

RELIABILITY AND FAILURE ANALYSIS

**Possible Compliance Approaches  
for Directive 2002/95/EC  
(The RoHS Directive)**

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## Executive Summary

Electrical equipment that is placed on the EU market from the 1<sup>st</sup> July 2006 will be presumed to comply with the requirements of the RoHS Directive. Enforcement authorities in Member States will carry out market surveillance. Producers will need to be confident that there are no banned substances within any of their products that are covered by the RoHS directive but the Directive does not specify how this shall be done. Analysis of every component would be unacceptably expensive. Both producers and enforcement authorities need a simple low cost procedure to ensure that no banned substances are present.

ERA was requested to assess four potential approaches:

- Self-Declaration
- Standards for Compliance Testing
- Standards on Reporting Formats
- Information Exchange Networks

Underpinning whatever approach is eventually agreed is the requirement for a clear understanding of how the definition of the maximum concentration values will be interpreted. An early decision is essential to both the electronics industry and the enforcement authorities. At present, several different interpretations of “homogeneous material” are in circulation:

- **“Assemblies” such as modules and printed circuit boards.** If this interpretation is adopted, the use of the six banned substances will not decrease significantly as manufacturers will be able to continue using them in many products. Tin/lead solders could be used on PCBs including heavy PCBs.
- **“Components” such as semiconductor devices, capacitors and resistors.** Adopting this interpretation would significantly reduce the quantities of most of the six substances that can be used but their widespread use could continue legitimately. For example, component manufacturers would be able to continue using tin/lead termination coatings, in some cases by reduction of the lead concentration. Recent research has shown that using tin/lead termination coatings, rather than lead-free coatings, with lead-free solders can lower reliability.
- **“In materials” such as metals, plastic and ceramic.** Adopting this interpretation would permit the use of the least quantities of the six substances and would prevent manufacturers using them as thin coatings on parts and components. For example, tin/lead coatings could not be used as the concentration of lead in the component termination coating metal would be too high.

The correct interpretation must be clearly understood and the best approach for achieving this would be to provide a clear definition accompanied by clarifying examples - an approach adopted by the Packaging Directive.

### Self-declaration

Self-declaration is used for many of the EC New Approach Directives and would be appropriate even though RoHS is not a New Approach Directive. Two main approaches are available to producers:

- **Obtain an assurance from suppliers that no banned substances are present.** These could take many formats but ideally a permanent record will need to be kept. If an enforcement authority finds a banned substance then the producer will need this information to show that he has taken reasonable steps to comply with legislation.
- **Carry out limited analysis to verify declarations.** In a survey carried out by Soldertec, 47% of manufacturers stated that they intend to carry out at least some analysis to check the accuracy of their suppliers' declarations. Analysis will be advisable to audit suppliers, where there are doubts over the reliability of declarations or if none can be obtained. Materials having a greater risk of containing a banned substance, for example PVC and red/orange plastics, could be analysed more frequently, ideally every batch.

The aim of the RoHS Directive is to protect the environment and human health. Efforts from producers must seek to achieve this aim but without excessive cost. In some cases, the erroneous use of a component containing a banned substance will have an insignificant effect on the environment. Analysis of every component by all producers is clearly unreasonable, would be hugely expensive and may even be harmful to the environment and human health. A balance is required between the need to protect the environment and the taking of "reasonable steps" to comply with the Directive. Circumstances when analysis is advisable to support this aim are discussed in this report.

### Standards for Compliance Testing

A small number of Standards exist that are suitable for analysis of parts used for or within electrical equipment to determine the concentrations of the banned substances. There are many more Standards for analysis of other materials such as pollutants in air and water, which could be utilised to develop new standards, but these will not be available by the 1<sup>st</sup> July 2006 deadline. The presence of banned substances can be determined using well established analytical techniques so that producers and enforcement authorities will not need to rely on Standards. However, it is recommended that some new Standards are developed where sampling and analysis are difficult or where a technique has significant limitations that could lead to erroneous results.

### Analysis technology

Several suitable analytical techniques exist that could be used for each of the banned substances and there is no need to specify a particular method for each. Analysis of some components will be technically challenging (but to date no impossible analytical challenges have been identified). In many cases, the choice of procedure will depend on the interpretation of "homogeneous materials".

Some analytical techniques have been proposed which have significant limitations that need to be understood. For example, energy dispersive x-ray fluorescence, available as a handheld instrument, has been proposed for screening with desktop instruments for more accurate analysis. Research has shown, however, that both have significant limitations which, if not fully appreciated, can result in the incorrect assessment of a product.

There will be issues over the analysis of certain components which are discussed in this report and in some cases, clear guidance would be beneficial.

### **Standards on Reporting Formats**

As a first step, equipment producers will need to obtain an assurance from their suppliers that no banned substances are present except where these are exempt from the requirements. This can take many forms and various methods are being used. It would be simpler for both equipment producers and component manufacturers if only a small selection of formats were used.

Several materials declaration formats are being developed but these are not designed for the RoHS Directive (as data on many other substances is included). Nevertheless it may be possible to use some of these for this purpose. A standard format for Supplier Declaration of Conformity, being developed by the IEC provides another option. Several organisations are working on markings on components, assemblies and products which would show their RoHS status. Once agreed these could become internationally accepted standards that could be used as “materials declarations”.

### **Information Exchange Networks**

Member State enforcement authorities rarely co-operate to enforce EU Directives. Information is passed between authorities via the Commission only when a safety risk is identified. The new General Product Safety Directive, however, encourages collaboration and an exchange network has been set up to ensure that Member States do co-operate.

This type of approach would be beneficial for those responsible for policing RoHS legislation in order to survey the huge variety of equipment in a systematic way and to avoid duplication of effort.

