BS EN 62321-2:2014

BSI Standards Publication

Determination of certain substances in electrotechnical products

Part 2: Disassembly, disjointment and mechanical sample preparation

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National foreword

This British Standard is the UK implementation of EN 62321-2:2014. It is identical to IEC 62321-2:2013. Together with BS EN 62321-1:2013, BS EN 62321-3-1:2014, BS EN 62321-3-2:2014, BS EN 62321-4:2014, BS EN 62321-5:2014, BS EN 62321-6, BS EN 62321-7-1, BS EN 62321-7-2 and BS EN 62321-8 it supersedes BS EN 62321:2009, which will be withdrawn upon publication of all parts of the BS EN 62321 series.

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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English version

Determination of certain substances in electrotechnical products - Part 2: Disassembly, disjointment and mechanical sample preparation
(IEC 62321-2:2013)

Détermination de certaines substances dans les produits électrotechniques -
Partie 2: Démontage, désassemblage et préparation mécanique de l'échantillon
(CEI 62321-2:2013)

Verfahren zur Bestimmung von bestimmten Substanzen in Produkten der Elektrotechnik -
Teil 2: Demontage, Zerlegung und mechanische Probenvorbereitung
(IEC 62321-2:2013)

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

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

Foreword

The text of document 111/301/FDIS, future edition 1 of IEC 62321-2, prepared by IEC/TC 111 "Environmental standardization for electrical and electronic products and systems" was submitted to the IEC-CENELEC parallel vote and approved by CENELEC as EN 62321-2:2014.

The following dates are fixed:

• latest date by which the document has to be implemented at national level by publication of an identical national standard or by endorsement	(dop)	2014-10-25
• latest date by which the national standards conflicting with the document have to be withdrawn	(dow)	2016-11-15

EN 62321-2:2014 is a partial replacement of EN 62321:2009, forming a structural revision and generally replacing Clause 5 and incorporating IEC/PAS 62596:2009 [1][1)] which will be withdrawn upon publication of IEC 62321-2.

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

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

Endorsement notice

The text of the International Standard IEC 62321-2:2013 was approved by CENELEC as a European Standard without any modification.

In the official version, for Bibliography, the following notes have to be added for the standards indicated:

IEC 62554	NOTE	Harmonised as EN 62554 (not modified).
IEC 62542	NOTE	Harmonised as EN 62542 (not modified).
IEC 62321-6	NOTE	Harmonised as EN 62321-6 (not modified).
IEC 62321-7-1	NOTE	Harmonised as EN 62321-7-1 (not modified).
IEC 62137-1-2	NOTE	Harmonised as EN 62137-1-2 (not modified).

[1)] Numbers in square brackets refer to the Bibliography.

Annex ZA
(normative)

Normative references to international publications with their corresponding European publications

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

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

Publication	Year	Title	EN/HD	Year
IEC 62321-1	-	Determination of certain substances in electrotechnical products - Part 1: Introduction and overview	EN 62321-1	-
IEC 62321-3-1	-	Determination of certain substances in electrotechnical products - Part 3-1: Screening electrotechnical products for lead, mercury, cadmium, total chromium and total bromine using X-ray Fluorescence Spectrometry	EN 62321-3-1	-
IEC 62321-3-2	-	Determination of certain substances in electrotechnical products - Part 3-2: Screening of total bromine in electric and electronic products by combustion-ion chromatography (C-IC)	EN 62321-3-2	-
IEC 62321-4	-	Determination of certain substances in electrotechnical products - Part 4: Determination of mercury in polymers, metals and electronics by CV-AAS, CV-AFS, ICP-OES and ICP-MS	EN 62321-4	-
IEC 62321-5	-	Determination of certain substances in electrotechnical products - Part 5: Determination of cadmium, lead and chromium in polymers and electronics, and cadmium and lead in metals by AAS, AFS, ICP-OES and ICP-MS	EN 62321-5	-

CONTENTS

INTRODUCTION

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

The use of certain substances (e.g. lead (Pb), cadmium (Cd) and polybrominated diphenyl ethers (PBDEs)) in electrotechnical products, is a source of concern in current and proposed regional legislation.

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

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

DETERMINATION OF CERTAIN SUBSTANCES IN ELECTROTECHNICAL PRODUCTS –

Part 2: Disassembly, disjointment and mechanical sample preparation

1 Scope

This part of IEC 62321 provides strategies of sampling along with the mechanical preparation of samples from electrotechnical products, electronic assemblies and electronic components. These samples can be used for analytical testing to determine the levels of certain substances as described in the test methods in other parts of IEC 62321. Restrictions for substances will vary between geographic regions and from time to time. This Standard describes a generic process for obtaining and preparing samples prior to the determination of any substance which are under concern.

This standard 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;
- guidance regarding other routes to gather additional information on certain substances in a product, although the information collected has relevance to the sampling strategies in this standard;
- safe disassembly and mechanical disjointment instructions related to electrotechnical products (e.g. mercury-containing switches) and the recycling industry (e.g. how to handle CRTs or the safe removal of batteries). See IEC 62554 [2] for the disjointment and mechanial sample preparation of mercury-containing fluorescent lamps;
- the definition of a “unit” as the sample;
- sampling procedures for packaging and packaging materials;
- analytical procedures to measure the levels of certain substances. This is covered by other standards (for example other parts of IEC 62321), which are referred to as the "test standard" in this standard;
- guidelines for assessment of compliance.

NOTE Further guidance on assessment procedures is provided by IEC/TR 62476 [3].

2 Normative references

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

IEC 62321-1, *Determination of certain substances in electrotechnical products – Part 1 Introduction and overview*

IEC 62321-3-1, *Determination of certain substances in electrotechnical products – Part 3-1: Screening – Lead, mercury, cadmium, total chromium and total bromine using X-ray fluorescence spectrometry*

IEC 62321-3-2, *Determination of certain substances in electrotechnical products – Part 3-2: Screening – Total bromine in polymers and electronics by combustion – Ion chromatography (C-IC)*

IEC 62321-4, *Determination of certain substances in electrotechnical products – Part 4: Determination of mercury in polymers, metals and electronics by CV-AAS, CV-AFS, ICP-OES and ICP-MS*

IEC 62321-5, *Determination of certain substances in electrotechnical products – Part 5: Determination of cadmium, lead and chromium in polymers and electronics and cadmium and lead in metals by AAS, AFS, ICP-OES, ICP-AES and ICP-MS* [2]

3 Terms, definitions and abbreviations

3.1 Terms and definitions

For the purposes of this document, the definitions given in IEC 62321-1, as well as the following, apply.

3.1.1
composite testing
testing two or more materials as a single sample that could be mechanically disjointed if necessary

3.1.2
certain substance
cadmium, lead, mercury, hexavalent chromium, polybrominated biphenyl, polybrominated diphenyl ether

NOTE IEC 62321-1 includes test methods for the evaluation of each of the substances identified in the definition above.

3.2 Abbreviations

AC	Alternating current
BGA	Ball grid array (electronic component)
CRT	Cathode ray tube (television)
DVD	Digital versatile disc
IC	Integrated circuit
JEDEC	Joint Electronic Devices Engineering Council
LCD	Liquid crystal display
MDL	Method detection limit
OEM	Original equipment manufacturer
PAS	Publicly Available Specification
PCB	Printed circuit board
PDA	Personal digital assistant
PWB	Printed wiring board
SIM	Subscriber identity module
SMD	Surface mounted device
TFT	Thin film transistor
TV	Television

USB	Universal serial bus

4 Introduction to sampling

4.1 Introductory remark

Obtaining a sample (i.e. sampling) is the first step in analysing electrotechnical products for the presence of certain substances. The strategy and process of sampling are 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 certain substances are 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 substances of concern

While many governments, industry partners and other stakeholders have their own requirements, it is not the intention of this standard to discuss fully all of these differences. However, awareness of different limits for certain substances is an important step in preparing the sampling strategy. Subclause 4.2 highlights the main areas of concern regarding the requirements for certain substances.

- Certain 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.
- Allowable limits for certain substances: generally speaking, the allowable levels of most certain 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 certain substances are above 1 000 mg/kg, e.g. lead in copper and aluminum alloys.
- Application of the allowable level: the manner in which the allowable level of a certain 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 standard, an “homogeneous material” is as defined in IEC 62542 [4]. However, the interpretation of "homogeneous material" is not consistent across the different regions.
- Applicable exemptions: some types of electrotechnical products are exempt from certain 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:

a) 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 cm^2 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.

b) 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 2). 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 lead). Similarly, present-day 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 Annex E this point is further illustrated in the disassembly of a mobile phone.

c) "Invisible" substances: another complicating factor in sampling and analysis is that generally certain substances are not visible. A component containing a certain substance may look and perform in an identical manner to one that does not. The presence or absence of certain 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 certain substances, visual detection is not practical.

d) 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. Notification of these changes does not always occur if the component still meets its technical specification.

e) 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 such as an LCD screen or IC, is considerably deeper.

These characteristics of the electrotechnical industry show that the management of certain 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 certain 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.

NOTE Oxidation states of certain substances may not be stable over time. For example, the concentration of hexavalent chromium in corrosion protection layers can change significantly with time and storage conditions.

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 illustrated by the iterative loops of sampling, disassembly and disjointment shown in Figure 1.

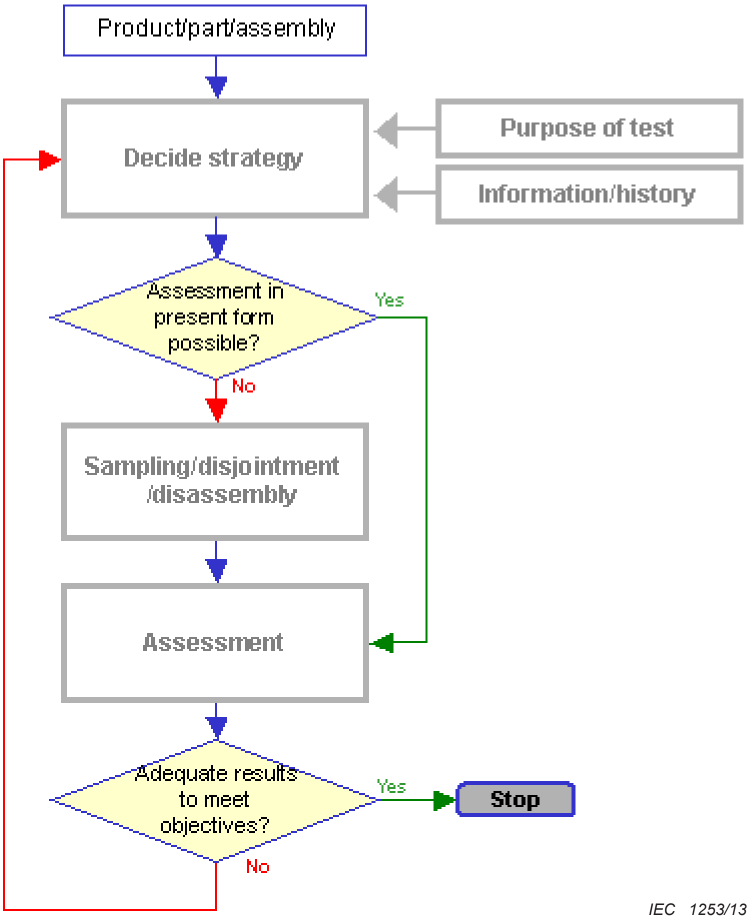


Figure 1 – Generic iterative procedure for sampling

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

- 1st iteration: partial disassembly (see 5.3);
- 2nd iteration: complete disassembly (see 5.4);
- 3rd iteration: partial disjointment (see 5.5);
- 4th – nth iteration: complete disjointment (see 5.6).

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 certain substances?
- Are there appropriate exemptions for the certain 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?

- Is there any published probability of the presence matrices for the materials or parts used in this product?
- Was any screening (e.g. X-ray fluorescence) 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 certain substances (less likely to contain restricted substances hence pose a lower risk of non-confomity if not tested, see Annex B);
- applicable exemptions for certain substances (the presence of certain substances would not affect conformity);
- material declarations;
- historical test data (evidence for the probability of containing certain substances);
- composite sampling and testing (covers several materials in a single test, but other factors shall be considered, see 5.7.3 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) is an analysis to verify if the product contains at least one certain substance exceeding the allowable limit. This approach involves gradual, selective sampling, targeting deliberately those parts of the product that are either known, or are likely to contain certain substances. Each sampling phase could be followed by analysis. If the results show no certain 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 certain substance in any part, the product as a whole is deemed non-compliant and no further sampling and analysis are necessary. Annex B provides a list of components which currently have a probability of the presence of one or more of substances of concern.

