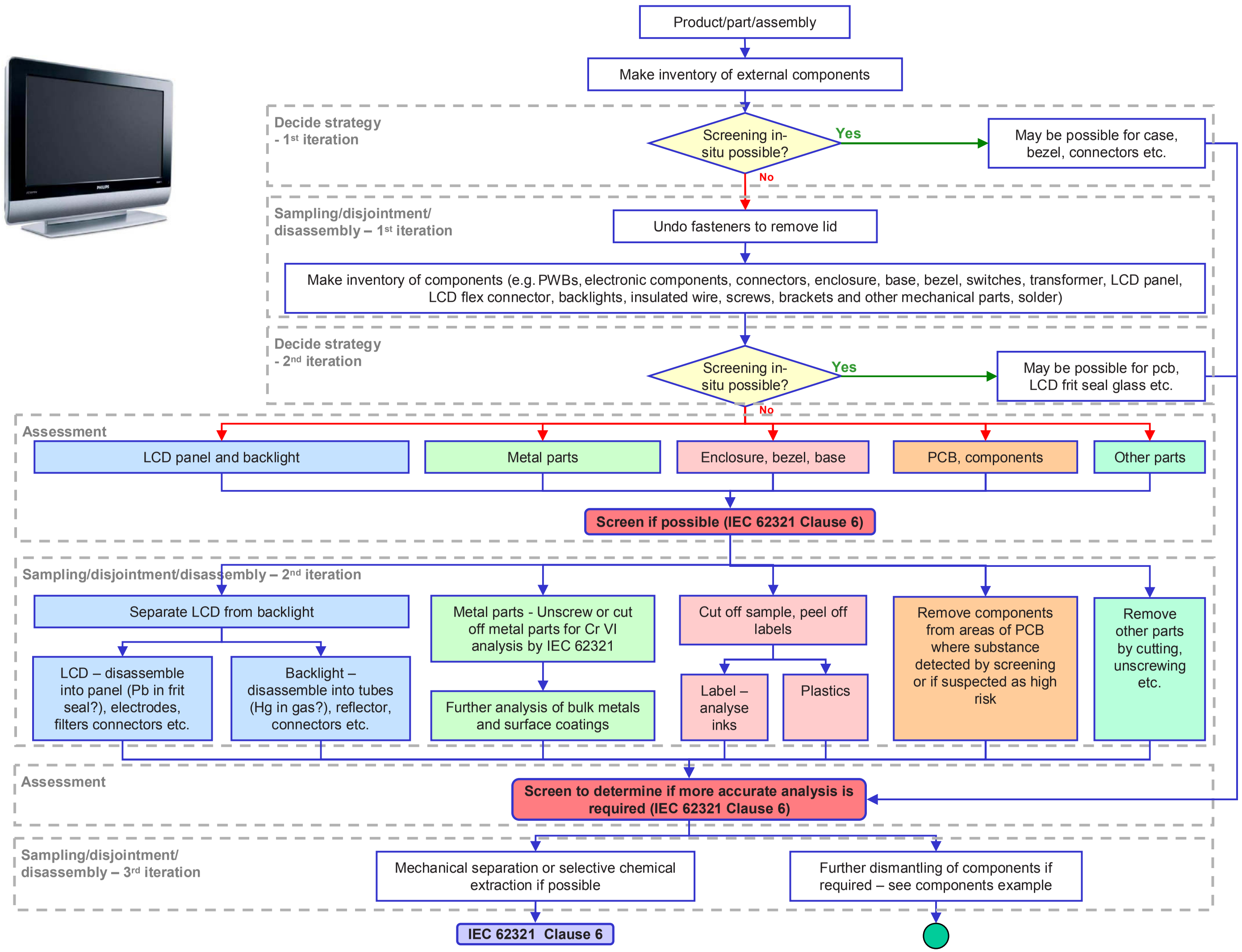


Figure A.5 – Sampling of LCD TV

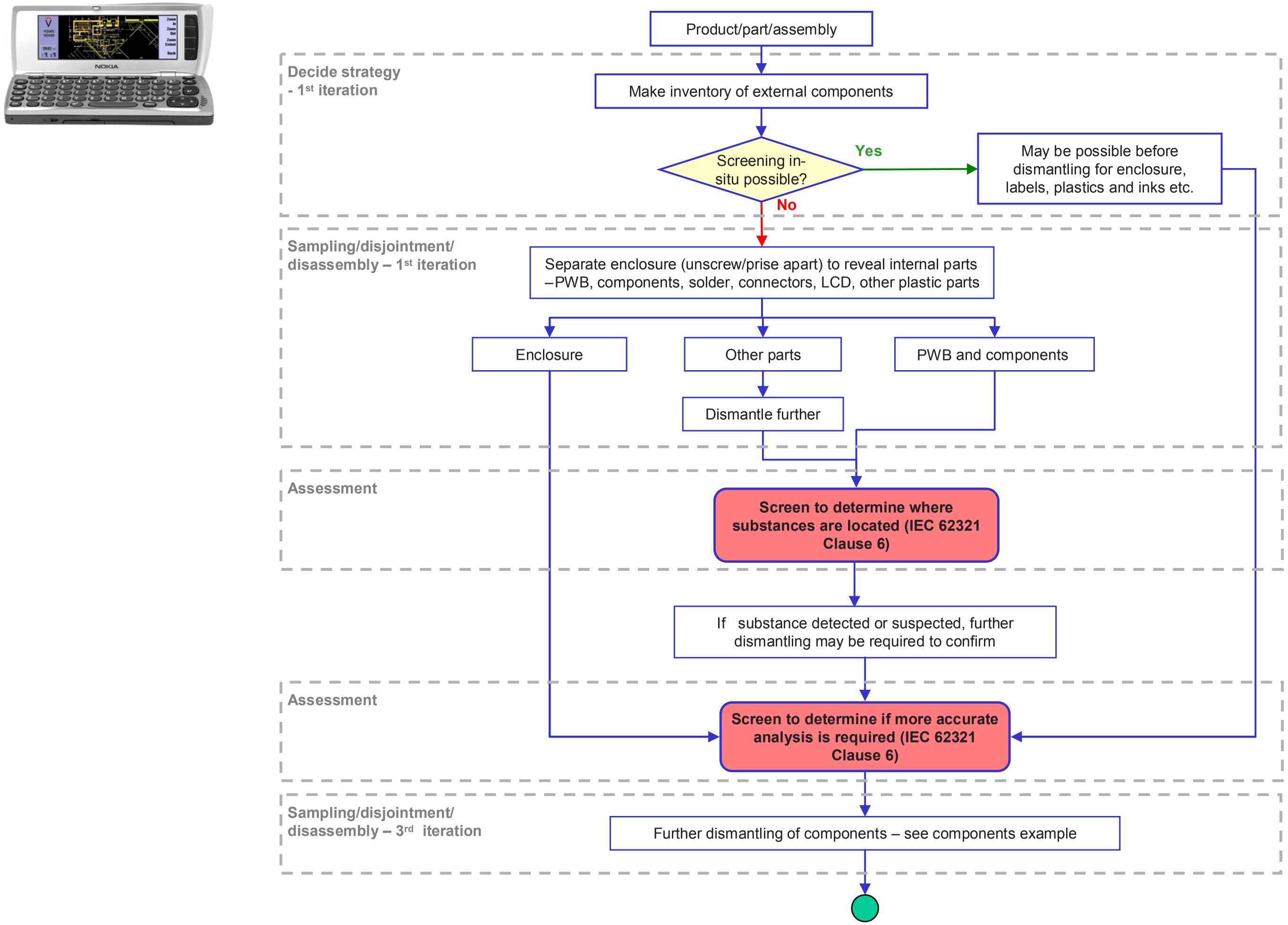