### **Guidance on Compliance**

Producers require guidance on how to comply with the RoHS directive. Information in this report will enable producers to decide on which approach to use; there is no universal procedure for all producers. Procedures could be further clarified by the use of a checklist or a decision tree approach and illustrative examples of these are included. The Commission could consider sponsoring a simple guide such as was produced for the Packaging Directive. Input should be provided by all parts of the electronics industry and experts on analysis technology.

## Executive Summary of Recommendations

### Definition of Maximum Concentration Values

1. It is essential that the correct interpretation of the “Maximum Concentration Values” definition is clearly understood by all producers and the enforcement authorities in all Member States. The TAC should decide whether the correct interpretation for the concentration limits is in “materials”, “components” or “assemblies” as illustrated by the indicative examples given in this report. The wording of the Directive implies that the interpretation should be “materials”. In order to avoid misunderstandings, indicative examples should be used as part of the definition.

### Self-Declaration

2. Electrical and electronic equipment placed on the market will be presumed to comply with the RoHS Directive and enforcement authorities will carry out market surveillance. Self-declaration is used for New Approach Directives and is suitable for the RoHS Directive even though RoHS is not a New Approach Directive.
3. The main approach used by producers should be to obtain assurances from all suppliers that materials, components and equipment do not contain the banned substances. Producers will need to maintain records to show enforcement authorities in the event that a banned substance is found in the course of market surveillance.
4. It would be unreasonable to expect producers to analyse every batch of incoming materials and components, as this would be prohibitively expensive. It is also unnecessary to specify what should be analysed, but producers will be expected to take “reasonable steps” to ensure that banned substances are not used. The extent to which analysis is used will depend on many factors including:
  - the relationship with suppliers,
  - the risk of a banned substance being present, and
  - the quantity of product put onto the market.
5. Enforcement authorities should take into account what is required to protect the environment and human health. Where a banned substance is identified but the effect on the environment and human health is negligible, then as long as the producer can show that reasonable steps were taken to comply, the part containing the banned substance could be modified for future production, but it may not be necessary to remove the product from the market.
6. It is unlikely that Standards will be available by the 1<sup>st</sup> July 2006 deadline. Producers require a simple guide showing how they can comply with the RoHS Directive. A checklist or decision tree approach could be used. The guide could be produced in collaboration by the TAC, representatives of the electronics industry and experts in analysis technology.
7. The approach used must be harmonised in all Member States.

**Standards for compliance testing**

8. Techniques for analysis of all six banned substances are available. As with the Packaging Directive, there is no need to specify any particular procedure or instrument as, in most cases, there are several sufficiently accurate analysis methods available. Producers would benefit, however, from a Standard defining what steps they should take to comply with the Directive. This could provide guidance on when analysis is advisable, appropriate sampling methods and records that should be maintained.
9. New Standards would be useful for certain analytical techniques, which could give inaccurate analysis of electrical products if their limitations are not understood. In particular these include ED-XRFA and SEM-EDX.
10. Analysts should be aware of the limitations of ED-XRFA. This is a very useful technique widely promoted for checking compliance, but research has shown that incorrect assessments can be obtained.
11. Consideration should also be given to modifying certain existing Standards, for example those that specify tin/lead solders.

**Standards for reporting formats**

12. Several approaches are being developed within the electronics industry. These are intended to provide equipment producers with an assurance that the material, component, etc. does not contain a banned substance. This is the minimum information that a producer needs.
13. Materials declarations that include all six banned substances, ideally explicitly stating that they are absent rather than simply not mentioning them, would be suitable. The standard "Substance Declaration of Conformity" (SDoC) being developed by IEC would also be suitable. (This requires records of analysis to be maintained which can be audited).
14. Markings on components providing a readily visible indication that the banned substances are absent would also be suitable. This approach would minimise the need to maintain paper or electronic compliance records. Any Marks used should be standardised.

### Information exchange networks

15. A RoHS Network to co-ordinate market surveillance should be established. Enforcement authorities in all Member States would benefit from close collaboration. Benefits would include

- pooling of limited resources,
- sharing of market intelligence and
- transfer of expertise in analysis technology.

A formal Network similar to that set up by the European Commission for the New General Product Safety Directive appears a sensible approach. This would ensure that surveillance included all categories of equipment and all Member States, and that higher risk products could be targeted.

16. Clear guidelines for product assessment procedures are required. Producers could use these also.

17. Analysis should be sub-contracted to laboratories with the required skills and expertise.

18. A standard reporting format should be used. All results should be circulated to all Member States including data on products found to comply with RoHS legislation (i.e. not just those that fail).

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## Abbreviations List

|         |  |
|---------|--|
| AAS     | Atomic Absorption Spectroscopy   |
| ATR     | Attenuated Total Reflection  |
| CoC     | Certificate of Conformity  |
| CrVI    | Hexavalent chromium  |
| DoC     | Declaration of Conformity  |
| ED-XRFA | Energy Dispersive X-Ray Fluorescence Analysis  |
| ED-XRFA | Energy dispersive X-ray fluorescence analysis  |
| EEE     | Electrical and Electronic Equipment  |
| IECEE   | Worldwide System for Conformity Testing and Certification of Electrical Equipment<br>Worldwide System for Conformity Testing and Certification of Electrical Equipment |
| EICTA   | European Information & Communications Technology Industry Association  |
| EMC     | Electromagnetic Compatibility  |
| FTIR    | Fourier transform infra-red  |
| GCMS    | Gas chromatography mass spectroscopy   |
| HASL    | Hot Air Solder Levelling   |
| HPLC    | High Performance Liquid Chromatography   |
| I/O     | Input / Output   |
| IC      | Integrated Circuit   |
| IC      | Ion Chromatography   |
| ICP     | Inductively Coupled Plasma spectroscopy  |
| JBCE    | Japan Business Council in Europe   |
| MLCC    | Multi Layer Ceramic Capacitor  |
| MRP     | Manufacturing Requirements Planning  |
| MSDS    | Materials Safety Data Sheet  |
| PBB     | Polybrominated biphenyl(s)   |
| PBDE    | Polybrominated diphenyl Ether(s)   |
| QFP     | Quad Flat Pack (a type of high density IC package)   |
| RoHS    | Restriction of Hazardous Substances  |
| SEM/EDX | Energy Dispersive X-ray Analysis with Scanning Electron Microscopy   |
| TAC     | Technical Adaptation Committee   |
| WD-XRFA | Wavelength Dispersive X-ray fluorescence analysis  |
| WEEE    | Waste Electrical and Electronic Equipment  |