Another strategy is to prove total compliance of the product, as far as possible down to the homogeneous material level. This approach would be typical for 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, feasibility of the testing is performed (e.g. is the sample mass/size/volume sufficient?). Further sampling and disjointment may be necessary, where a choice can be made to either completely disjoint or only select materials with a high probability of containing certain substances. Table B.1 is used to assist in the identification of these components and materials.

If testing is appropriate, the relevant testing procedure shall be followed. Where certain 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 depend on the objective of the sampling strategy. After the screening steps, further analytical testing is undertaken as necessary.

5 Sampling

5.1 Introductory remark

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

Before sampling, the following questions need to be addressed:

a) Based on available knowledge of the product, can any sections/parts be identified that are likely to contain certain substances (see Annex B)?

b) Is it practical to analyse any of these sections/parts without disassembly?

c) Can the section/part selected be regarded as homogeneous material for the purpose of the analysis?

d) Is the section/part selected for analysis representative?

e) Does the sample selected fulfil the criteria of minimum mass, area, thickness or volume required by the chosen analytical methods?

The answers to these questions will determine the sampling strategy and the extent of disassembly and disjointment, if any, required to provide representative samples that are homogeneous and of sufficient quantity to permit a valid analysis.

Whenever possible, sampling shall 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 (see IEC 62321-3-1 and IEC 62321-3-2). Depending on the results of the assessment and objectives of the analysis, further disassembly, disjointment and sampling shall be performed, as required, especially for verification analysis of the product’s components and materials. This approach offers the least expensive, fastest and the most efficient means of analysis, especially when undertaken on the finished product.

The numerous types and diversity of electrotechnical products make it impractical to provide detailed sampling strategies for each one. Instead, procedures covering five increasingly destructive levels of sampling are described.

Annex A provides generic sampling flowcharts based on Figure 1 for certain 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.

When determining certain substances it is strongly recommended to ensure that the tools used are free from the substances of interest to avoid possible contamination.

Annex E gives examples of the disassembly and disjointment, covering for two types of mobile phone, along with the disjointment of various small electronic components, in particular integrated circuit lead frame and ball grid array (BGA) packages.

5.2 Complete product

Sampling of the complete product is the first, non-destructive step in the iterative sampling strategy where representative parts of the product can be analysed in their present form without disassembly or disjointment.

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 TV set. Complete products may be evaluated without disassembly if they have a very simple construction, or if the locations of expected certain 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.

NOTE Even a seemingly simple product such as a power cord may be composed of perhaps 10 to 20 different homogeneous materials.

5.3 Partial disassembly

The product is disassembled down to its major components and subassemblies and tested non-destructively, where possible. See Annex E.

5.4 Complete disassembly

Complete disassemby separates all components as far as possible, while still permitting reassembly to give an operational product. See Annex E.

5.5 Partial disjointment

Frequently, the detailed analysis of a complete electrotechnical product for certain substances requires disassembled 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 and partial disjointment of subassemblies and components shall focus on those parts with a high probability of containing certain 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 certain substances within them.

After disjointment, the separated subassemblies and/or components will not be able to reassembled into an operational state.

Subassemblies and components form discrete parts within complete electrotechnical products such as mobile 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.

5.6 Complete disjointment

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

Before attempting the “complete disjointment” of complex components into their homogeneous materials, professional experience, skill and non-destructive analysis can help to determine their internal structure and materials. This can be achieved by

- professional knowledge,
- 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, caution shall be exercised when interpreting the results.

5.7 Considerations of sampling and disjointment

5.7.1 Introductory remark

In 5.3 to 5.6 disassembly and disjointment were discussed from the high level down to the detailed level. At the detailed level, certain 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.

Sampling and analysis of certain substances at the homogeneous material level are often difficult and a proper understanding of the basis of interpretation is crucial, particularly when dealing with composite samples.

5.7.2 Sample size required

Based on practical experience the following minimum sample amounts are recommended for analysis:

- IEC 62321-3-1
  - Strongly dependent on the instrument, geometry and material. For polymers and aluminium samples, a depth of several millimetres may be required, whereas for other metals the thickness can be approximately 1 mm or less.
- IEC 62321-3-2
  - 10 mg for $<$ 10 mg/kg minimum detection limit (MDL)
- IEC 62321-4
  - 0,5 g for $<$ 5 mg/kg minimum detection limit (MDL)
- IEC 62321-5
  - 0,5 g for $<$ 50 mg/kg MDL
  - 1 g for $<$ 5 mg/kg MDL
- IEC 62321-6 [5]
  - 100 mg for $<$ 100 mg/kg MDL per congener group
- IEC 62321-7-1 [6]

  - 50 cm^2 for < 0,02 $\mu g/cm^2$ MDL for chromate coatings on metals
- IEC 62321-7-2 [7]
  - 2,5 g for < 15 mg/kg MDL for polymers and electronics.

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

For small components, it is often impractical to obtain enough sample mass to conduct analytical testing. For illustrative purposes, Table 1 summarizes how weights in a typical industry lead frame material declaration report as well as the minimum sample size of IEC 62321 test methods are 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 Table 1.

Table 1 – 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	368	N/A	N/A
Die attach [b,c,d,e,f]	0,7	893	N/A	179
Lead frame [b,c,d,e,f]	42,0	15	N/A	N/A
Lead frame plating [b,c,d,e,f]	0,6	1 042	Unknown	N/A
Mould compound [b,c,d,e,f]	52,9	12	N/A	3
Silcon die [b,c,d,e,f]	6,0	104	N/A	N/A

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, according to 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 the IEC 62321 series.

[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 1 shows that theoretically 15 samples are required for lead testing of the lead frame material layer. However, as discussed in Annex E, 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 1 shall be viewed as the "best case" or "theoretical minimum".

Based on the above recommendations, to run all six of the certain substance tests per the IEC 62321 series requires a minimum sample mass of 3,6 g. However, depending on the material, there may be as much as a 20 % loss during the mechanical sample preparation (see Clause 7) before testing. Therefore, 3,6/(1 – 0,2) = 4,4 g could be the actual minimum sample amount to be obtained from the parts or components to analyse for all six given substances. Flame retardants are not typically used for metallic material layers, so for the analysis of only these materials the minimum sample size can 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 and/or specification remain the same. Consequently, the analytical result could be a misleading mean of the samples tested (diluting high levels of a certain substance in only a few of the samples) or even be inconclusive.

Hence, sampling large numbers of small components, such as those shown in Table 2 and the lead frame example in Table 1 can make definitive analytical testing 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?

5.7.3 Sample size versus detection limit

There is an inverse relationship between sample size and detection limit. As the amount of material available to analyse decreases, the method 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 is helpful to specify a minimum size beyond which disjointment is no longer required. Components smaller than this minimum size are pulverized and the resultant material analysed and treated as a "homogeneous material" for technical limitations of sampling and analysis, even though it is actually a composite of several homogeneous materials. In this case the detection limit issues discussed in Table 2 become important. It should be noted that setting a minimum sample size at a certain level does not solve certain 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 7) and the analytical method. For example, when testing for lead in polymers by ICP-MS using dry ashing as the sample preparation procedure a 0,02 g sample is required for a 1 mg/kg reporting limit, or when using ICP-AES 0,2 g sample is required for a 5 mg/kg reporting limit.

In general, every combination of matrix, sample preparation technique 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, using ICP-AES, 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 technique and analytical method used to determine a certain substance.

5.7.4 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 the presence of a certain given substance so as to avoid unnecessary testing (see Table B.1). Ignoring these considerations could lead to false positive results. 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 can 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 given 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 2. When expressing the concentration of the total composite material this can be below the tolerable level (e.g. 1 000 mg/kg), while the results on the basis of the homogeneous material may well exceed the tolerable level. An example of such a situation is depicted in Table 2, where material A contains a significant level of lead (Pb) which is diluted in the overall lead result of the mixed composite sample. Further considerations for composite samples are covered in Annex C.

Table 2 – Levels of a certain substance in a composite sample

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

5.7.5 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 may be necessary to homogenize such samples and treat them as a single “homogeneous” material for testing purposes. However, far more knowledge of the component’s structure and materials is required to obtain valid analytical results.

When the component is prepared (e.g. ground into a powder) and tested as a single sample the analytical result will be the mean level of certain substances present with respect to 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 2 for a resistor that cannot be mechanically disjointed, but contains lead in restricted (lead-based plating, upper cross-section) and exempt (lead oxide (PbO) glass, lower cross-section) applications. The composite sample from this non-disjointable component cannot distinguish between the different sources of lead. Hence it is not possible to determine the lead concentrations in each of the homogeneous materials by testing the resistor as a composite sample. In essence this holds true for several sizes of components that cannot be mechanically disjointed.

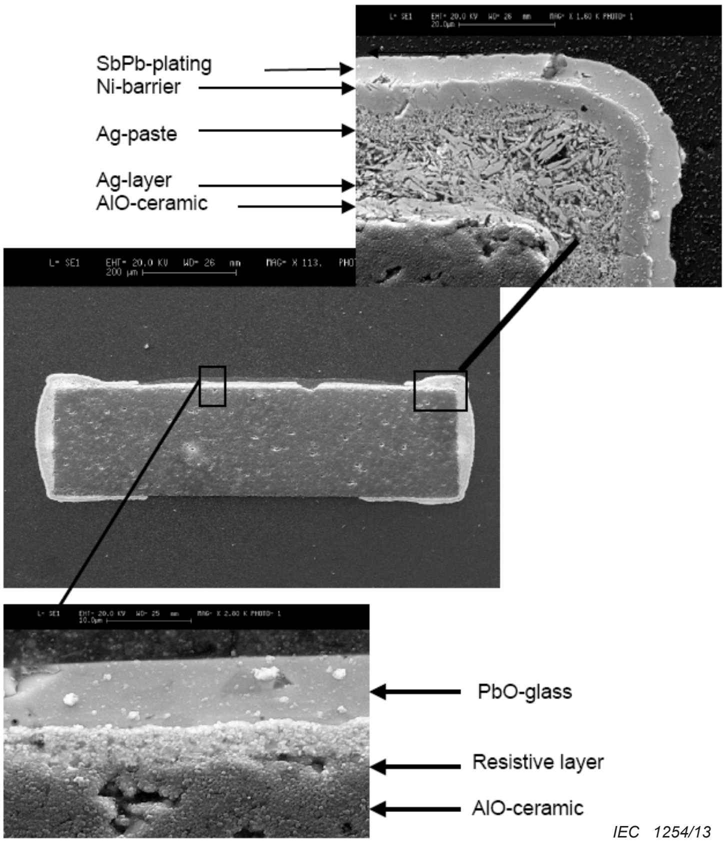


Figure 2 – Cross-section of a 900 µm wide lead oxide-based resistor (SMD)

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

Strategies need to be developed to address these challenges, particularly as electrotechnical products continue to shrink in size and grow in complexity, while the number of certain substances increases and the permitted limits fall. Screening techniques are used to gather more information on the certain substances, and it may be possible to analyse materials in-situ as a cross-section, as shown in Figure 2 (see IEC 62321-3-1 and IEC 62321-3-2). However, it remains difficult to distinguish between exempt and restricted forms of the same substance.

5.7.6 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 shall be made at several locations. For example, sampling positions shall be selected from different areas, including at least one geometric centre and the ends of two diagonals.

6 Conclusions and recommendations for sampling

The strategy and process of sampling are critical preliminary steps in the valid analysis of certain substances in electrotechnical products. The quality of analytical results and specifically their representativeness of the analysed sample are directly affected by the sampling technique. Selection of the sampling strategy depends on the substance(s) to be determined, their allowable limits, the basis on 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 detail in Clause 4, concluding that the selection of the sampling strategy is driven by the objectives of the analysis, which shall be born 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 shall 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 and Annexes A and E (see also IEC 62321-3-1) show that the proper sampling strategy and intelligent use of analytical techniques can substantially reduce the amount of work required.