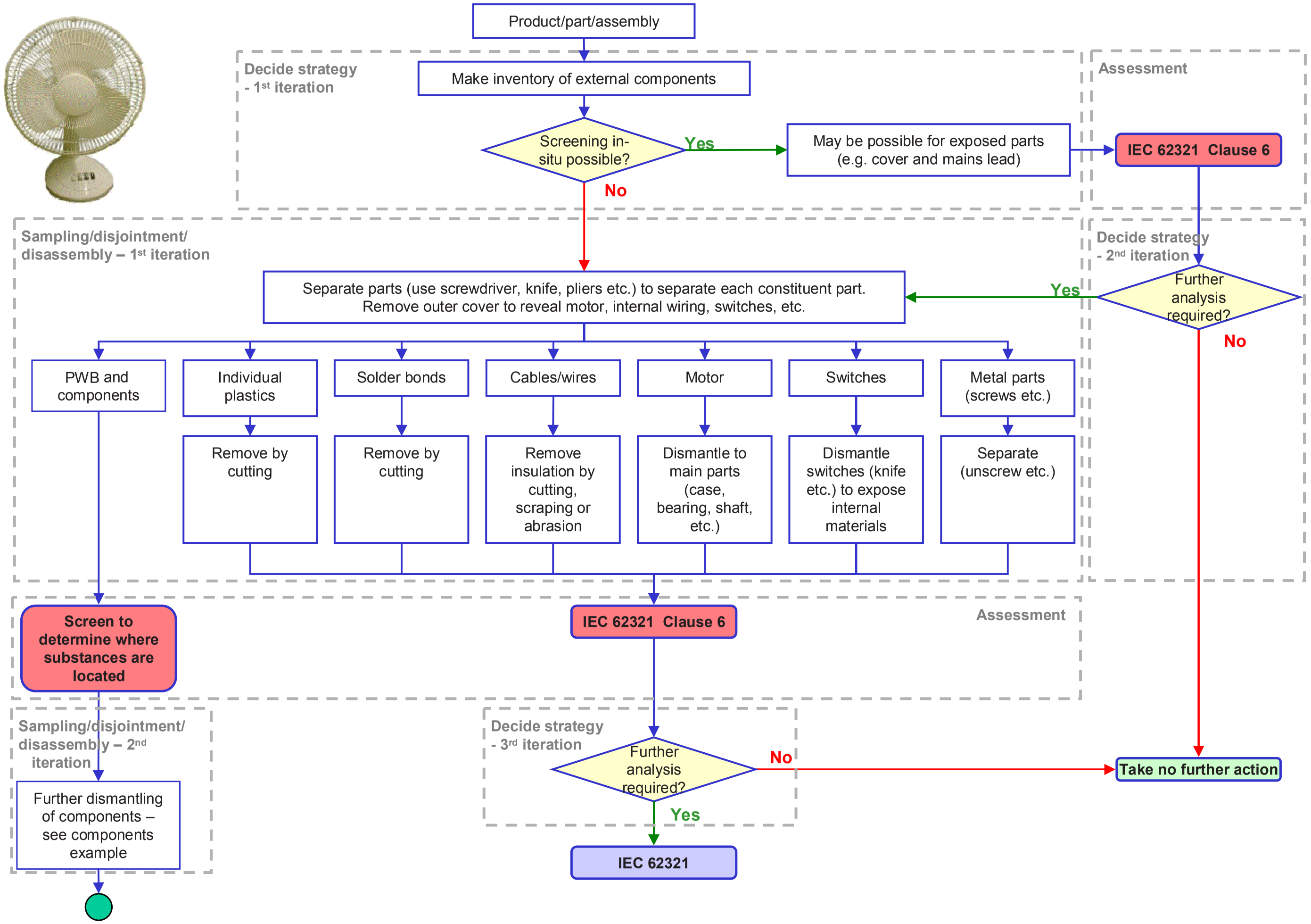


Figure A.6 – Sampling of PDA/phone



Note: Do not forget to analyse labels (cut off) and inks (in-situ)