## 1. Context to this report

Individual Member States of the EU have adopted various measures to restrict the use of certain hazardous substances. The aim of the RoHS Directive (2002/95/EC) <sup>1</sup> is to harmonise legislation within the EU and to restrict the use of six hazardous materials (lead, mercury, hexavalent chromium, cadmium and two flame-retardants) in certain specified types of electrical and electronic equipment.

The Directive was published in the Official Journal on 13<sup>th</sup> February 2003 and Member States have until 13<sup>th</sup> August 2004 to transpose this into national legislation. The RoHS Directive directs Member States to enforce their national RoHS legislation. Article 8 of the Directive states that *Member States shall determine penalties applicable to breaches of the national provisions adopted pursuant to this Directive*. It does not, however, state how the authorities are to determine whether a producer has complied with legislation or how producers can demonstrate that their products comply.

Electrical equipment manufacturers at present do not know how to demonstrate that their products are compliant. One approach is to rely on declarations provided by their suppliers. At the other extreme, a full analysis of every homogeneous material would be possible but prohibitively expensive. Clearly an approach is required that protects the environment but can be achieved at a reasonable cost, is reliable and has the buy in of all parties.

The Directive does not impose requirements on producers to prove that their products comply with national RoHS legislation. Although RoHS is not a New Approach Directive, producers will be expected to comply with legislation, and national enforcement authorities to carry out market surveillance.

Banning the use of these substances is intended to give health and environmental benefits mainly during the production and end of life stages of the life cycle of electrical products.

- Workers involved with the production of the six hazardous materials, components that contain these materials and equipment that contain these components may be exposed to these six substances. This will involve workers within and outside of the European Community.
- The main environmental concern at the end of life is from the hazardous materials entering the environment via landfill or during recycling processes. The WEEE Directive (2002/96/EC) <sup>2</sup>, which was adopted on the same day as the RoHS Directive aims to minimise the disposal of WEEE into landfill, however the EU believe that it is likely that significant quantities will continue to be disposed of by this route. As the quantity of WEEE disposed of to landfill decreases there is a concern that the six banned substances may be emitted into the environment from recycling processes.

## 2. Objective and scope of this report

The purpose of this study is to investigate ways in which the producers of electrical and electronic equipment can ensure that their products meet the requirements of the RoHS Directive. Producers are not required by the RoHS Directive to prove that their products comply but, if challenged by the enforcement authority, they will need to show that they have procedures in place and supporting documentation that shows that they have taken reasonable steps to comply.

ERA was requested to assess four potential approaches:

- Self-Declaration
- Standards for Compliance Testing
- Standards on Reporting Formats
- Information Exchange Networks.

## 3. Report Structure and Project Methodology

Producers and enforcement authorities require a clear understanding of the definition of the maximum concentration values and how “homogeneous materials” is to be interpreted. This is discussed in *Section 4.2* of this report. All that follows relies on this being clearly understood.

*Sections 5 to 8* discuss the four ways forward that have been proposed by the WEEE and RoHS TAC.

Many analytical procedures are available and can be used for the analysis of electrical equipment. Methods that are suitable for the six banned substances, their limitations and how they can be used for electrical products are discussed in *Section 6* and the techniques described in more detail in *Annex A*.

It became clear during this study that producers need clear guidance regarding how they can determine whether their products comply with the requirements of the RoHS directive. One suggestion is a guide produced in collaboration with the electronics industry. Check lists or decision trees are used in guides for other EU directives and some preliminary ideas are given in *Section 9*.

Each main section includes a short summary of the main conclusions. More comprehensive discussion of the conclusions is provided in the Executive Summary at the beginning of the report.

The approaches used during this study included:

- Obtaining views from all sectors of the electronics industry. Commercial analytical organisations and manufacturers of analytical equipment were also consulted.
- Literature surveys to obtain information on standards, analysis technology and materials declarations.

- Investigation of existing EU Directives, especially the New Approach Directives, to obtain ideas on how self-declaration could be used, setting of maximum concentration limits and information exchange networks.

#### 4. Discussion of the RoHS Directive

The WEEE and RoHS Technical Adaptation Committee (TAC) has identified four potential ways forward and these are investigated in turn in this report. In practice, it is likely that a combination of all four approaches will be used. Any procedure that is developed must be easy for all manufacturers to carry out at reasonable cost. It must be acceptable to the electronics industry including small and medium size manufacturers (SMEs) but also must also protect the environment. Procedures will also be required by enforcement authorities for market surveillance.

To guarantee that no banned substances are present in a piece of equipment it would be necessary to destructively analyse every individual homogeneous material that is present. It has been estimated that to do this for a typical piece of IT equipment could cost £80,000. This is clearly excessive and would be unacceptable to producers; a simpler procedure is required. Equipment producers can obtain materials declarations from their suppliers to ensure that the components and materials they use comply with RoHS. Analysis could be carried out as spot checks or where there is any doubt. As the aim of this Directive is to protect the environment and human health, the amount of effort should be sufficient to achieve this aim. The effort required to avoid inadvertently including a banned substance should be proportional to its likely impact on the environment and human health.