This standard illustrates that sampling for the analysis of certain 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 2). This scenario illustrates the deficiency of the definition of homogeneous material. Where no further mechanical disjointment is possible, analytical testing alone cannot determine whether the sample complies with certain 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 may be insufficient for a meaningful analysis. To alleviate this difficulty, sampling strategies shall prudently utilize information from material declarations and knowledge of the probability of the presence of a certain substance in a specific material type (see Table B.1). Subclause 5.7 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 shall also be aligned with the objectives of the analysis.

7 Mechanical sample preparation

7.1 Overview

7.1.1 Field of application

This standard provides strategies on processing selected portions of an item. Common techniques are covered for mechanical size reduction of electrotechnical products, their sub-units or portions thereof, prior to the determination of certain substances.

NOTE Sample handling and preparation for specific analytical methods are addressed in other parts of the IEC 62321 series.

The user shall elect to apply one or more of the approaches, or an equivalent, to create samples suitable for analysis. Selection of the appropriate technique(s) depends on the required particle size for the analytical method to be used. Alternative methods of mechanical sample preparation can be used, provided that the required particle size of the sample is achieved without contaminating or compromising the sample with certain substances.

7.1.2 Quality assurance

Due to the risk of analytical bias resulting from contamination, evaporation of volatile components (e.g. volatilization due to heat) or from loss of material through dust emissions, it is important to select the appropriate equipment and cleaning procedures.

Contamination can be caused by the grinding equipment and any accessories that contact the sample. For the chosen equipment, it shall be known which elements may be released that will contaminate the analysis sample, e.g. cobalt (Co) and tungsten (W) can be released from tungsten carbide (WC) equipment, and chromium (Cr), nickel (Ni), molybdenum (Mo) and vanadium (V) can be released from stainless steel equipment.

The laboratory shall demonstrate by experiment that a mechanical process does not result in contamination by, or loss of, detectable amounts of certain substances. Similarly, that the procedure employed for cleaning the mechanical sample preparation equipment prevents contamination of the sample with certain substances from the previous sample. For example, processing and analysing certified reference materials and blanks before or after processing a material known to contain significant levels of certain substances may be necessary. Use of certified reference materials is not mandatory. However, the materials used shall have a known regulated substance content to determine that the mechanical sample preparation and cleaning processes do not cause contamination or loss of regulated substances. The effectiveness of the mechanical sample preparation technique shall be continuously monitored by using quality control practices, including matrix spikes or control samples.

7.2 Apparatus, equipment and materials

The following apparatus, equipment and materials are required, subject to the type of material being prepared:

a) coarse grinding or cutting mill with 4 mm and 1 mm or similar stainless steel bottom sieve;

b) centrifugal mill with 250 μm tungsten carbide-coated (WC) steel sieve, and a 6-fold WC-coated rotor (for uniform plastic material a 1 mm steel sieve is appropriate). To avoid the risk of introducing impurities during milling, a 1 mm titanium sieve and a steel/titanium sieve rotor shall be used;

c) “freezer” bladeless cryogenic impact grinder/mill with self-contained LN_2 tub, insulated case, speed control, programmable timer and safety interlock;

d) homogenizing mixer (e.g. blender);

e) analytical balance capable of weighing accurately to 0,000 1 g;

f) brushes (different sizes);

g) paper;

h) scissors, heavy plate shears;

i) glass beaker;

j) liquid nitrogen (LN_2).

   Liquid nitrogen is quite volatile and can cause oxygen deficiency in the area of use, especially if the area is enclosed. The laboratory is responsible for ensuring that the proper safety procedures are followed, and that protective equipment is used during cryogenic grinding.

k) powder funnel.

l) gloves;

m) safety glasses;

n) polyethylene receptacle (for use with LN_2).

7.3 Procedure

7.3.1 Manual cutting

Manual cutting is suitable for rough cutting and preparation of samples for further reduction by grinding, etc. Recommended maximum sample sizes are listed below, but will depend on the specification of the equipment used in the subsequent preparation processes:

a) electronics: samples are pre-cut to a size of 40 mm × 40 mm using heavy plate shears (see 7.2 h);

b) metal sheeting: samples are pre-cut to a size of 40 mm × 40 mm using heavy plate shears (see 7.2 h).

c) polymers: samples are pre-cut to a size of 5 mm × 5 mm using heavy plate shears or scissors (see 7.2 h). Thin polymer foil shall be cut into small pieces with shears (see 7.2 h).

7.3.2 Coarse grinding/milling

Coarse grinding is suitable for reducing samples to approximately 1 mm in diameter. Cool the samples if needed with the LN_2 (7.2 j). For organic samples, cryogenic milling is recommended. An example of cryogenic preparation is to put the samples in a polyethylene receptacle (7.2 n) to cool with LN_2 (7.2 j). Wait until the LN_2 (7.2 j) has dissipated, plus an additional 10 min thereafter. Grind the samples in the mill (7.2 c) using a 4 mm stainless steel bottom sieve. During grinding, maintain a sample temperature of < -20 °C. Carefully sweep out and collect all particles. Refit the mill (7.2 c) with a pre-weighed 1 mm stainless steel bottom sieve and reprocess the 4 mm material. Carefully sweep out the mill (7.2 c) and collect all particles. Use a 5 min cooling period between grinding cycles.

NOTE It may only be possible to mill metallic materials to a particle size of 4 mm (although 1 mm particles are preferred).

7.3.3 Homogenizing

Homogenizing is suitable for preparing the coarsely ground sample in the mixer prior to further size reduction in the centrifugal mill (7.2 b). Use a container with double the capacity of the amount of powder to be mixed. Set the mixer (7.2 d) to an appropriate speed and mix the powder until it is homogeneous.

7.3.4 Fine grinding/milling

Fine grinding or milling is suitable for reducing samples to <1 mm in diameter.

For organic samples that have no metal parts, cryogenic milling (7.2 c) is recommended. Be careful not to allow the LN_2 (7.2 j) to come into direct contact with the powder in order to prevent spattering and sample loss, e.g. by using a polyethylene receptacle (7.2 n).

Mill the sample powder with the centrifugal mill (7.2 b). Carefully sweep out the centrifugal mill (7.2 b) and collect all the powder. The collected material shall be sieved to obtain a sufficiently homogeneous portion of known particle size range.

7.3.5 Very fine grinding of polymers and organic materials

This procedure is suitable for the reduction of samples as small as 500 μm in diameter or less. It is not suitable for metal, glass or similar hard and sharp materials.

Between 3 g and 10 g of rough-cut (3 mm to 5 mm sections) are placed in the sample vial so that it is two-thirds to three-quarters full. Add the grinding rod and secure the ends of the vial. Cool the bladeless cryogenic impact grinder (7.2 c) at room temperature for 15 min by filling the reservoir with LN_2 (7.2 j). Place the grinding vials with the samples in the mill (7.2 c) and lock the cover into place. One or more sieves shall be added to ensure a sufficiently homogeneous sample.

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 (Figures A.1, see also Figure 1) along with several worked examples:

- sampling of DVD player (Figure A.2);
- sampling of CRT (Figure A.3);
- sampling of LCD TV (Figure A.4);
- sampling of PDA/phone (Figure A.5);
- sampling of desk fan (Figure A.6);
- sampling of components – thick film resistor (Figure A.7);
- sampling of components – SMD potentiometer (Figure A.8).

The following points shall be considered in conjunction with the flowcharts.

a) Analysis can be 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 the presence of certain substances, an enforcement authority pursuing particular intelligence);
   - screening as part of an incoming goods inspection;
   - the initial stage of a comprehensive review of the whole product.