Figure A.7 – Sampling of desk fan

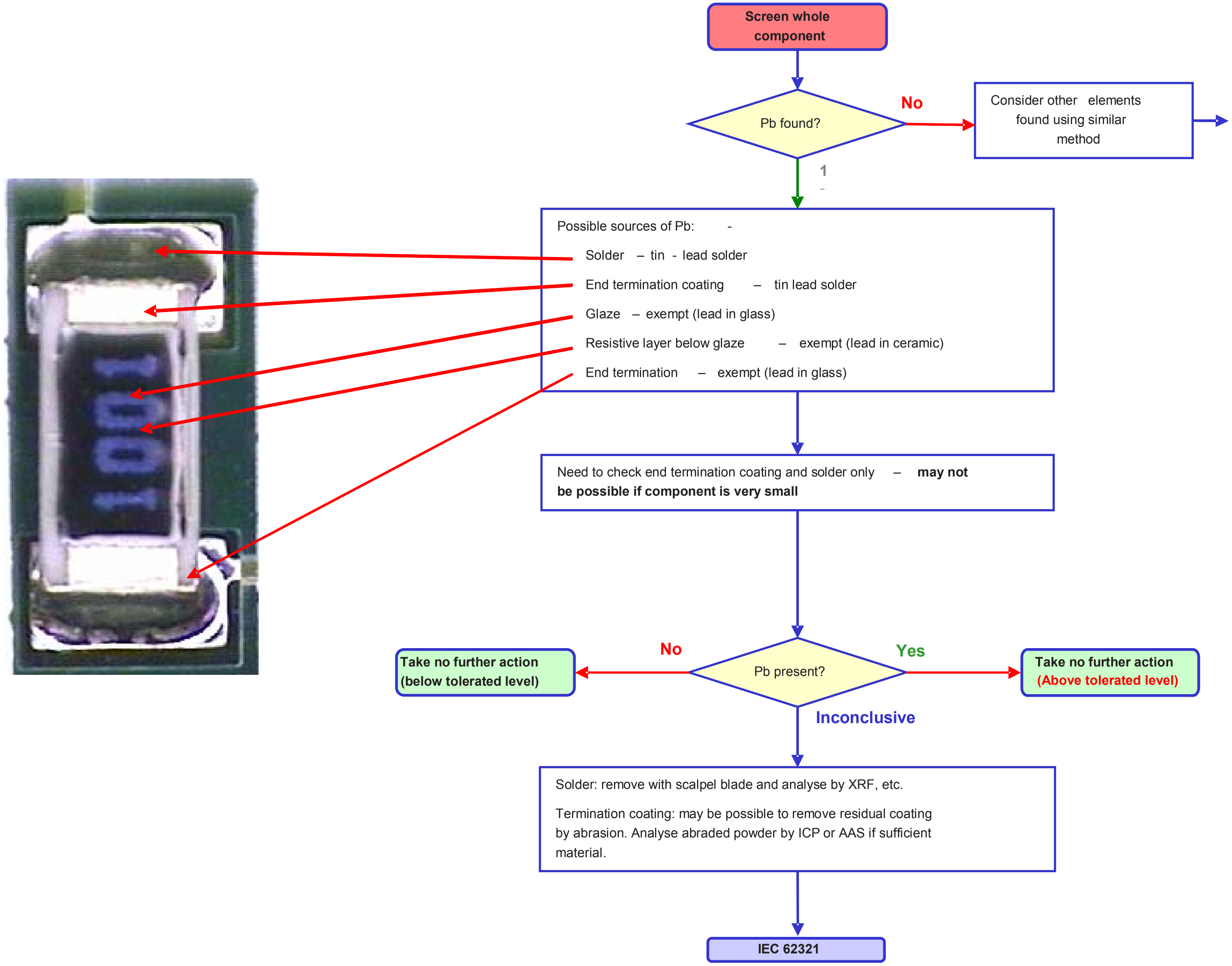


Figure A.8 – Compoments – Example 1 – Thick film resistor

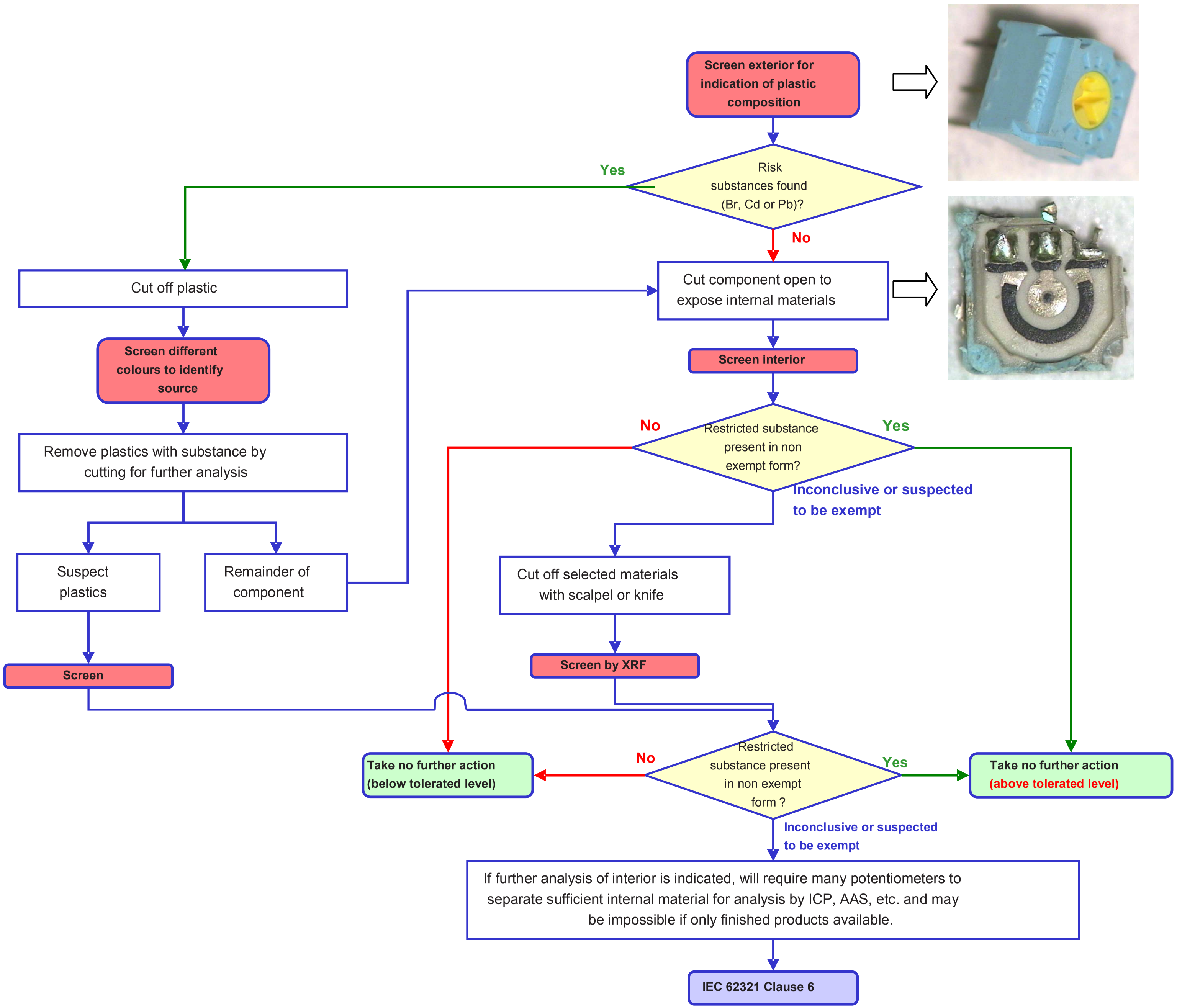


Figure A.9 – Components – Example 2 – SMD potentiometer

Annex B
(informative)

Probability of presence of restricted substances

B.1 Introductory remark

In order to optimize the sampling and analysis in the flowcharts of Annex A, it is important to have knowledge of the presence of the restricted substances in electrotechnical products. It is unnecessary to test materials/components for substances that are known not to be present, e.g. flame retardants in stainless steel. Furthermore for compliance verification materials/components that have a higher probability of presence of restricted substances would need more frequent and extensive analysis. Table B.1 provides an overview of the typical materials and components in electrotechnical products with their probability of presence for six restricted substances. This table provides a first indication of the relevant areas for sampling. However as the electrotechnical industry covers many materials and components, and new innovations are made daily this matrix does not intend to be comprehensive.