Note: In this report the term “RoHS compliant” has been used for “equipment that meets the requirements of the RoHS Directive”. While the RoHS Directive does not apply to materials, components and assemblies, the term “RoHS compliant” is sometimes used to indicate that the six banned substances are absent.

##### 4.1 The banned substances

The six substances that are subject to the materials bans in the RoHS Directive are:

- Lead (Pb) - Some exemptions are permitted.
- Mercury (Hg) - Permitted in certain types of lamps.
- Cadmium (Cd) - Exemption for electroplated coatings.
- Hexavalent chromium (CrVI) - Chromium is banned only in the hexavalent form and chromium metal and trivalent chromium are not restricted. The Directive allows one specific exemption for CrVI.
- Polybrominated biphenyls (PBB) - PBB was used as a flame retardant in plastics but PBB is no longer produced but some may possibly be present in recycled plastics.

- Polybrominated diphenyl ethers (PBDE) - PBDE comprises a range of flame retardants including:
  - Tetrabromodiphenyl ether (Tetra-BDE). This is no longer in production but may occur in recycled materials.
  - Pentabromodiphenyl ether (Penta-BDE). Not normally used in electrical equipment, main use is in furniture foam.
  - Octabromodiphenyl ether (Octa-BDE). Used in relatively small quantities in electrical equipment.
  - Decabromodiphenyl ether (Deca-BDE). Widely used in electrical equipment but less than previously. The European Commission has stated that the status of this substance will be reviewed on the completion of a comprehensive risk assessment. A final decision has not been made but preliminary findings are that this substance poses no threat to human health or the environment<sup>3</sup>.

The material bans include lead, cadmium and mercury as metals and as their compounds but some exemptions are permitted.

## 4.2 Concentration limits and definitions

Although a final decision had not been made at the time of writing, the maximum concentration values for the banned substances are likely to be:

- 0.1 weight % of lead, mercury, hexavalent chromium, PBB and PBDE “in homogeneous materials” and
- 0.01 weight % cadmium “in homogeneous materials”.

The EC published a consultation document<sup>4</sup> which gives these limits for the RoHS Directive as well as a definition of “*homogeneous material*” as, “*a unit that cannot be mechanically disjointed into single materials*”. This definition has been interpreted in various ways within the electronics industry and the exact interpretation needs to be clarified.

These limits are based on the concentration limits used for the End of Life Vehicle (ELV) Directive (2000/53/EC). The latter bans the four metals in vehicles with a list of exemptions. Since its adoption, however, this has not been enforced because there is no clear procedure for either demonstrating compliance or for market surveillance. The ELV Directive includes the phrase “*not intentionally introduced*” as a way of preventing deliberate use of these substances however it would be very difficult for enforcement authorities to prove this. This phrase has therefore not been included in the RoHS Directive definition.

The Packaging Directive (94/62/EC) also has substance restrictions but uses a different approach. The concentration limit is currently a 100 ppm (0.01%) total of the four metals in the “*component*”.

The “*component*” is an assembly of materials such as a steel drum, which has a paint coating and a soldered seam. Owing to this definition, it is possible to use lead driers in the paint or have a hexavalent chromium coating as the concentrations of these restricted metals totals in components are less than 100 ppm. As a result some lead and hexavalent chromium can be used to produce packaging.

|                      | ELV   | Packaging                             | Proposed RoHS                        |
|----------------------|---|---------------------------------------|--------------------------------------|
| Concentration limits | 0.1 % for each material (Cd = 0.01%) not intentionally introduced | Pb + Cd + Hg + CrVI = 100 ppm (0.01%) | 0.1 % for each material (Cd = 0.01%) |
| Limits as:           | In materials and components                                       | In components                         | In homogeneous materials             |

The definition of the concentration limits is important as this affects how analysis can be carried out and whether producers are able to deliberately use any of the banned substances. The definition of homogeneous materials is being interpreted in various ways within the electronics industry; some interpret this to mean single substances, others interpret this to mean discrete devices (what has traditionally been called a component by many in industry), and a few as meaning assemblies (e.g. PCBs).

What producers are permitted to use in their products depends on whether the substance ban applies to a material, a discrete device or an assembly. The potential impact of using each of these interpretations is illustrated in the following examples.

- **Materials:-** metals (e.g. electroplated coatings, solder alloy), plastics (e.g. ABS, nylon), ceramics (e.g. dielectrics). This definition would permit the least amount of banned substance in electrical equipment. Tin/lead solders and terminations could not be used. It could be argued that the RoHS directive implies that this interpretation is the correct one because the exemptions all apply to materials, e.g. high melting point solders (not components that contain these), mercury in certain lamps (the weight percent of mercury in whole exempt lamps would be much less than 0.1%) and lead in solders for servers. The only apparent exception is lead in ceramic parts, which implies the complete device.
- **Components:-** e.g. semiconductor packages, resistors, capacitors, etc. >0.1% lead could be used in termination coatings as long as this lead accounts for <0.1% of the component mass. Some component manufacturers have openly stated that they would produce tin/lead terminated components if this was permitted. These manufacturers include some who are already changing over to tin coatings. Tin/lead solders (a material) would not be permitted however. Many examples of cracking and early failure caused by contamination from low levels of lead have been observed and reported in recent published literature<sup>5</sup>. The use of tin/lead terminations with lead-free solders could result in less reliable solder bonds resulting in earlier equipment failure. Another example is high current relays that use silver/cadmium oxide contacts. This application would not be permitted if the ban applied to materials but, because of the weight ratio of contact material to relay mass, this would be permitted if the ban applied to components. Hexavalent chromium is usually used as a thin coating which accounts

for only a small percentage of components, nearly always less than 0.1%, and so there would be very little reduction in its use if this interpretation is used.