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

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

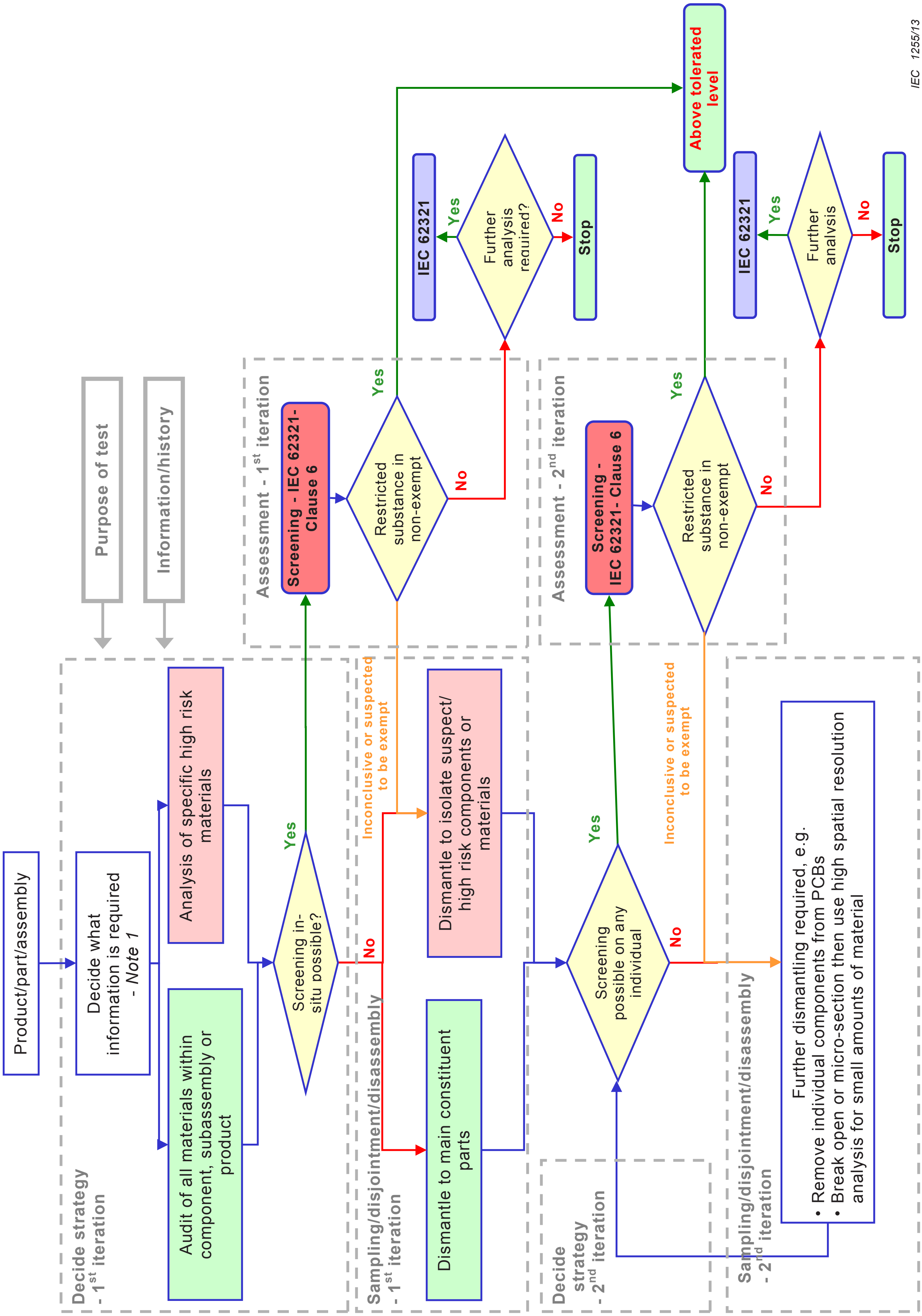


Figure A.1 – Methodology for sampling and disjointment

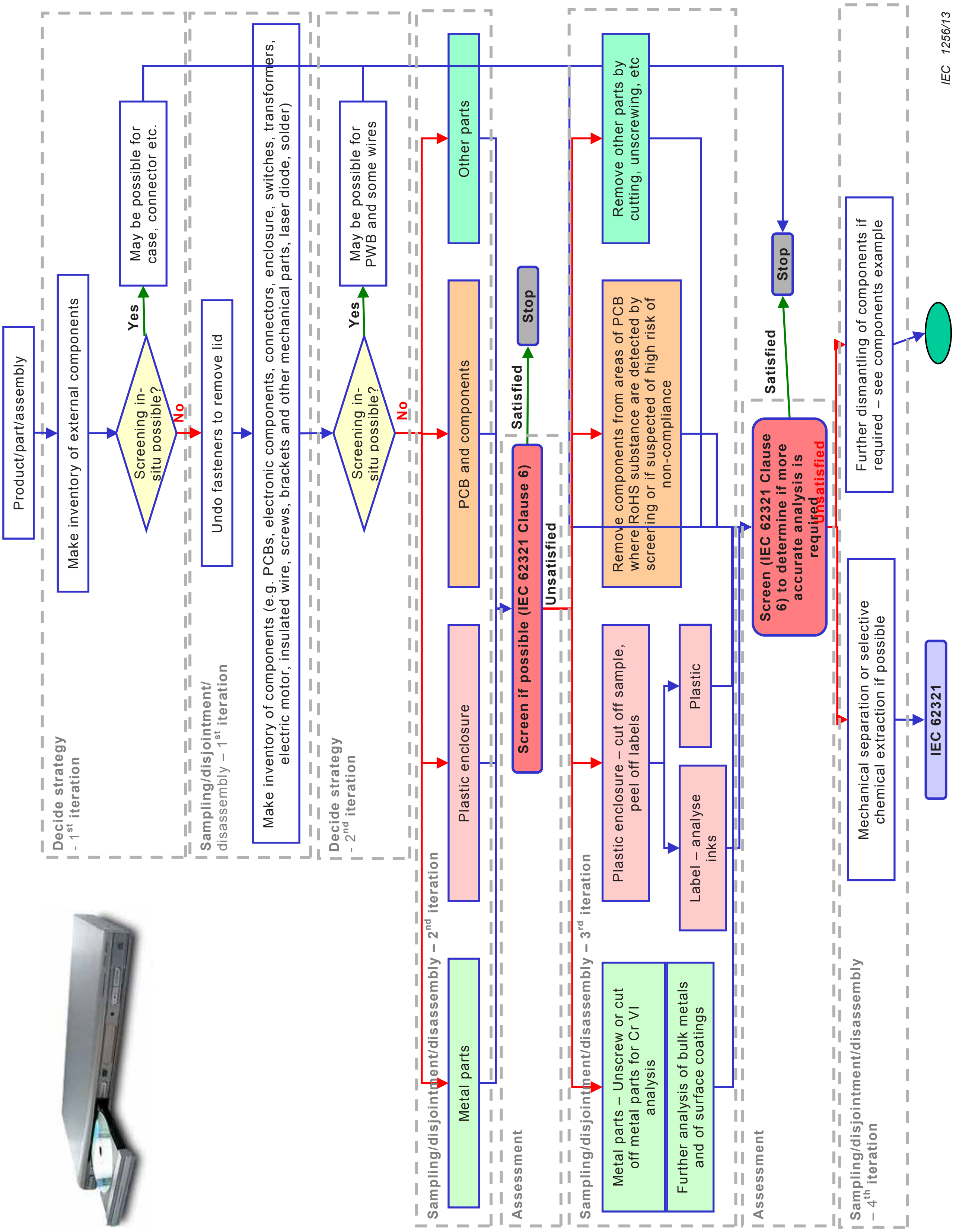


Figure A.2 – Sampling of DVD player

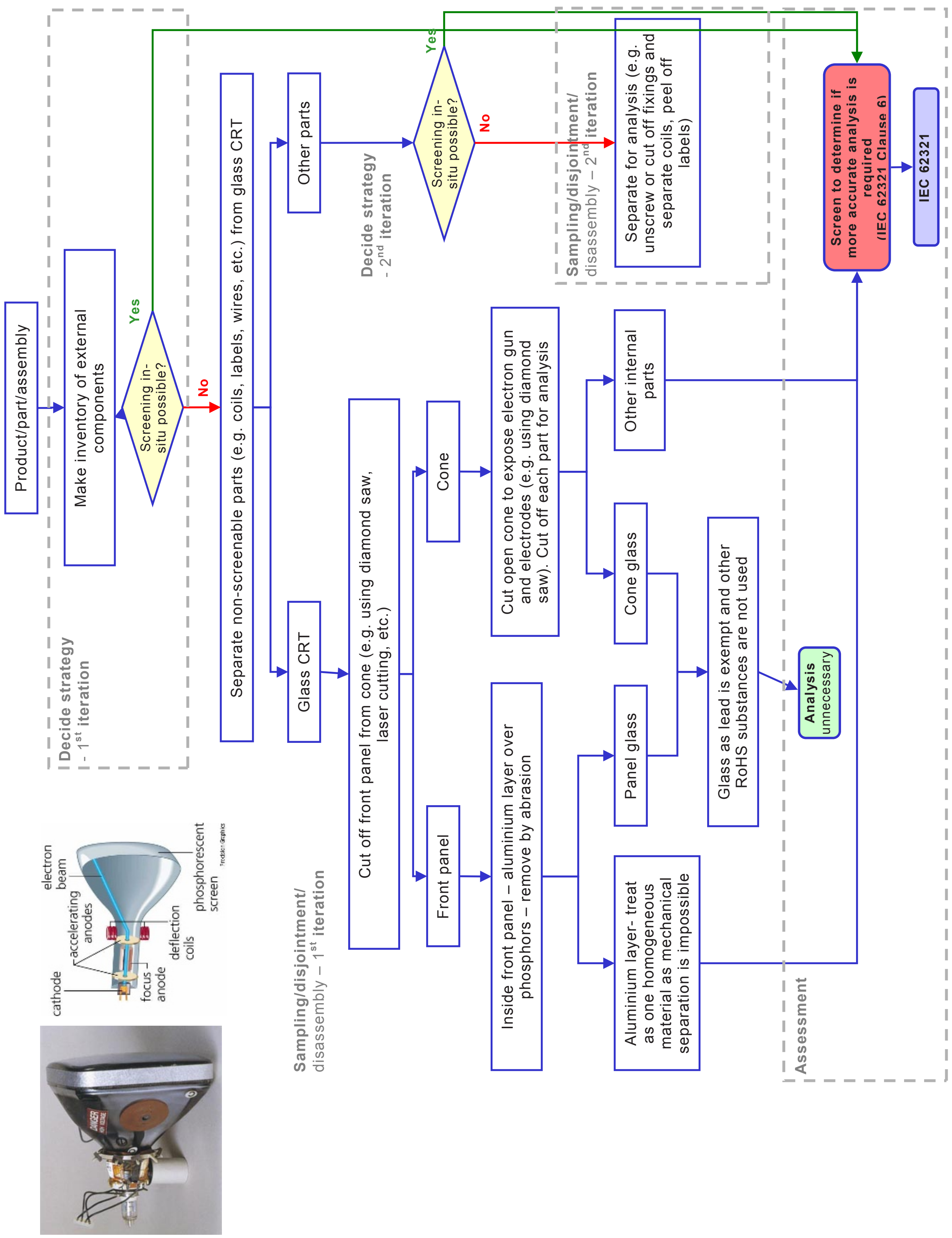


Figure A.3 – Sampling of CRT

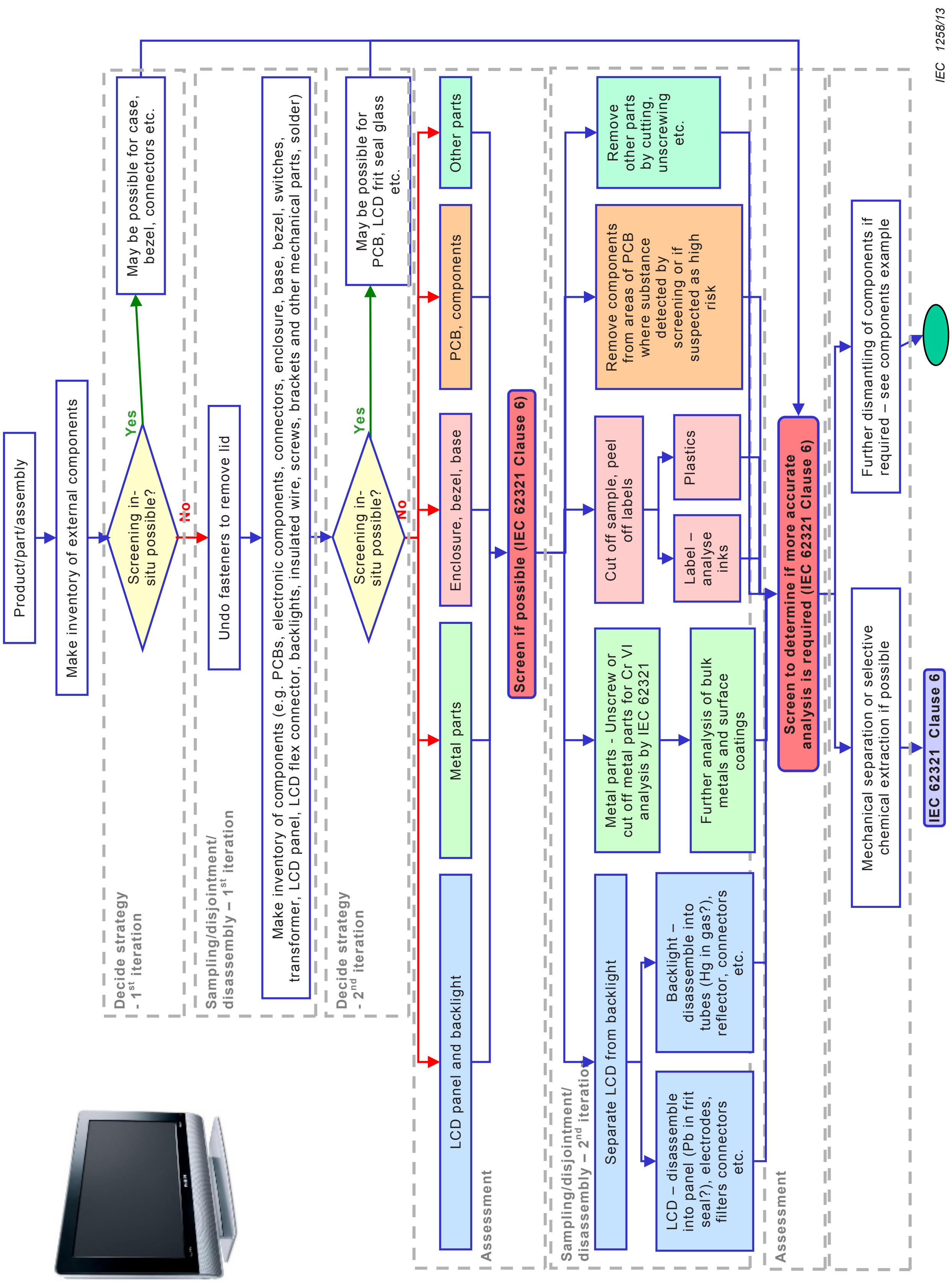