Table B.1 – Probability of presence of restricted substances in materials and components used in electrotechnical products

Components/ materials	**Restricted substances[a]**						**Number of homogeneous materials[b]**	**Remarks**
	Hg	**Cd**	**Pb**	**Cr (VI)**	**PBBs**	**PBDEs**		
Mechanical parts								
Framework – metal							1	Unpainted
Housing – plastic	L	L	L	L	L	M	1	
Power cord/cable	L	H	H	L	L	M	>1	
Thick film sensor	L	H	M	L	L	M	>1	
Heat sink	L	L	L	L	N/A	N/A	1	
Screw, washer, fastener – metal	L	M	M	H	N/A	N/A	1 and >1	Some are coated e.g. black and yellow chromate
Glass – CRT, lamp glass-to-metal seal	L	M	H	L	N/A	N/A	>1	Pb in glass could be exempted
Phosphorescent coating (e.g. CRT)	L	H	L	L	N/A	N/A	>1	
LCD panel/screen	H	L	H	H	L	L	>1	
Plasma panel/screen	H	L	H	H	L	L	>1	Pb in glass could be exempted
Lamps, back light	H	L	H	M	N/A	N/A	>1	Hg used in backlights could be exempted
Magnetic head	L	L	H	M	N/A	N/A	>1	
Printed wiring board (PWB)								
PWB substrate/laminate	L	L	L	L	L	N/A	>1	
Connector	M	L	H	L	L	H	>1	
Capacitor – electrolytic	L	M	H	L	L	M	>1	
Capacitor – chip-type	L	M	M	L	L	M	>1	
Resistor – IMT-type	L	M	H	L	L	L	>1	
Resistor – chip-type	L	H	M	L	L	L	>1	
Diode	L	M	M	L	L	L	>1	
Fuse	L	M	H	L	L	L	>1	
Solder (process and hand soldering)	L	M	H	L	N/A	N/A	1	
Glue (red and white)	L	L	M	L	M	M	1	Used to fix components
Component termination coating	L	H	H	L	N/A	N/A	1 and >1	

Components/ materials	**Restricted substances**[a]						**Number of homogeneous materials**[b]	**Remarks**
	Hg	**Cd**	**Pb**	**Cr (VI)**	**PBBs**	**PBDEs**		
Component mouldings	L	L	L	L	L	H	1 & >1	
Integrated circuit (IC) and BGAs	L	L	H	L	L	L	>1	
Relay – mercury	H	L	M	L	L	L	>1	
Relay – electromagnetic	L	H	M	L	L	L	>1	
Switch – mercury	H	L	M	L	L	L	>1	
Switch – mechanical	M	H	M	L	L	L	>1	
Thermostats	H	M	M	L	L	L	>1	
Flame sensors	H	M	M	L	L	L	>1	
Thermal imaging semiconductors	H	M	M	L	L	L	>1	
Transformer (LOT)	L	M	H	L	L	M	>1	
Accessories								
Remote controls	L	H	H	L	L	L	>1	
External cable (e.g. Scart, USB, cinch)	L	H	H	L	L	L	>1	
External power supply	L	H	H	L	L	M	>1	
Materials								
Paint, ink and similar coating	L	H	H	M	L	L	1	
Adhesive			M		M	M	1	
Polyurethane – high gloss	H	M	M	L	L	M	>1	
Polyvinyl chloride (PVC)	L	H	H	M	L	M	1	
Styrene, polystyrene (HI-PS), ABS, polyethylene (PE), polyester	L	M	M	L	L	H	1	
Rubber	L	M	M	L	L	M	1	
Plastics – other	L	M	M	L	L	M	1	
Colorants (all plastics) red, orange, yellow, pink, green	M	H	H	H	N/A	N/A	1	
Metal	L	M	H	H	N/A	N/A	1 and >1	
Steel – other	L	L	L	H	N/A	N/A	1	
Steel – free-machining	L	L	H	L	N/A	N/A	1	
Copper alloy	L	H	H	L	N/A	N/A	1	Pb in metal could be exempt
Aluminium alloy	L	L	H	L	N/A	N/A	1	Pb in metal could be exempt
Metallic chromium plating	L	L	L	L	N/A	N/A	>1	
Zinc coating	L	H	H	H	N/A	N/A	>1	
Other metallic coatings	L	H	L	H	N/A	N/A	>1	
Glass – other	L	M	H	M	N/A	N/A	U	Pb could be exempt
Ceramics	L	M	H	L	N/A	N/A	U	Pb could be exempt

NOTE This table is to be used as guidance to assist in selecting components/materials for testing that have a high probability of contining a restricted substance. Not every part listed in this table requires testing and not every “high” probability substance is intended to be tested. See 4.3 for additional guidance on sampling strategy (e.g. Table 2). It is very important to identify any applicable exemptions before beginning testing to ensure an accurate interpretation of the analytical results and avoid any unnecessary follow-up testing.

[a] L Low probability
M Medium probability
H High probability
N/A Not applicable

[b] 1 One homogeneous material
>1 Two or more homogeneous materials
U Unknown

Annex C
(informative)

Composite testing and sampling

C.1 Introductory remark

As discussed in 5.6.4, composite testing of a product made up of several different parts and/or material layers can be used as a screening technique in certain circumstances to minimize the number of samples and consequently the analytical costs. There are two approaches:

1) calculating the maximum sample concentration based on the analytical detection limit;
2) calculating the minimum analytical detection limit based on the sample concentration.

In the first approach, the maximum level of a restricted substance in one material of the composite can be calculated when a "not detected" result is obtained from chemical analysis of the entire composite sample.

C.2 Example 1

Example 1 is based on the following presumptions:

- The component/part contains four different homogeneous materials (weighing a total of 18 mg).
- Only the smallest contributor to the compsite (material A) could contain restricted substances, Pb and Cd.
- The method of chemical analysis on the composite sample had a detection limit of 20 mg/kg.
- For chemical analysis, the component/part was first ground to a powder (a homogeneous sample).

Results of "not detected" mean that up to 20 mg/kg of Pb and Cd could be present in the composite sample taken from the component. Based on the worst case (both Pb and Cd are present at 20 mg/kg), the maximum contamination or error of Pb and Cd can be calculated (see Table C.1). For the maximum levels of 20 mg/kg for both Pb and Cd in the composite sample, homogeneous material A may contain upto 360 mg/kg Pb and Cd. For Pb this is below the allowable limit of 1 000 mg/kg. However, for Cd the maximum allowable limit of 100 mg/kg may be exceeded.