- **Assemblies:-** e.g. PCBs, power supply modules. This allows the largest amount of banned substance to be used. It could be interpreted to mean that PCBs can be constructed using tin/lead solders as long as the board contains some heavy components that result in the total lead content being less than 0.1%.

The following example for an electronic component shows how the definition of “concentration limits” affects what component producers would be allowed to use in their products;

**Example 1:** Many “components” have tin/lead coatings as solderable termination coatings. Coatings contain from 5% to 40% lead so, if the maximum concentration limit applies to homogeneous materials, these coatings clearly exceed the 0.1% limit and the electronic component should not be used in “RoHS compliant” equipment. If the maximum concentration limit applies to whole “components” then tin/lead termination coatings could be used as long as the lead content of the “component” is less than 0.1% lead.

Philips Semiconductors and some other manufacturers publish the material composition of their components on their websites. Calculation of the lead content of a random selection of these semiconductor packages (assuming lead is present only in termination coatings) gave concentrations of 0.09% to 0.5%. Those with the lowest lead content would be used in RoHS compliant products if the concentration limit applies to “components” instead of materials. The following table illustrates the dramatic difference that the definition of has on the lead concentration for one device.

| Definition terminology   | Lead concentration in Philips QFP64 |
|--------------------------|-------------------------------------|
| In homogeneous materials | 15%                                 |
| In components            | 0.09%                               |

It would not be difficult for component manufacturers to modify their products to continue using smaller quantities of the restricted substances if the limits are 0.1% in whole “components”. Reduction of the limit to 0.01% would prevent this but would create other commercial problems. Standard “lead-free” solder always contains a small lead content even if virgin tin is used. This is typically about 0.03% in commercial purity tin. The world’s production capacity for tin containing less than 0.01% lead is sufficient to supply all the electronic solder needs but the cost of this high purity solder is higher than for standard electronic tin.

If the additional wording used in the ELV Directive “not intentionally introduced” is included, then clearly, in a termination coating with 15% lead, this lead is deliberately used and so the component could not be used. This does not account for case of unintentional uses of lead (i.e. in error). There would be instances where proving that a material was deliberately introduced will be difficult or impossible.

ERA has received many comments that the proposed definition in the consultation document published by the Commission gives a definition of “homogeneous materials” that is unclear and further clarification is required. Above all, most manufacturers and distributors are concerned that

- there is clear understanding of this definition,
- that this understanding is the same in all EU Member States and
- that it is interpreted in the same way by both manufacturers and enforcement authorities.

### 4.3 Existing self-declaration schemes - Packaging Directive

#### 4.3.1 Definitions

There are parallels between the Packaging Directive and the RoHS Directives in that both are non-CE mark Directives, and both restrict lead, cadmium, mercury and hexavalent chromium. RoHS is not a New Approach Directive unlike the Packaging Directive.

The Packaging Directive sets a total concentration limit for lead, cadmium, mercury and hexavalent chromium. Other substances are also restricted and subject to limits.

The limit is <100ppm (0.01%) for the total of the four substances in the packaging “component”. A packaging component could be a steel drum with a paint coating, cardboard boxes or glass bottles with printed labels and the concentration limit refers to the whole “component”, not to its “constituents” or the “homogeneous materials” that comprise the component. Consequently, this definition enables some of these metals to be used in limited quantities. For example, lead driers in paints used on steel drums are permitted as the total concentration of the four substances in painted drum is less than 100 ppm although the lead concentration in the paint is much higher.

In practice, where an operation is carried out which might add any of these metals (which could be converting plastic chip into bottles, printing onto labels or attaching labels), an assessment procedure needs to be carried out. In the case of packaging, it is usually the “converter” (for example the company converting plastic chip into bottles), which does this. The converter usually sub-contracts chemical analysis of their product only once and a certificate is issued by the analytical laboratory to state that this material complies with this part of the Packaging Directive. The reason analysis is not carried on a regular basis is because heavy metals are almost never detected in most types of packaging materials.

The Packaging Directive was adopted by the EU in 1994. A series of Standards has since been published, the earliest was approved in 1999 and published in 2000, but the new Standards still do not provide harmonised methods for the testing of heavy metal concentrations. In the interim, users of packaging have had to rely on national legislation and the Directive itself to determine how to implement its requirements.

One CEN report <sup>6</sup> (CR 13695-1:2000) provides guidance on the requirements for measuring and verifying the four heavy metals present in packaging. This does not specify analysis procedures and any suitable method may be used. Unlike in the RoHS Directive, the maximum total concentrations of the four banned metals correspond to “packaging components” such as printed labels, printed

multi-layer films and glass bottles. This CEN report defines packaging components and packaging constituents:

- Packaging component – any part of packaging that can be separated by hand or by simple physical means.
- Packaging constituent – the smallest part from which packaging or its components are made and which cannot be separated by hand or by using simple physical means.

The term “constituent” corresponds closely to the term “homogeneous material” that is likely to be used in the RoHS Directive (although this has not been agreed yet by the TAC).

The approach used for the material restrictions in the Packaging Directive is to include examples to clarify the definitions and these are included in this CEN report <sup>6</sup>. A similar approach comprising a clear definition with examples could usefully be applied with the RoHS Directive. It should be clear from this which parts of components are classed as “homogeneous materials”.