Figure A.4 – Sampling of LCD TV

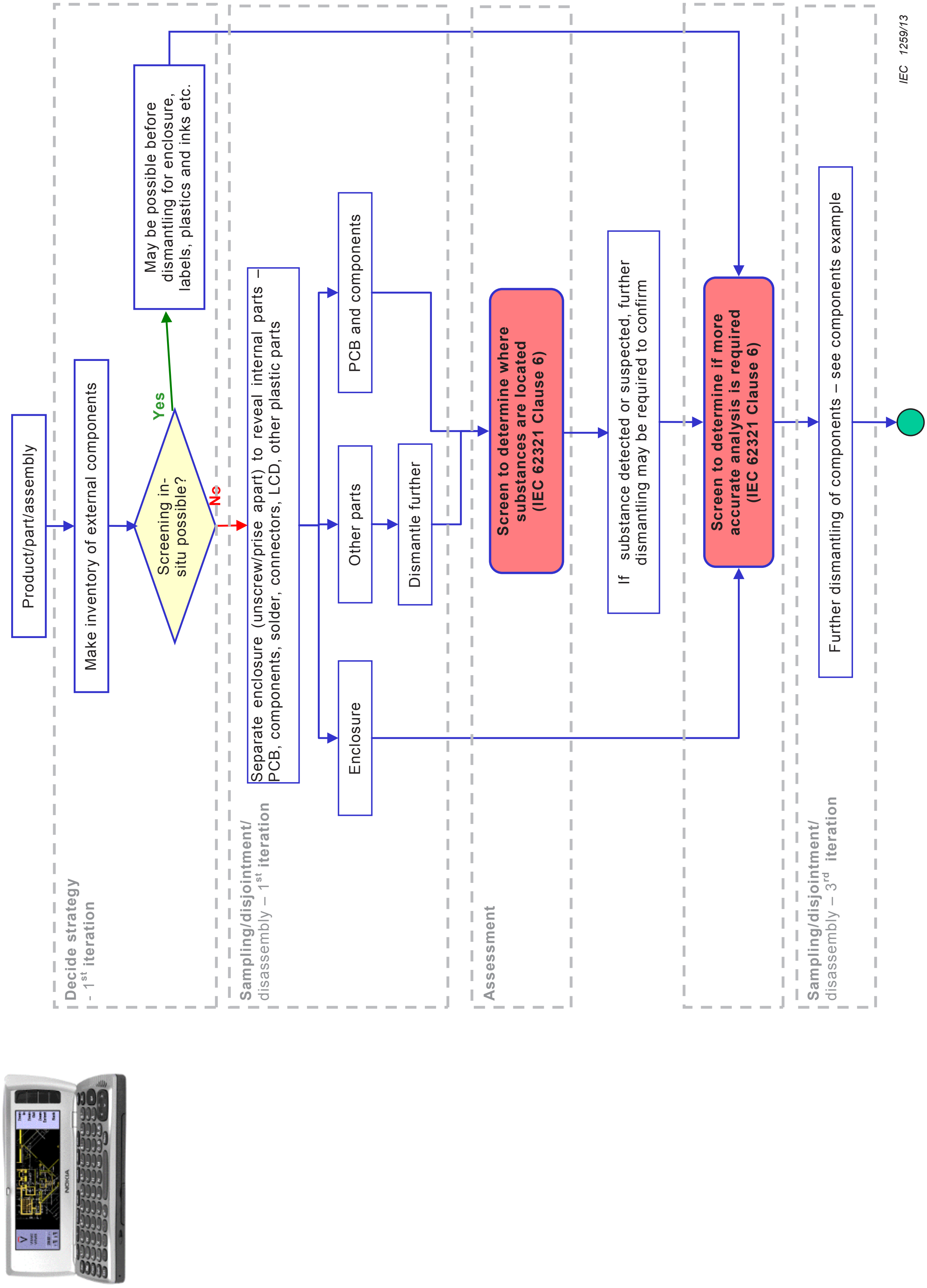


Figure A.5 – Sampling of PDA/phone

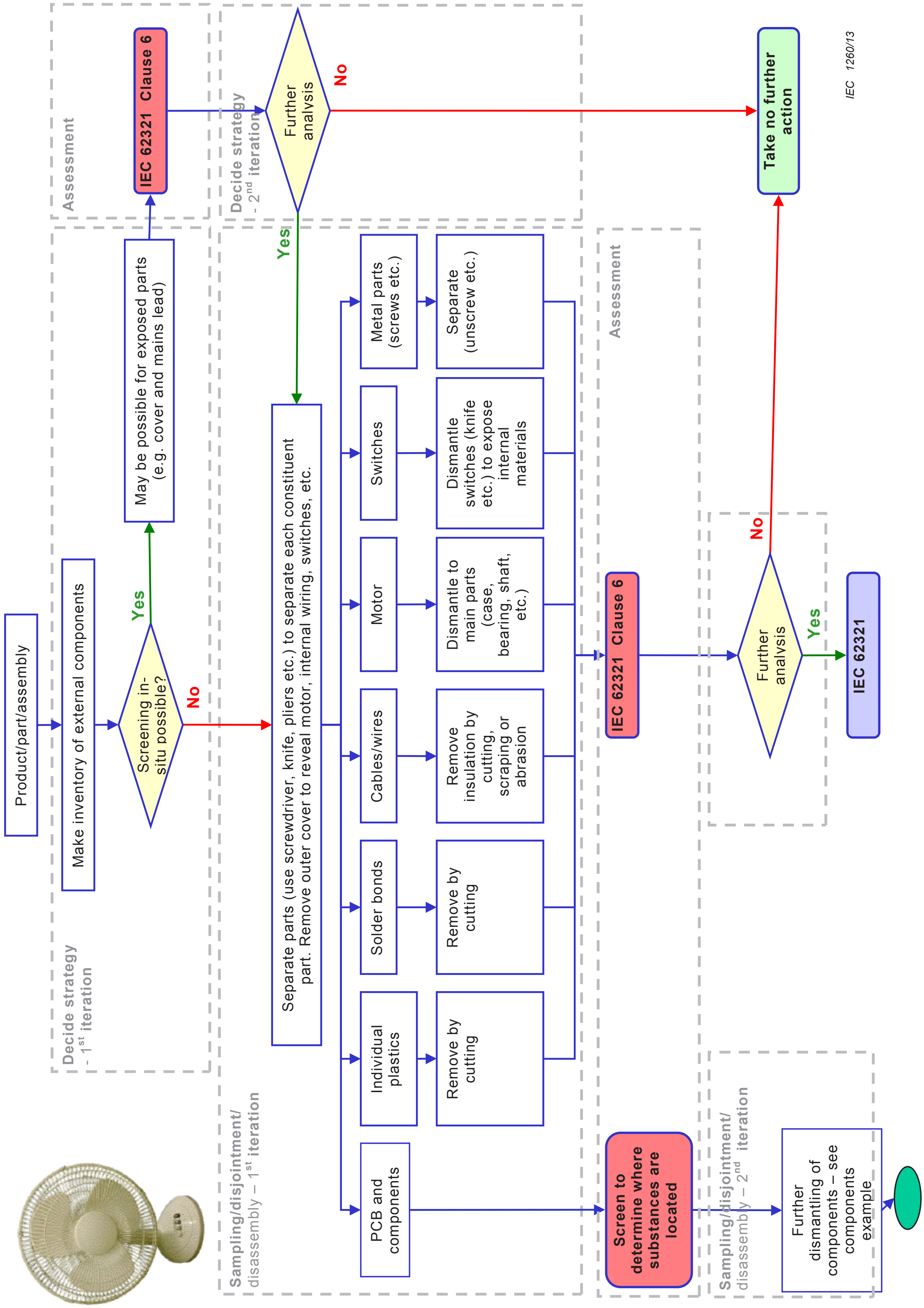


Figure A.6 – Sampling of desk fan

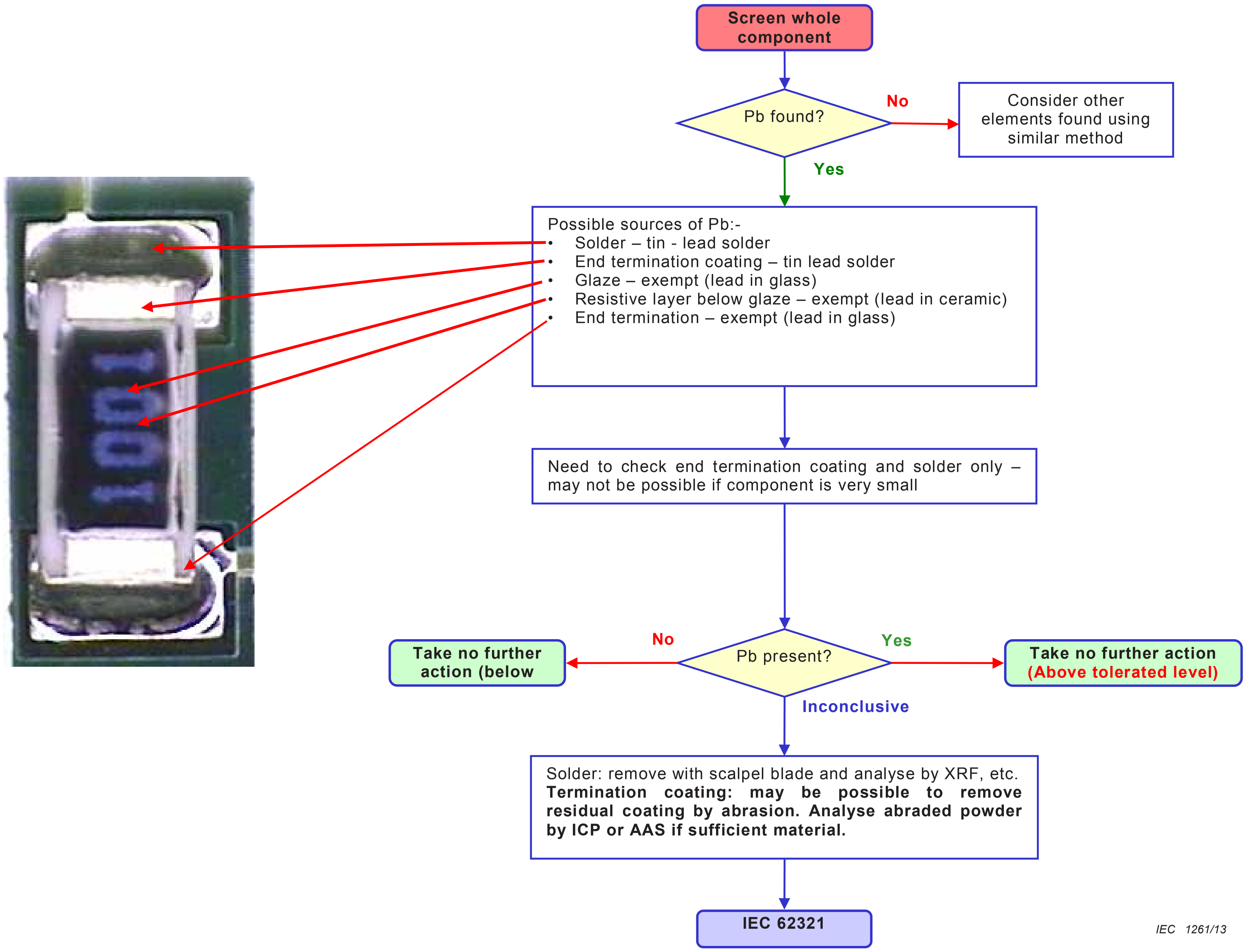


Figure A.7 – Sampling of components – Thick film resistor

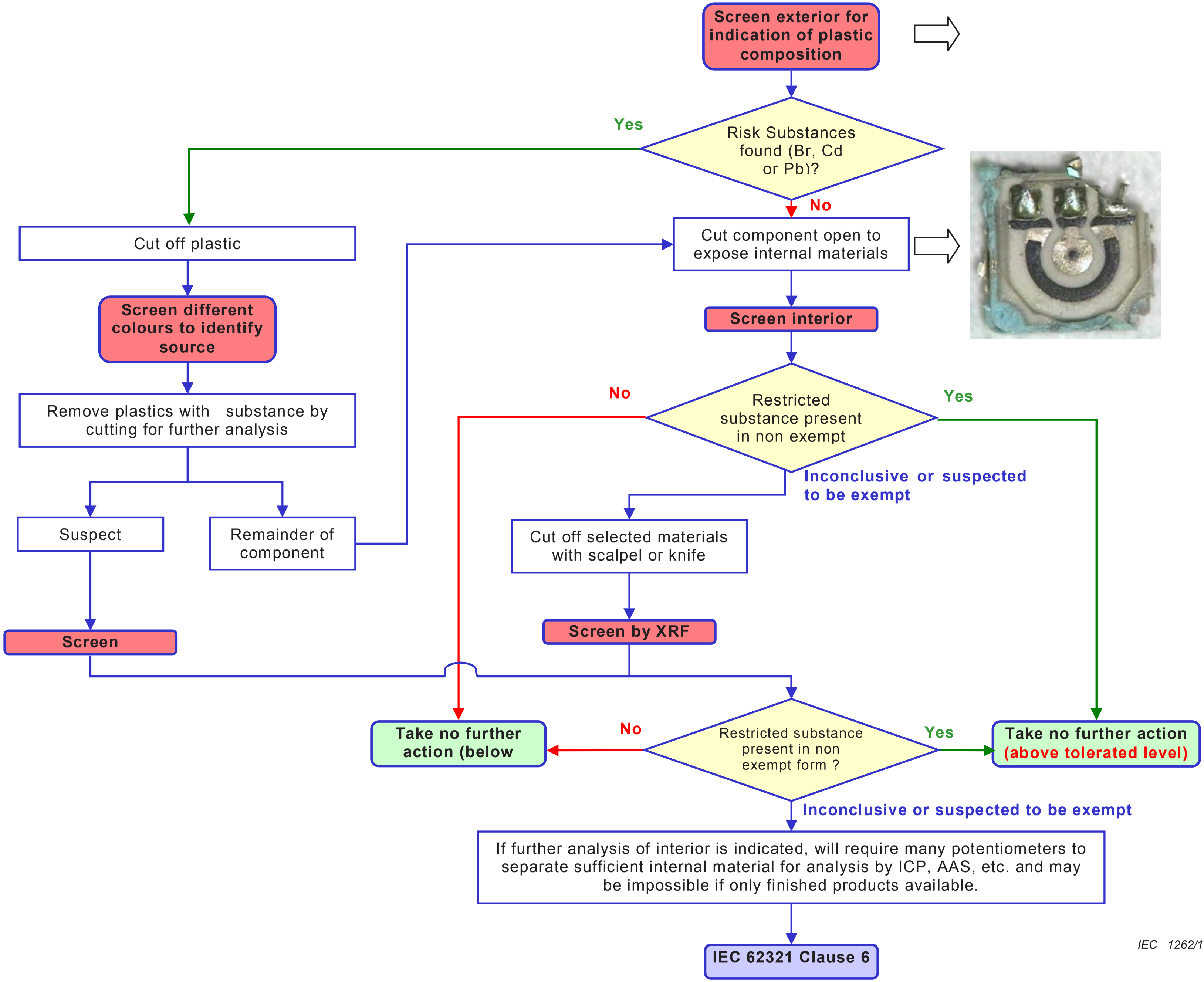


Figure A.8 – Sampling of components – SMD potentiometer

Annex B
(informative)