The conclusion for this composite sample is that further analysis is needed for Cd to determine if it meet the requirements.

NOTE Composite testing is only a screening method.

Table C.1 – Calculated maximum concentration for a composite sample based on detection limit

Material	**Weight** mg	**Percent of total weight of composite sample**	**Maximum Pb content (for 20 mg/kg detection limit)** mg/kg	**Maximum Cd content (for 20 mg/kg detection limit)** /mg/kg
Material A	1	6 %	360	360
Material B	4	22 %	0	0
Material C	5	28 %	0	0
Material D	8	44 %	0	0
Total (composite)	18	100 %		
Average (composite)			20	20

In the second approach, the required detection limit can be calculated to ensure that no restricted substance is present above a certain level.

C.3 Example 2

Example 2 (see Table C.2) is based on the following presumptions:

- Contamination of the same composite sample used in Example 1 with 1 000 mg/kg Pb and 100 mg/kg Cd.

To verify these levels of Pb and Cd by chemical analysis would require a method with a detection limit of around 50 mg/kg for Pb and 5 mg/kg of Cd, as illustrated in Table C.2.

Table C.2 – Required detection limit for a composite sample based on the maximum allowable concentration

Material	**Weight** mg	**Percent of total weigh of composite samplet**	**Pb content** mg/kg	**Cd content** mg/kg
Material A	1	6 %	1 000	100
Material B	4	22 %	0	0
Material C	5	28 %	0	0
Material D	8	44 %	0	0
Total (composite)	18	100 %		
Average (composite)			56	5,6
Required detection limit/ mg/kg			56	5,6

Further, it is important to keep in mind that both the detection limits of analytical methods and the concentrations of restricted substances have a margin of error and the presence of restricted substances can vary in a “homogeneous material” (see Figure E.7). Therefore it is advisable to include a margin of safety when applying this concept.

Annex D
(informative)

Tools used in sampling

Commonly used tools for sampling by disassembly and disjointment tools are as follow:

- Soldering iron
- Screw driver (electrical)
- Cable stripper
- Stanley knife
- Spanners (open ended/ ring)
- Wrench
- Hammer
- Drill
- Solder wick, i.e. wire that sucks up molten solder
- Allen-keys
- End-cut pliers
- Pliers
- Hand saw
- Shears
- Tweezers
- Plastic bags

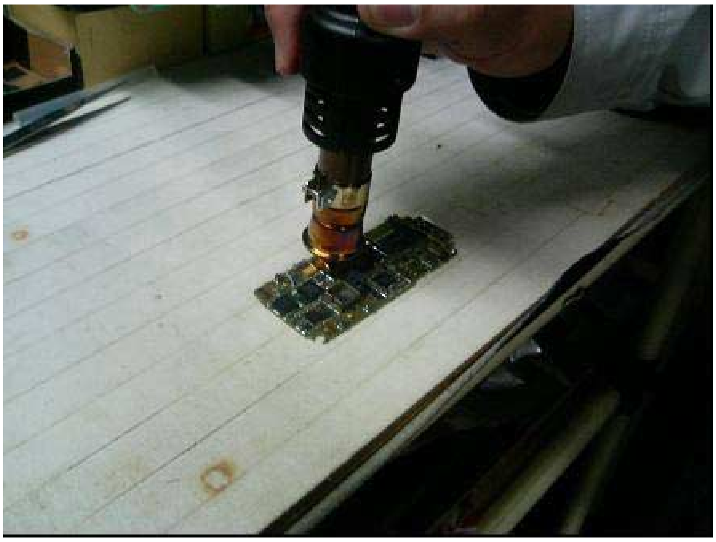


Figure D.1 – Hot gas gun for removing the electronic components

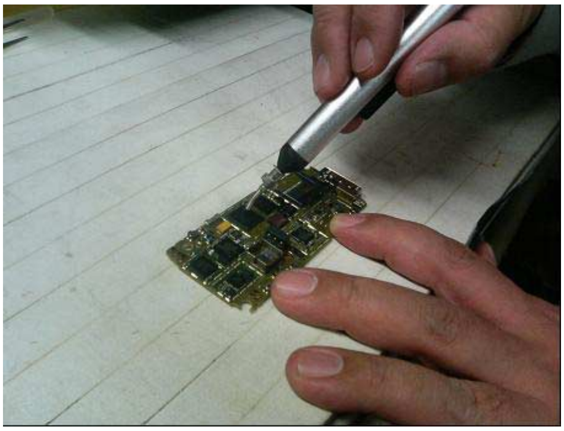


Figure D.2 – Vacuum pin to remove the target electronic devices

Clause 5 of IEC 62321 includes more sample preparation tools that may be needed to prepare the sample for an analytical testing technique.

Annex E
(informative)

Use of XRF screening techniques in sampling

E.1 Introductory remark

Clause 6 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.

E.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 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 in the instrument. The typical spot size of hand-held portable XRF instruments ranges from about 3-10 mm^2, 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.

E.3 Factors affecting XRF results

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 of the analyser.
- It is essential to understand the depth of penetration of X-rays 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.

E.4 Practical 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 affect further sampling decisions.

E.4.1 AC power cord

Figure E.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 E.1 summarizes the screening of the product.

Table E.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 1)	High	Yes
Plastic body of plug	Polymer	Pb, Br, Sb 1)	High	Yes
Metal prongs	Metal alloy	Cu, Zn,(Pb)	Moderate	Yes
1) Presence of bromine (Br) and antimony (Sb) could indicate the use of a restricted brominated flame retardant.				