For the RoHS Directive, clarifying examples include:

- Components:
  - printed circuit boards
  - integrated circuits
  - capacitors
  - transformers
  - lamps, connectors
  - galvanised steel screws
- Homogeneous materials:
  - Metals
    - Solder alloys in the form of solder that, for example, bonds components to PCBs.
    - Single layer termination coatings, for example electroplated tin on a copper lead-frame. Tin plated over electroplated nickel on a copper substrate is three homogeneous materials.
    - Lead-frame metal (not including any coatings).
    - Electrodes within MLCCs.
    - Bonding wires within integrated circuit devices.
    - Stainless steel screws, electroplated or hot dipped termination coatings, etc.
  - Plastics (e.g. used for housings)
    - Plastic encapsulation coatings.

- Plastics contain a wide variety of additives (pigments, stabilisers, fillers, flame retardants, etc.). It is assumed that a plastic including additives is a homogeneous material as these cannot be easily removed by physical means. PCB laminates are usually glass-fibre reinforced materials with copper conductors. These cannot be separated by physical means and would be difficult to analyse separately.
- Paint coatings which are a mixture of resin, pigment and other additives.
- o Ceramics
  - Dielectric material within MLCCs.
  - Passivation coating on aluminium casting. (These are complex mixtures of compounds which can not be separated by physical means).

Some homogeneous materials will contain several substances but separation by hand or simple physical methods is not possible. An example would be plastic containing fillers, pigments, flame retardants, stabilisers and other additives.

There are some “grey areas” which need to be clarified and some of these are discussed in Section 6.5.

### **4.3.2 Analysis and enforcement**

Analysis of packaging is more straightforward than that of electrical equipment partly because these limits apply to components and not constituents but also because packaging is usually of a much larger size and less complex than most electronic components.

The Packaging Directive itself is more complex than the RoHS Directive as it has many different requirements. EUROPEAN <sup>7</sup> has produced a guide to self-assessment for the Packaging Directive and this includes all aspects of the Directive that producers need to consider. Compliance is demonstrated by completion of a summary assessment form, which is included in the guide.

Enforcement authorities expect manufacturers to take reasonable steps to comply with the requirements of this Directive. However, “reasonable steps” are not defined and so any disputes have to be argued in court. A “reasonable steps” defence is applicable to other Directives as well as national legislation. Producers of electrical and electronic equipment would prefer that the reasonable steps that are expected for the RoHS Directive should be defined. This is a difficult point as it would not be acceptable for the EU to issue a procedure that permits producers to take actions that do not prevent the use of non-compliant components.

Producers will do whatever they think necessary to avoid the use of a banned substance in their products because if one were to be found by an enforcement authority, the penalty may include a requirement to remove these products from the market. This would be a severe penalty in most cases. “Reasonable steps” are not defined in legislation; if they were, producers could do this and no more.

## 4.4 Uses of the banned substances in electrical equipment

The main uses of the six substances in electrical equipment are listed in Table 1.

**Table 1. Main uses of the six banned substances in electrical equipment**

| Material            | Used in  | Comments   |
|---------------------|--|--|
| Lead                | Solders  | One of the main uses of lead in electrical equipment. Solders containing >85% lead are exempt                                  |
|                     | Glass such as cathode ray tubes, glass to metal seal, lamps, etc.                            | Lead in glass of cathode ray tubes, electronic components and fluorescent tubes is exempt                                      |
|                     | Component of ceramics such as in piezoelectric devices, some types of dielectric materials   | Components may contain lead in the form of a ceramic but shall not be used in solderable termination coatings of these devices |
|                     | Free machining steel, aluminium and copper   | RoHS gives specific concentration limits for lead in these alloys  |
|                     | Batteries  | Lead/acid batteries. These are permitted by the Battery Directive and in vehicles.   |
|                     | Plastics as stabiliser or pigment  |  |
| Cadmium             | Paints   | Driers, pigments   |
|                     | Plastics as pigment or stabiliser  | Already banned by the Cadmium Directive (91/338/EEC)   |
|                     | Solders  | Uncommon   |
|                     | Thick film circuits and in ceramic materials   | Largely already phased out as a component of this technology   |
|                     | Electric contacts  | Widely used in relays and switches   |
|                     | Batteries  | Nickel cadmium batteries. These are permitted by the Battery Directive   |
| Mercury             | Semiconductors, for example cadmium sulphide is used in light sensors and in new solar cells | Some solar powered equipment would be banned by RoHS if it contains cadmium based solar cells                                  |
|                     | Batteries  | Restricted by the Battery Directive (91/338/EEC)   |
|                     | Electrical contacts  | Relays, microswitches, tilt sensors, etc.  |
| Hexavalent chromium | Fluorescent lamps  | Limited quantities in fluorescent lamps exempt from RoHS Directive   |
|                     | Passivation coatings   | Thin surface coatings on various metals. Used on exposed metal and to improve adhesion of paint and other coatings             |
|                     | Corrosion resistant paints and coatings  | Component of paint or coating materials  |
|                     | Hard chrome plating  | Used to produce coating but chrome plate is chromium metal, does not contain CrVI  |
| PBB and PBDE        | Plastics etchant for metallised plastics   | Used to etch plastic before coating but not present in final product   |
|                     | Flame retardants used in plastics  | There are many other types of brominated flame retardants  |

Some of the materials in Table 1 are present on exposed surfaces, whereas others are used within the equipment and often inside components and are not accessible without dismantling, cross-sectioning or grinding.

The choice of an appropriate analytical method depends critically on whether the maximum concentrations apply to whole components or to single materials. It is important, therefore, to define precisely to what the maximum concentration limits apply.

#### **4.5 Conclusions and recommendations concerning concentration limits and definitions**

1. A clear interpretation of the definition of the maximum concentration values is essential. It is important that the same interpretation is understood in all Member States and by both producers and enforcement authorities. It is also important for deciding how equipment is analysed.
2. Three interpretations have been made within the electronics industry, the differences lie in the definition of “homogeneous materials” which has been interpreted as:
  - Materials (metals, plastics, ceramics).
  - Components (semiconductor devices, resistors, capacitors, etc.).
  - Assemblies (PCBs, etc.).