Probability of the presence of certain substances

B.1 Introductory remarks

Many electrotechnical products contain a large number of different types of materials and substances. Thus, having knowledge of the presence or absence of certain substances in the components and materials of electrotechnical products would be advantageous for compliance verification, since it would allow for optimization of the sampling and analysis as described in Annex A. Having an understanding of the following can provide guidance in the correct sampling and testing approach:

- the function of a substance in a material set;
- the chemical compatibility of a substance in its matrix material;
- the chemical transformations that occur during processing/manufacturing of a substance/ material;
- and other considerations.

Efficiencies are gained by eliminating testing for substances that are known to be absent in a particular material or component e.g. PBB/PBDE flame retardants in metal components. Materials and components that have a higher probability for the presence of certain substances would generally require more frequent and extensive analysis.

Table B.1 provides an overview of the typical materials and components in electrotechnical products with their probability for the presence of six substances of concern. It provides an initial indication of the relevant areas for sampling. This table is not intended to be exhaustive, since new materials are constantly being introduced as a result of technology and product innovations.

Table B.1 – Probability of the presence of certain substances in materials and components used in electrotechnical products *(1 of 3)*

Components and materials	**Certain 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	

Table B.1 *(2 of 3)*

Components and materials	Certain substances[a]						Number of homogeneous materials[b]	Remarks
	Hg	Cd	Pb	Cr (VI)	PBBs	PBDEs		
Printed wiring board (PWB)								
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	
Component mouldings	L	L	L	L	L	H	1 and >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	

Table B.1 *(3 of 3)*

Components and materials	Certain substances[a]						Number of homogeneous materials[b]	Remarks
	Hg	Cd	Pb	Cr (VI)	PBBs	PBDEs		
Materials								
Paint, ink and similar coating	L	H	H	M	L	L	1	
Adhesive			M		L	M	1	
Polyurethane – high gloss	H	M	M	L	L	M	>1	
Polyvinyl chloride (PVC)	L	H	H	M	L	M	1	
Styrene, polystyrene (PS-HI), 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 should be used as guidance to assist in selecting components/materials for testing that have a high probability of containing a certain 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 – No historical use of the certain substance.
M Medium probability – Historical use of the certain substance, but alternative substances are currently used.
H High probability – Historical use of the certain substance for which there are no known alternatives, or alternatives are not commonly used.
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 remarks

As discussed in 5.7.3, composite testing of a product made up of several different parts and/or material layers may be used as an effective screening technique. Under certain circumstances this technique can allow for a more efficient utilization of samples for cost savings by elimination of superfluous testing. There are two approaches that can be taken:

a) calculating the maximum sample concentration based on the analytical detection limit of the method used;

b) calculating the required analytical detection limit to ensure that a substance is below a certain concentration level in a sample.

The first approach estimates the amount of an analyte that could be present, but would not be detected because it was diluted below the detection limit as a result of compositing.

C.2 Calculated maximum concentration for a composite sample based on detection limit

This first example 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 composite (material A) contains certain substances, lead (Pb) and cadmium (Cd);
- the method of chemical analysis on the composite sample has a detection limit of 20 mg/kg;
- for chemical analysis, the component/part is first ground to a powder (a homogeneous sample).

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

The conclusion for this composite sample is that further analysis is needed for cadmium 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
A	1	6	360	360
B	4	22	0	0
C	5	28	0	0
D	8	44	0	0
Total (composite)	18	100		
Mean (composite)			20	20

In the second approach, the required detection limit is calculated to ensure that particular substance is not present above a certain level.

When a substance is detectable for a composite sample, the concentration of the substance containing in the homogeneous material A can be calculated as follows:

$$C_{\mathrm{A}} = \frac{\mathrm{MDL} \times m_{\mathrm{C}}}{m_{\mathrm{A}}}$$

where

C_{A} is the concentration of a substance containing in the material A (mg/kg);

MDL is the detection limit of the method of chemical analysis on the composite sample;

m_{C} is the weight of the composite sample (mg);

m_{A} is the weight of the material A (mg).

C.3 Required detection limit for a composite sample based on the maximum allowable concentration

This second example (see Table C.2) is based on the following presumptions:

- contamination of the same composite sample used in the example given in Clause C.2 with 1 000 mg/kg lead (Pb) and 100 mg/kg cadmium (Cd).

To verify these levels of lead and cadmium by chemical analysis would require a method with a detection limit of 50 mg/kg for lead and 5 mg/kg of cadmium, 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 sample** %	**Pb content** mg/kg	**Cd content** mg/kg
A	1	6	1 000	100
B	4	22	0	0
C	5	28	0	0
D	8	44	0	0
Total (composite)	18	100		
Mean (composite)			56	5,6
Required detection limit/ mg/kg			56	5,6

The required detection limit for a composite sample can be calculated as follows:

$$\mathrm{MDL} = \frac{C_\mathrm{L} \times m_\mathrm{A}}{m_\mathrm{C}}$$

where

MDL is the required detection limit for the composite sample (mg/kg);

C_L is the maximum allowable concentration of a substance containing in the material A;

m_A is the weight of the material A (mg);

m_C is the weight of the composite sample (mg).

Further, it is important to keep in mind that both the detection limits of analytical methods and the concentrations of certain substances have a margin of error and the presence of certain substances can vary within a “homogeneous material” (see IEC 62321-3-1 and IEC 62321-3-2). Therefore it is advisable to include a margin of safety when applying this concept.

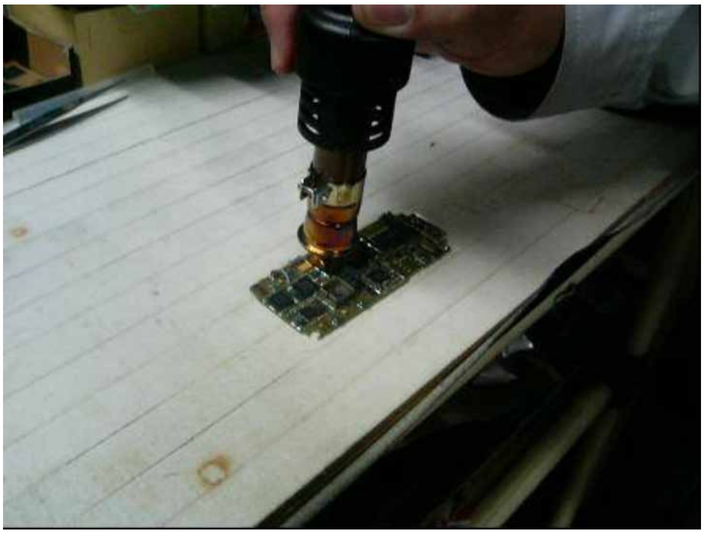
Annex D
(informative)

Tools used in sampling

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

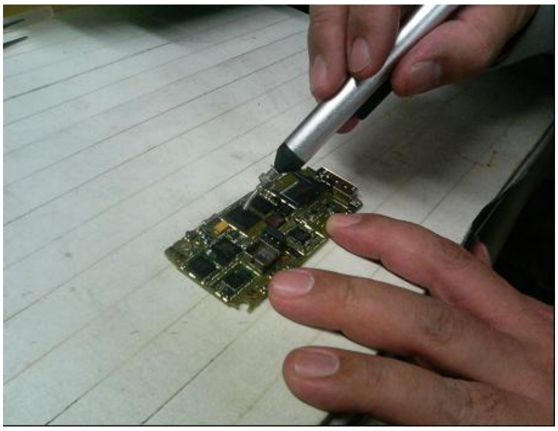
- Soldering iron
- Screw driver (electrical)
- Cable stripper
- Knife/scalpel
- Shears
- Wrenches (open ended/ring)
- Hammer
- Drill
- Solder wick, i.e. wire that sucks up molten solder
- Allen-keys
- End-cut pliers
- Pliers
- Hand saw
- Tweezers
- Plastic bags

Use of a hot gas gun (Figure D.1) and vacuum pin (Figure D.2) are shown below.



IEC 1263/13

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



IEC 1264/13

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

Annex E
(informative)

Examples of mobile phone disassembly and component disjointment

E.1 General

The mobile phone is a compact and complex product containing a large number of small components. Therefore, it offers a good example of how sampling strategies can be developed for other electrotechnical products.

This annex provides the following examples:

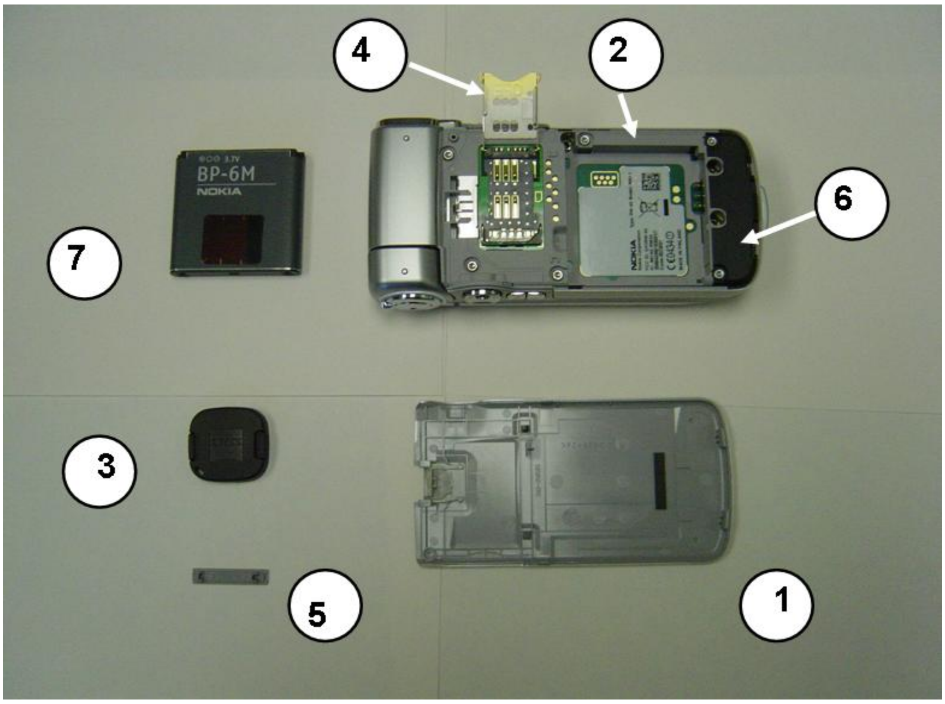
- partial disassembly without tools – mobile phone type A;
- partial disassembly with simple tools – mobile phone type B;
- complete disassembly – mobile phone type B;
- partial disjointment – mobile phone type B;
- complete disjointment – examples of disjointment of small electronic components;
- complete disjointment of integrated circuit lead frame package;
- complete disjointment of ball grid array (BGA) package.