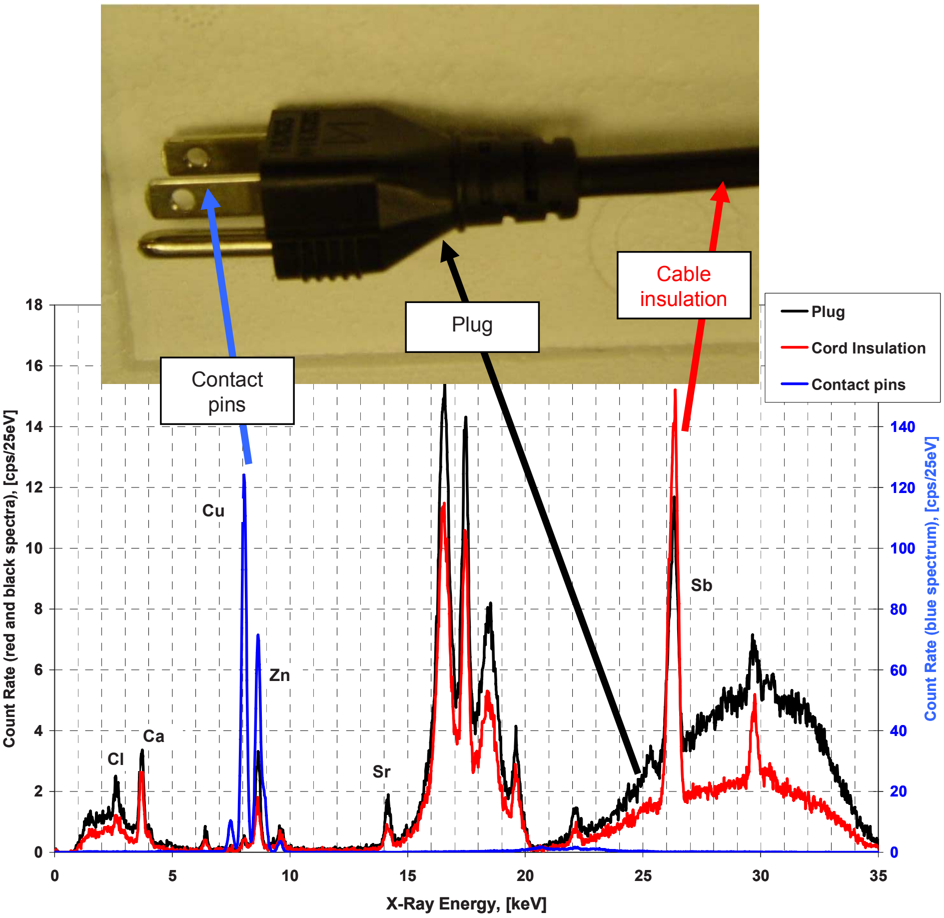


Figure E.1 – AC power cord, X-ray spectra of sampled sections

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 E.1. Neither cord insulation nor plug polymer contains any of the restricted substances. 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, neither of these two parts contains Pb or Br. The connecting pins are made of nickel-plated brass. Up to this point in the sampling and screening process the cable is compliant. Therefore, the cord needs to be disassembled (in this case destructively) and to test its parts 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.

E.4.2 Serial RS232 cable

This example, illustrated in Figure E.2, shows a printer cable that does contain 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.

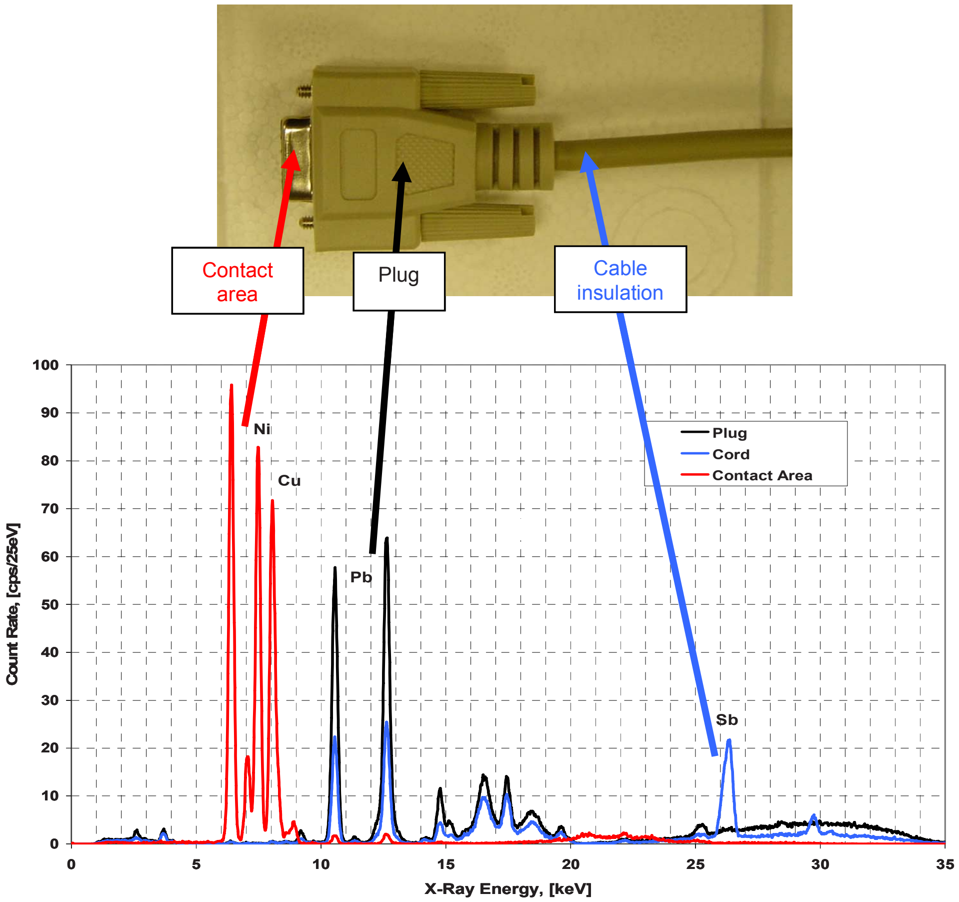


Figure E.2 – RS232 cable and its X-ray spectra

E.4.3 Cell phone charger

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

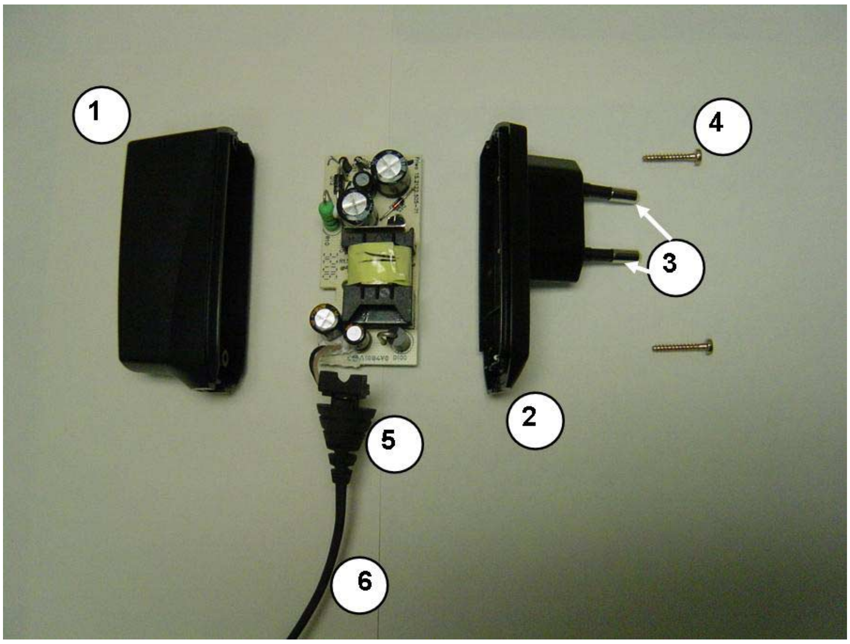


Figure E.3 – Cell phone charger shown partially disassembled

Table E.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+})