## 5. Self-Declaration

### 5.1 Overview

Self-declaration is used by producers of electrical equipment to show that their products comply with EU New Approach Directives. These include the CE mark schemes and a small number of non-CE mark Directives such as the Packaging Directive. Most manufacturers are conscientious and endeavour to meet the requirements of these Directives. The RoHS Directive is not a CE or a New Approach Directive but it will be presumed that products that are placed on the market comply with the Directive's requirements.

The Directive requires that Member States provide "effective, proportionate and dissuasive" penalties in the event that its provisions are breached. In most cases the most severe penalty for a producer, in the event that one of its products were to be found to be non-compliant by enforcement authorities, would be not being able to sell their product. This would constitute a severe financial penalty for many and would be a highly dissuasive threat.

Under circumstances where the accidental inclusion of a non-compliant component does not pose a threat to the environment or human health or if taking the product off the market poses a greater threat than allowing the producer to sell the products, then alternative, more proportionate action may be considered by the enforcement authority. Preventing the producer from selling these products may not be the most suitable penalty.

Component manufacturers are in the best position to control the substances used in the manufacture of their products. Although the RoHS Directive places the responsibility on equipment producers, component manufacturers, distributors and others in the supply chain are already looking at ways of ensuring that they are able to provide suitable materials and components to their customers. The RoHS Directive applies to equipment producers only and there is no requirement for component suppliers and distributors to produce RoHS compliant products and no legal action, under RoHS legislation, could not be taken against material and component manufacturers that make false claims.

Component producers and distributors, however, are taking very seriously their obligations to provide materials and components that are suitable for use in RoHS compliant products. Selling items purporting not to contain the banned substances but which inadvertently do might be very damaging commercially.

### 5.2 Issues

Under the New Approach Directives, self-declaration is the main means used by manufacturers to demonstrate claims that products are compliant with the requirements of Directives. By comparison, the act of "putting onto the market" will indicate that the producer is declaring that it is RoHS compliant.

For enforcement authorities to be able to monitor products that are put onto the market, they will need to know who the producers are and which products they produce. It is becoming clear that in most EU Member States “producers”, as defined by the WEEE Directive, will be required to register and provide certain information. This is also a possible option for the RoHS Directive which would enable enforcement authorities to identify producers of any products that they find are not RoHS compliant. EICTA, however, does not favour registration because this would create a great deal of work for honest producers and for enforcement authorities with little benefit as dishonest producers could fail to register. The identity of a producer is often obvious from other marks on electrical equipment.

When a non-compliant product is found by an enforcement authority, they would expect to see documentary evidence from the producer to show that reasonable steps to comply have been taken.

The major issue for control of RoHS-restricted materials in equipment, materials and components is the identification and tracing of these materials on an on-going basis, especially since none of them will be “visually obvious” or easily detected by testing. Unlike with most other product Directives, it is not possible to carry out one or two standard tests on a product to determine RoHS compliance.

This poses considerable problems for manufacturers, especially small to medium enterprises (SMEs), as the resources needed to trace, control and document these materials in multitudinous components would be substantial; especially where all such components must be traced through the “supply chain” from base material formulation to end-use equipment.

Various different strategies could be used to address the particular issues that arise, as outlined below, and most of these must, by default, begin with the formulators of the base materials used in the components which are then used in various levels of sub-assemblies and thence the final equipment.

### **5.2.1 Issues For Materials & Component Suppliers**

A substantial problem for component manufacturers and fabricators is to determine the RoHS-restricted chemical content of raw materials & components that they use, and then to ensure that the reported content is consistent from batch to batch, and over time.

This problem – whilst probably never totally soluble – can be eased by the choice of appropriate measures. The suggestions below outline the advantages and disadvantages of various measures identified to date:

#### **Standards for Materials**

Standards are discussed in Section 6. Electrical equipment is made up of three types of material or component as far as Standards are concerned:

- Materials - “standardised” (solders, metals, standard liquids & solvents)
- Materials – “non-standardised” (plastics, printed circuit boards, ceramics)

- Discrete components (as opposed to materials) used as “n” off per sub-assembly.

At present Standards do not exist for most of these and will take several years to be agreed. In the interim, compliance must be achieved without Standards. Recommendations for new Standards are listed in Section 6.6.

Some additional points to consider include:

- Specific problems could exist with some common “standardised” materials such as “commercial grade mild steel” and aluminium where the specifications are loose and there is large-scale use of material reclaimed from unknown sources.
- Specific problems could exist in some industries with “non-standardised” materials – notably low cost plastics - where there may be large scale use of use of material reclaimed from unknown scrapped equipment and consumer items, as well as of material from “floor sweepings” (as they are colloquially known).
- Use of industry databases (see Section 7.2.3) to allow remote extraction of RoHS data on each material to the manufacturer’s local “Bill of Materials” (BoM) database used for each sub-assembly.
- 3<sup>rd</sup> party Standards (e.g. USA Underwriters Laboratories [UL] Standards) may exist in some cases and it may be possible to adapt these to include RoHS materials controls – providing the issuing organisations are both in agreement and cooperative.

### **Certification Aspects**

The certification *of* – as opposed to the Standards *for* – the materials and components discussed above is a large issue, for which no single solution exists. With RoHS, certificates would need to be issued to declare that a component, assembly or material complies with the requirements of 2002/95/EC, the RoHS Directive. This approach is already used for the heavy metals restrictions in the Packaging Directive.