E.2 Partial disassembly without tools – Mobile phone type A

IEC 1265/13

Figure E.1 – Mobile phone type A with battery charger and camera lens cap

This mobile phone (Figure E.1) offers limited opportunities for analysis as a complete product. The charger includes at least six individual areas that are made of different materials, and is screened without disassembly. The phone back cover is removed and the phone battery taken out. As the back cover is metallized on the outside it is analysed (screening) on both sides. This partial disassembly of the mobile phone is the limit of disassembly without the use of tools, as shown in Figure E.2 and listed in Table E.1.



IEC 1266/13

Figure E.2 – Mobile phone type A with battery and back cover removed

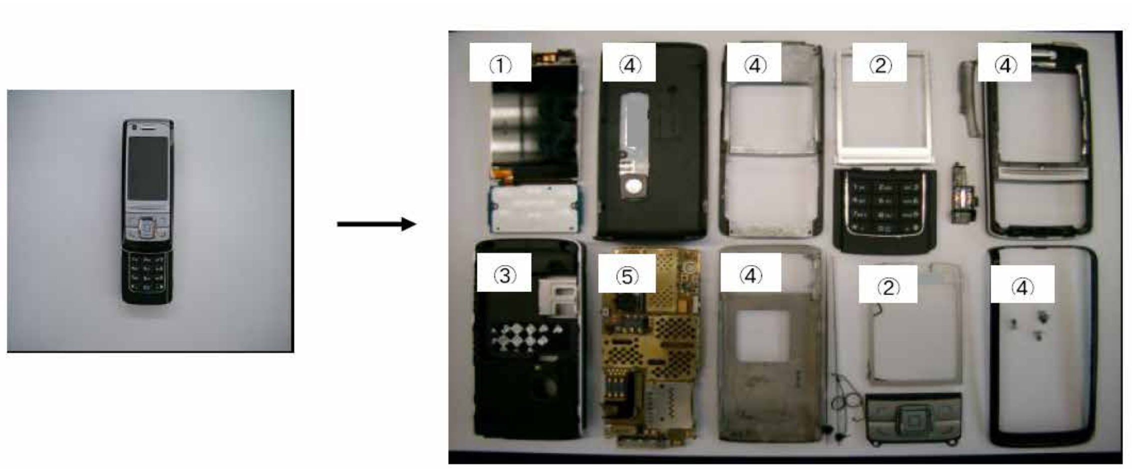
Table E.1 – Possible certain substances or screening substances from a mobile phone

Sample number	Component/assembly	Materials	Probability of presence[a]	Elements related to certain substances[b]	Options for further 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 E.2 can be screened directly. Subclause 5.7 shows considerations for further actions.					

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

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

E.3 Partial disassembly with simple tools – Mobile phone type B



IEC 1267/13

Figure E.3 – Partial disassembly of a mobile phone (type B) into its major components

In this example, another mobile phone undergoes partial disassembly using only simple tools such as screwdrivers. Please note that after disassembly the parts of the phone can be easily put back together and the mobile phone will be functional.

The major parts and components shall first be separated as shown in Figure E.3. The parts shown are the TFT display, keypad, bottom housing, main PWB, housing /frame etc. as listed in Table E.2.

Table E.2 – Possible certain substances in major components of the mobile phone

Sample number	Component/ assembly	Materials	Probability of presence[a]	Elements related to certain substances[b]	Options for further 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 the presence indicates the likelihood of finding the listed certain substances (see Table B.1).

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

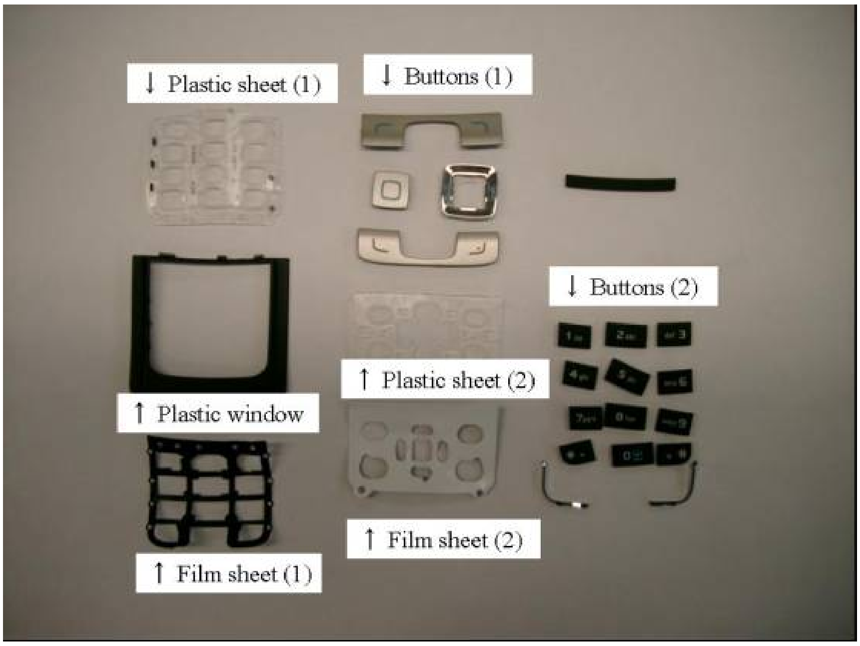
Not all components of the mobile phone separated after this partial disassembly are suitable for direct analysis. The TFT display and PWB are complex structures, containing many different materials. Their construction and size make it difficult to analyse the materials without further disassembly/disjointment steps, regardless of the analytical method used. However, if the first step in the analysis is XRF screening, the elements present relating to certain substances within these complex assemblies can still be detected (see IEC 62321-3-1). A decision then has to be made as to the need for confirmatory testing. Samples 3 and 4 appear to be homogeneous materials for testing directly with XRF in the first instance.

Sample 2, keyboard assembly, shall be screened at this stage to decide whether further disassembly is requred.

E.4 Complete disassembly – Mobile phone type B

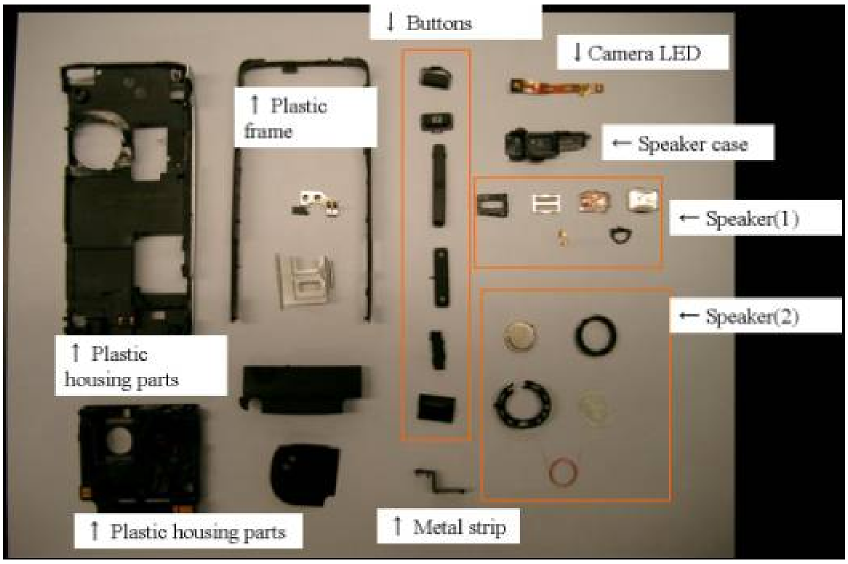
As illustrated in Clauses E.2 and E.3, very few parts of the mobile phone are simple enough in construction and composition for meaningful direct analysis. Almost all parts require further disassembly, or even disjointment which would render them nonfunctional.

Clause E.6 describes the complete disassembly of the mobile phone type B. Figures E.4 to E.6 show the disassembly process of the key pad (Figure E.4), bottom housing (Figure E.5) and the other housing/frame materials (Figure E.6). These parts of the mobile phone shall be disassembled with the commonly used tools (see Annex D) to obtain almost single materials such as plastics or metals. Again, sample selection shall be based on the probability of the presence of certain substances guidance given in Table B.1.



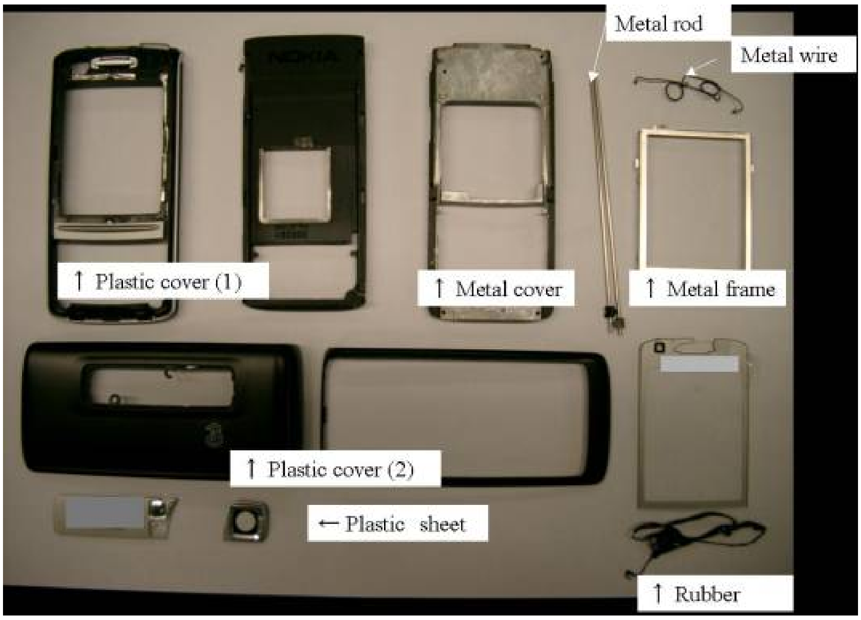
IEC 1268/13

Figure E.4 – Complete disassembly of the key pad



IEC 1269/13

Figure E.5 – Complete disassembly of the bottom housing

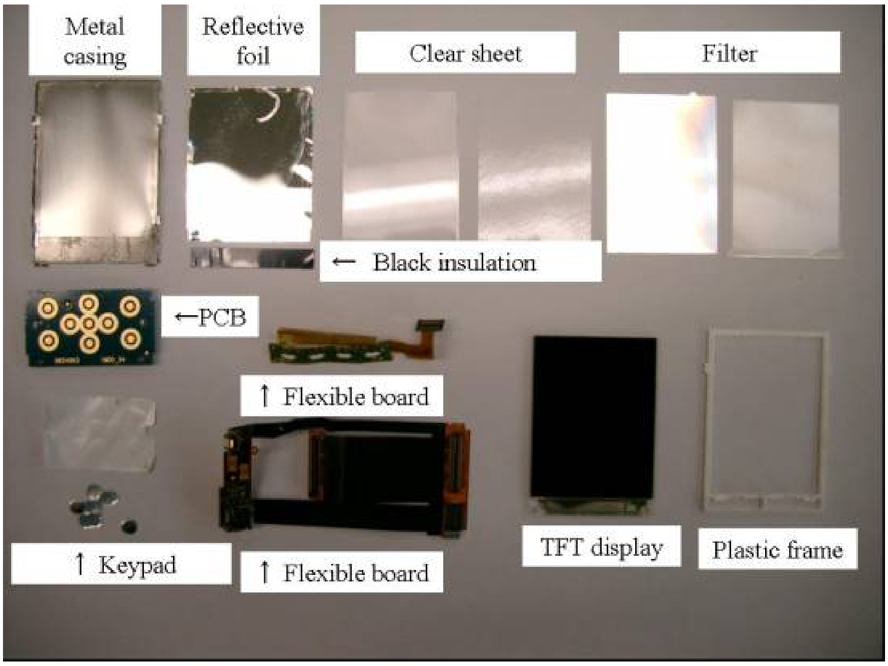


IEC 1270/13

Figure E.6 – Complete disassembly of the other housing/frame

E.5 Partial disjointment – Mobile phone type B

Figure E.7 shows the partial disjointment of the TFT subassembly of the type B mobile phone (previously disassembled in Clauses E.3 and E.4). Separation of certain components of the display such as casing, insulation and filter is only achievable destructively (disjointment). Similarly, partial disjointment of the main PWB of this mobile phone into its components is shown in Figure E.8. The tools required for this disjointment were screwdrivers, a soldering iron and small knife (see Annex D for other tools).