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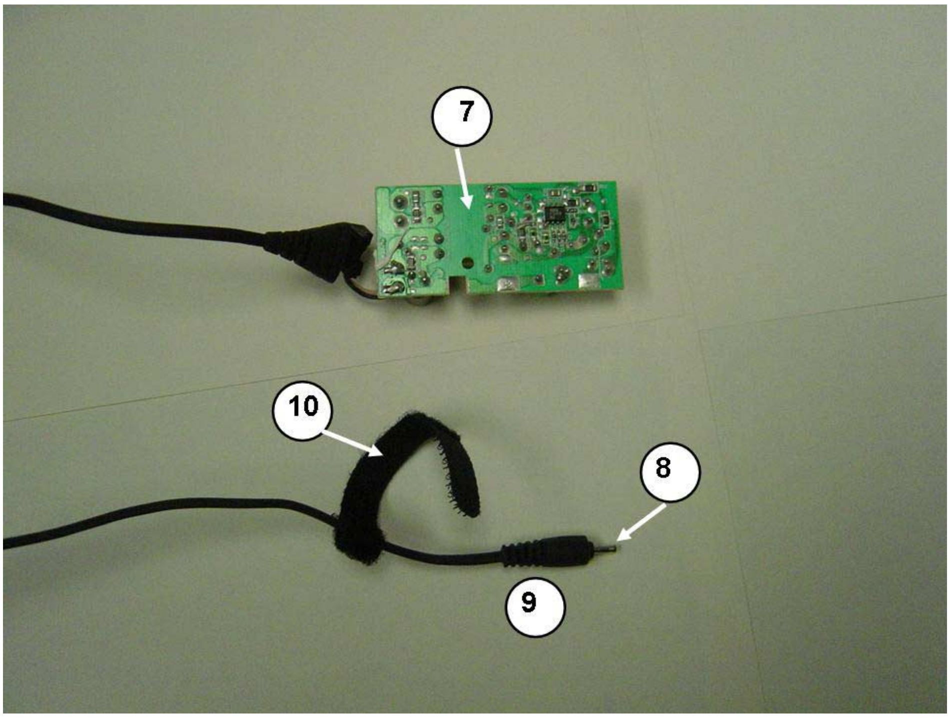

Figure E.4 – PWB and cable of cell phone charger

The case of the cell phone charger is very educational. Firstly the charger could be sampled and analysed without disassembly. When its case was analysed (sample 1 in Figure E.3) prior to disassembly, it showed – depending on location – between 2 600 mg/kg to 7 000 mg/kg of Br. If the analysis was stopped at that stage, 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. Next sample 7 was analysed. It is a section of the PWB board with no components, which therefore can 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 pictured in Figure E.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 analyzing without disassembly, it was possible to determine elevated levels of Br in the whole product.

E.4.4 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. Figure E.5 shows the measurement area resulting from two different collimators when attempting to analyse a single solder joint on a PWB. In the case of a 1,27 mm diameter collimator (Figure E.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 0,3 mm diameter collimator (Figure E.5b) the measurement area is small enough that only the solder will contribute to the measurement.

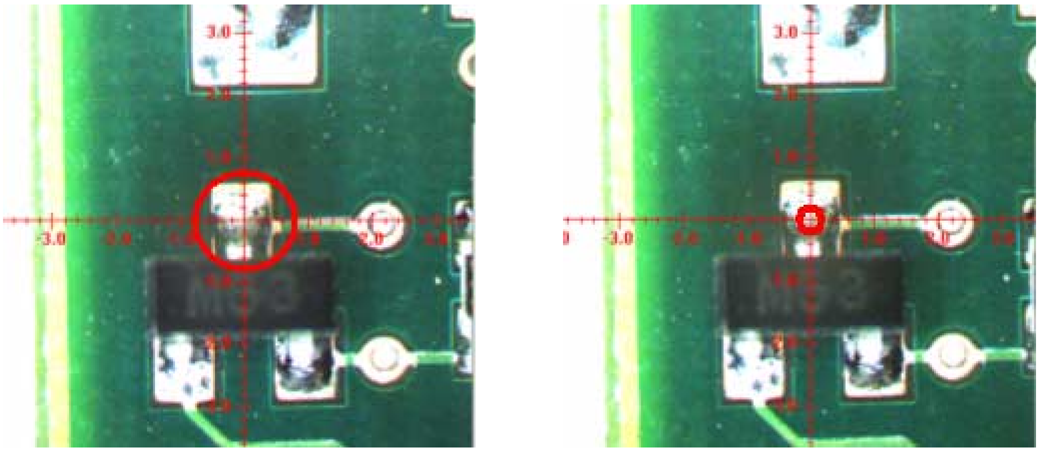

Figure E.5a – Spot from 1,27 mm collimator

Figure E.5b – Spot from 0,3 mm collimator

Figure E.5 – Spots from 1,27 mm and 0,3 mm collimaters

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 1,27 mm collimation, the instrument was analysing 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 the 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 0,3 mm 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.

E.4.5 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 in the determination of 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 E.6. The main concern is whether the Pb found on the board is exempt or not. The bottom part of Figure E.6 shows the combined map of Pb and Sn. Pb is marked in green while Sn is red in green.

The quantitative results of the analysis of the PWB at spots 1 and 2 are reported in Table E.3. At spot (1), Pb is present with Sn (Pb/(Sn+Pb) = 85 %) which suggests that Pb is contained in a high temperature solder, and therefore its presence is 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 (see Figure 15). 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.

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 a solid mater, the SEM-EDX is typically a qualitative tool at best. This technique will analyse only the material 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 E.7 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.