IEC 1271/13

Figure E.7 – Components of the TFT display of the mobile phoneafter partial disjointment

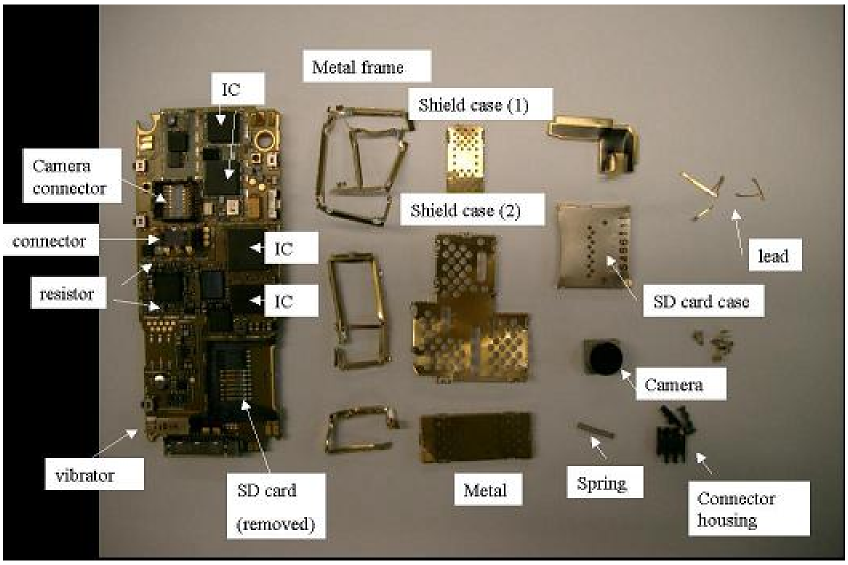


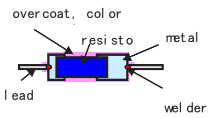
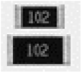
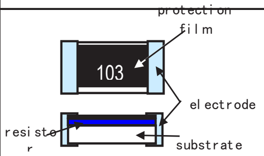
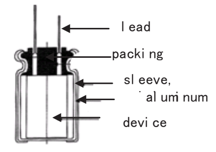
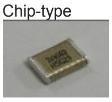
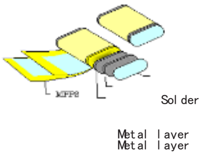
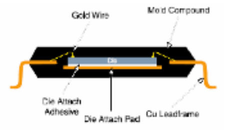
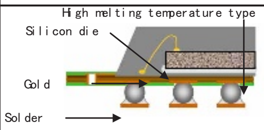
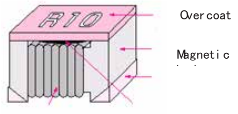
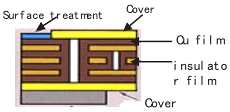
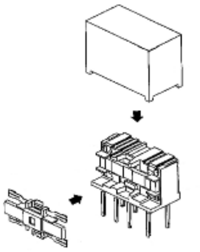
Figure E.8 – Components of the main PWB of the mobile phone after partial disjointment

E.6 Complete disjointment – Examples of disjointment of small electronic components

Table E.3 provides examples of the structure and materials of typical small electronic components used in electrotechnical products. These components shall be obtained from the supplier, after disassembly from the PWB, or other equivalent sources. Materials shall be sampled from the component using appropriate tools (see Annex D) while focusing on those materials with the highest probabilty of of the presence of certain substances (see Table B.1).

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

Table E.3 – Examples of disjointment for typical small electronic components

Assemblies and components		Structure	Sampling point	Possible hazardous materials	Possible 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
			Colour code (ink)	Pb, Cr6+		No practical mechanical	cross-contamination, # of samples to get adequate sample size, sample prep
			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 (glass)	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 (Surface)	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		Cutting or scraping	# samples to get adequate sample mass; sample prep time
			Movable contact	Cd		Cutting or scraping	# samples to get adequate sample mass; sample prep time

E.7 Complete disjointment of integrated circuit lead frame package

In this example, an integrated circuit lead frame package is disjointed, in which lead (Pb) is likely to be present in the tin (Sn) plated termination coating.

Step 1 – Metal leads disjointment from integrated circuit lead frame package

Leads are disjointed from the package (Figure E.9a) by cutting off those metal parts which extend beyond the rest of the package. In this case, the leads removal procedure takes 2 h for 36 individual packages and provides 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 package body, then the lead sample collected will not be contaminated with other materials. Considerable preparation time and number of samples are needed to obtain enough mass for the analysis of the lead frame. Such an analysis is perhaps only practical in some laboratories. Figure E.9b shows the lead sample after disjointment.

Step 2 – Package disjointment

Figure E.9c shows the package after disjointment. This assumes that the disjointment is accomplished in a manner that does not cause cross-contamination with other material layers.

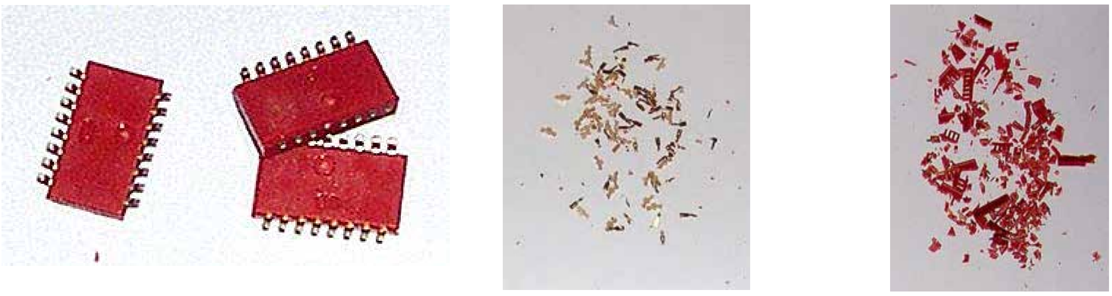


Figure E.9a – Integrated circuit lead frame package

Figure E.9b – Metal leads after disjointment

Figure E.9c – Integrated circuit lead frame package after disjointment

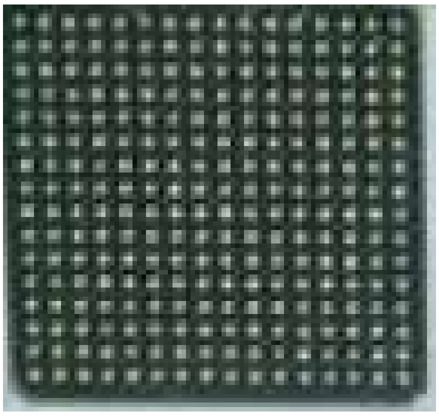
IEC 1273/13

Figure E.9 – Disjointment of lead frame component

E.8 Complete disjointment of ball grid array (BGA) package

E.8.1 General

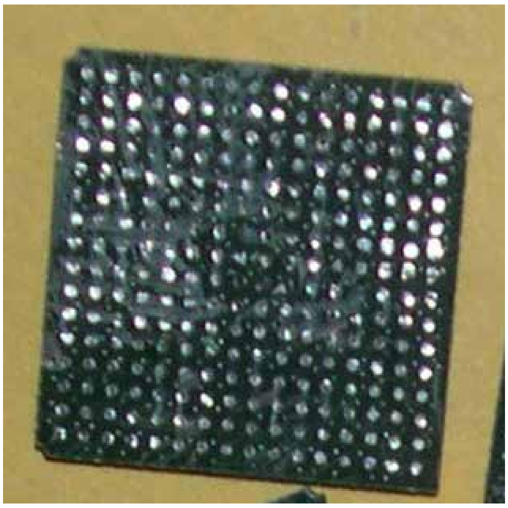
A typical ball grid array (BGA) package is made up of several different material layers: substrate, underfill, silicon die, C4 solder bumps, solder paste and solder balls. Figure E.10 shows an example of BGA package prior to disjointment.



IEC 1274/13

Figure E.10 – BGA package prior to disjointment

E.8.2 Solder ball removal from BGA package – Hand removal procedure



IEC 1275/13

Figure E.11 – BGA package disjointed by the hand removal procedure

The solder balls seen in Figures E.10 and E.11 are disjointed from the BGA package by a hand removal procedure using a sharp utensil, to shave or cut the solder balls away from the substrate. While care is taken not to include any substrate, flux or solder paste with the detached balls of solder, there is inconsistency in the solder ball collection using this method. Some solder balls can only be cut away, while others simply "pop" out of the package instead of being cut or scraped away. As can be seen in Figure E.11 the balls of solder do not disjoint from the package in a manner that guarantees only solder ball material is collected.

IEC 1276/13

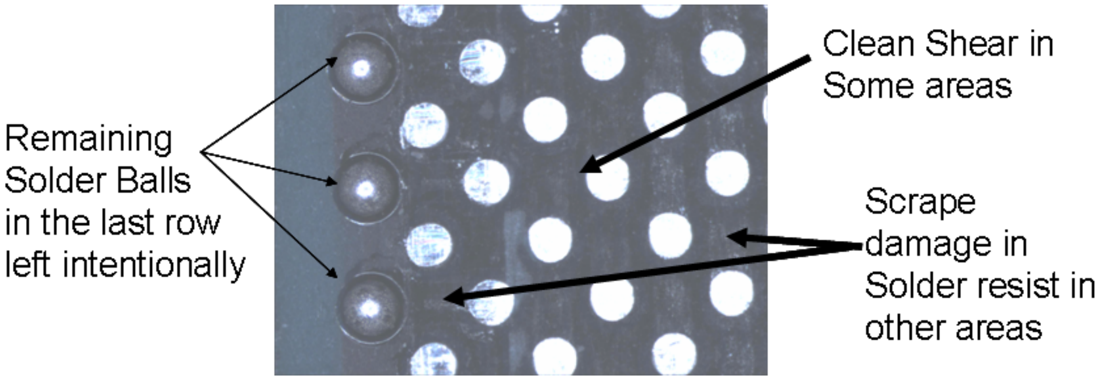
Figure E.12 – Solder ball material collected from BGA using a hand removal procedure

Using the hand removal procedure requires 2 h to remove the solder balls from 15 BGAs. The mass of solder ball material collected is only 1,5 g and contains some flux, solder paste and substrate materials. Thus, 45 BGAs and 6 h work would be required to collect the required minimum sample mass of 4.4 g, per 5.7.1.

Figure E.12 shows the collected solder ball material. However, the sample collected using this hand removal procedure cannot be considered as a "homogeneous" solder ball material as it contains other materials (such as flux, fragments of substrate) collected during the disjointment procedure.

E.8.3 Solder ball removal from BGA package – Solder ball shear procedure

Since the hand removal procedure is not able to provide a homogeneous solder ball sample, another solder ball removal technique is required, namely the shear strength test of IEC 62137-1-2 [8] or the solder ball shear procedure JEDEC JESD22-B117 [9]. 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.



IEC 1277/13

Figure E.13 – BGA solder ball removal using the ball shear procedure

Once again, to obtain enough sample mass for analysis, 45 BGAs are required, which means 6 h for sample preparation, under optimum conditions.

As can be seen in Figure E.13 above, the ball shear method also creates contamination through the scraping of solder resist along with the solder ball material in some areas or lifting of the substrate. Nevertheless, the amount of contamination using the ball shear procedure is significantly less than that from the hand removal procedure. However, contamination remains unavoidable and the time required to yield a significant amount of sample renders this approach impractical.

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[5] IEC 62321-6, *Determination of certain substances in electrotechnical products – Part 6: Determination of polybrominated biphenyls and polybrominated diphenyl ethers in polymers and electronics by GC-MS, IAMS and HPLC-UV* [5]

[6] IEC 62321-7-1, *Determination of certain substances in electrotechnical products – Part 7-1: Determination of the presence of hexavalent chromium (Cr(VI)) in colourless and coloured corrosion-protected coatings on metals by the colorimetric method* [3]

[7] IEC 62321-7-2, *Determination of certain substances in electrotechnical products – Part 7-2: Determination of hexavalent chromium (Cr(VI)) in polymers and electronics by the colorimetric method* [3]

[8] IEC 62137-1-2, *Surface mounting technology – Environmental and endurance test methods for surface mount solder joint – Part 1-2: Shear strength test*

[9] JEDEC JESD22- B117, *Solder ball shear procedure*

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IEC/TS 62239:2008, *Process management for avionics – Preparation of an electronic components management plan*

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[3] Under consideration.

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