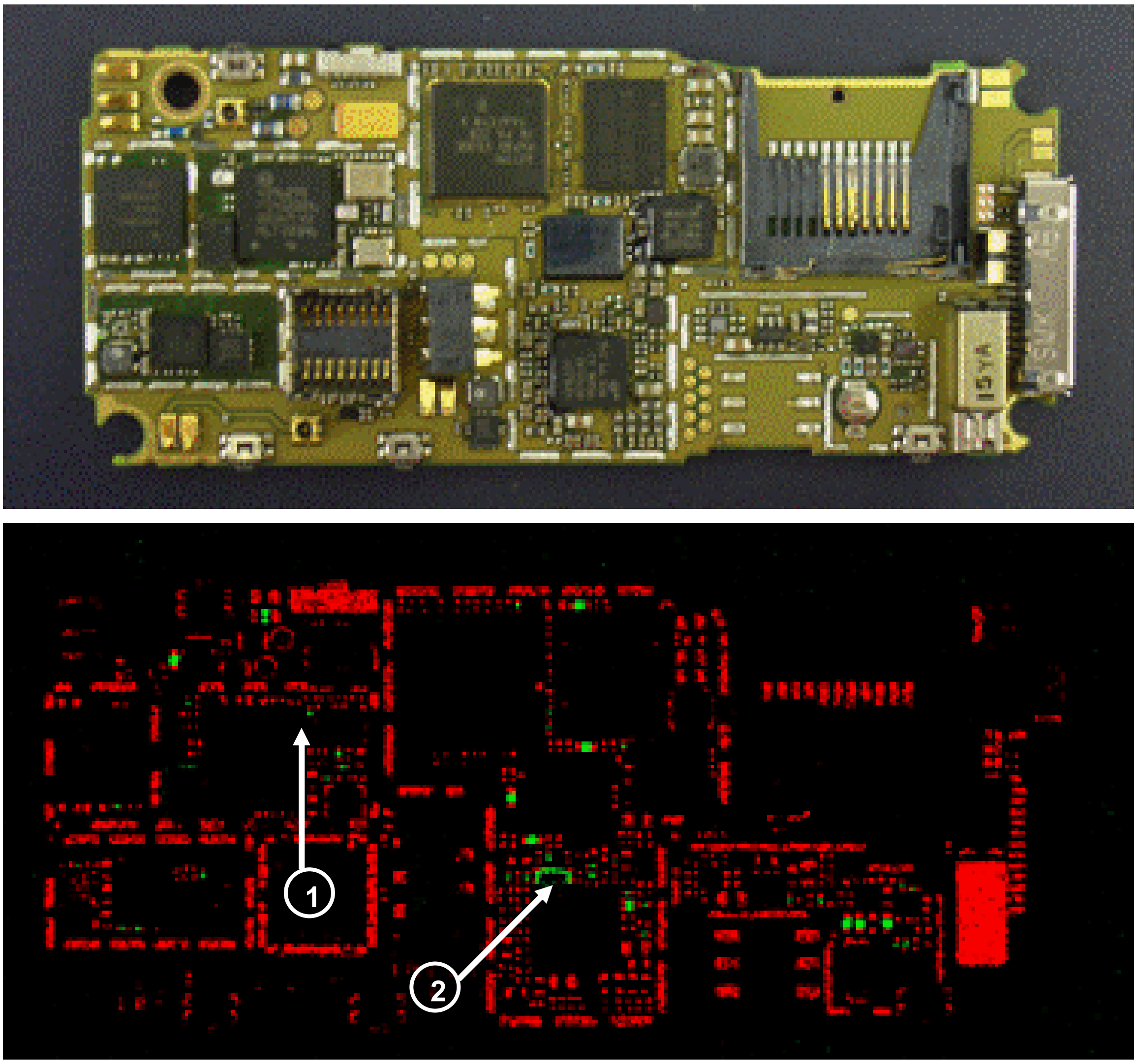

Figure E.6 – Examples of substance mapping on PWBs

Table E.3 – Results of XRF analysis at spots 1 and 2 as shown in Figure E.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	-	82,9	3,9	1,2

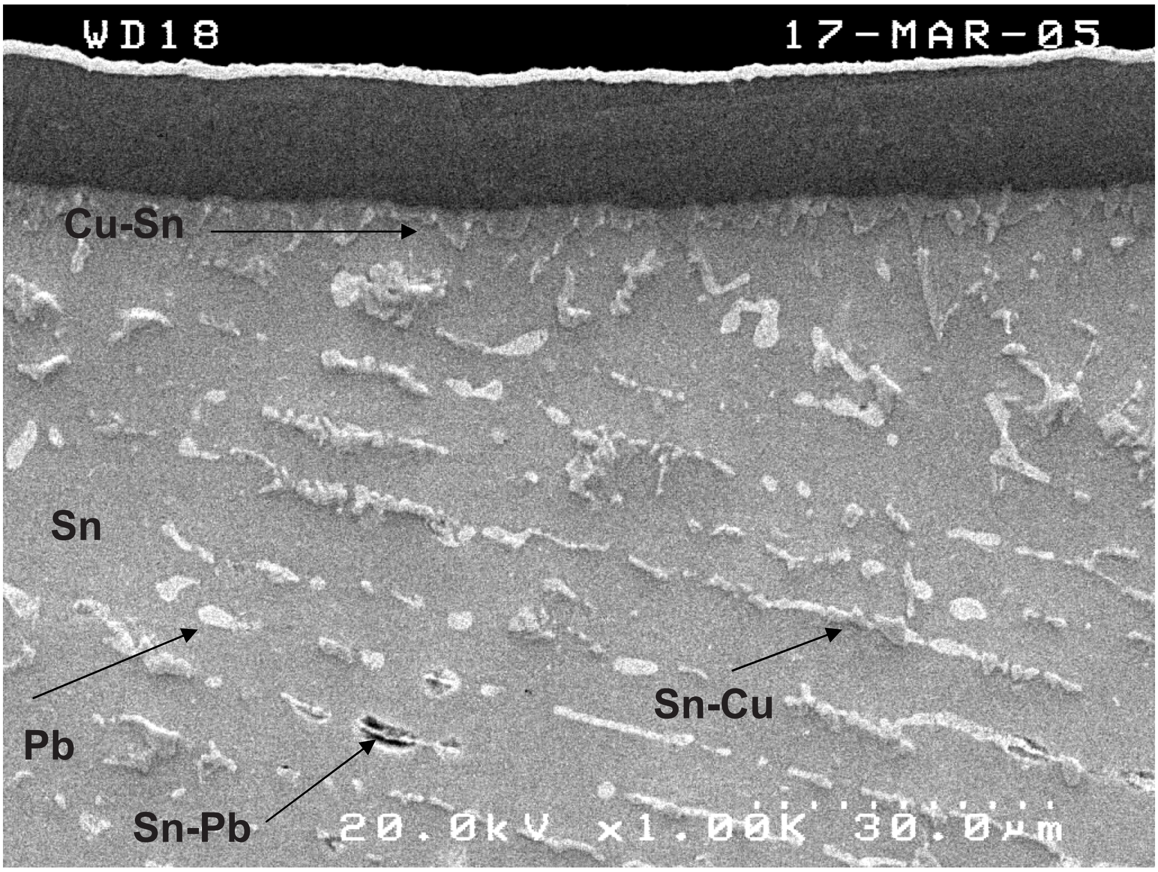

Figure E.7 – SEM-EDX image of Pb free solder with small intrusions of Pb (size = 30 µm)

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