Some obvious possibilities include:

- a) Supplier self-certification
  - Viable on a purely batch-by-batch analysis basis where the criteria are defined at least by appropriate verifiable RoHS-restricted materials limits.
  - Viable for raw material where the supplier has adequate analysis, reporting and recording facilities which are used on a routine basis.
  - Depends on the existence of implemented Quality Assurance schemes to ensure materials analysis & declarations are reliable.

- Could be backed-up by surveillance from customers which use their products (but most would not want to get involved, and would ask for independent verification).
- b) Formal 3<sup>rd</sup> Party verification schemes
  - Viable on a purely batch-by-batch analysis basis where the criteria are defined at least by appropriate verifiable RoHS-restricted materials limits.
  - Viable for items where independent standards and tests are sufficiently defined.
  - Viable for items where suppliers' own standards are sufficiently defined to permit independent verification on routine, or batch-by-batch, bases.
  - Where none of the above are possible, then it will be necessary to ensure that the analyst has the expertise required which include both analysis skills and a good knowledge of the construction of electronic components. To enable this process to be used by unskilled personal, it would be necessary for them to either follow detailed standard procedures which are currently not available or to follow a "guide to analysis of electronics". The EU may wish to sponsor the preparation of such a guide.

Existing schemes that may be useful for some materials and components:

- Underwriters Laboratories (UL Inc.) "Recognized Materials" schemes for plastics formulators and plastic components (including Printed Circuit Boards) fabricators.
- General materials quality inspection schemes run by BSI Inspectorate, ITS and SGS etc., which are large independent inspection organisations .
- Electronic component quality schemes (e.g. like the old BS 9000 & CECC schemes) run by various organisations.

### **Certification Terminology & Issues**

Conventionally, and in simple terms, the following terms are in common usage:

"Certification" – "someone" "certifies" (attests, confirms, in writing) that "something" meets a given set of criteria, generally after some form of testing and/or assessment.

Certification can be issued for the assessment of a single unit, for a single complete batch, or for repetitive batch or continuous production , of the "something".

Certification of routine batches or continuous production generally requires and includes some form of routine periodic review and auditing system to verify continued compliance with the required criteria.

- The term "Certification" is generally qualified to indicate the source of the confirmation, i.e.:
  - "1<sup>st</sup> Party" – the "someone" is the manufacturer or supplier.

- “2<sup>nd</sup> Party” – the “someone” is the purchaser or user.
- “3<sup>rd</sup> Party” – the “someone” is entirely independent of either the 1<sup>st</sup> Party or the 2<sup>nd</sup> Party, and has no financial or other interest in favouring either.

In general 3<sup>rd</sup> Party certification is usually financed by the 1<sup>st</sup> Party in order to provide the 2<sup>nd</sup> Party (or Parties) with independent assurance that the “something” does meet the required criteria.

- Any of the above approaches to certification could be applied to the control of RoHS-restricted materials.
- Physical Form of the Certification – Various methods are in common use, notably:
  - Certification markings on products (“CE”, BSI Kite mark, VDE “Triangle” mark, “GS”, “S”, etc).

These are normally protected trade-mark symbols which are applied additionally to formal “paper” certification of some form - and do not replace the latter - as a visual method of indicating the basic certification status of the item to 2<sup>nd</sup> Parties. No markings are required by the RoHS Directive as it is assumed that if a product is put onto the market, it is presumed to comply the RoHS Directive. Marks for equipment are a possibility but this will be impractical for most components which are too small and have no space for any marks, or for materials which are supplied in bulk format and used as required (e.g. solder, sheet metal and plastic) where marks would be destroyed during processing.

- The “Certificate of Conformity” (“CoC”) conventionally used by a 1<sup>st</sup> Party to certify that a batch of items, *and only those items*, supplied to a 3<sup>rd</sup> Party meets the requirements stated in the relevant purchase order. Normally, a CoC is a general document and makes no reference to compliance with regulations and / or standards.
- The “Declaration of Conformity” (“DoC”), or more correctly the “Supplier’s Declaration of Conformity”, per ISO/IEC Guide 22 and EN45014, which is a more formalised version of the “CoC” – and is still a 1<sup>st</sup> Party document. A DoC may refer to one item, one batch, or to continuously produced items – but is typically the latter
- The “EU Declaration of Conformity”, as described in various EU “New Approach Directive”, which is required under those Directives as a 1<sup>st</sup> Party statement by the EU manufacturer or importer that the “something” complies with the relevant requirements of those Directives. Once the EU DoC has been prepared and signed, the CE Marking can be applied where required. A DoC may refer to one item, one batch, or to continuously produced items, but is typically the latter.

Note: Not all EU Directives require the CE Marking, and it is an offence to apply it in respect of requirements covered by Directives which do not require it!

- IECEE (Worldwide System for Conformity Testing and Certification of Electrical Equipment) certification body schemes with IEC standards could be used when these standards exist. This would enable products made outside of the EU to be tested locally against the standards. However, these are typically applicable only to complete products in respect of existing IEC standards which do not address RoHS requirements.
- “Materials Safety Data Sheet” (“MSDS”), also sometimes referred to as a “Safety Data Sheet” or a “COSHH Data Sheet”) which the 1<sup>st</sup> Party is required under hazardous substances legislation to make available to 2<sup>nd</sup> Parties. It gives details of any hazardous materials that are present and the safety precautions to be taken in transporting, storing, using and disposing (etc.) of them. This is not ideally suited for RoHS as the absence of hazardous materials is currently not required or indicated; and MSDS are nearly always for materials and only very rarely for fabricated parts.

The MSDS is currently not a “certificate” as such, is generally considerably larger than a CoC or DoC and is not required to carry all the 1<sup>st</sup> Party details and authorities listed in ISO Guide 22/EN45014.

The MSDS could be expanded to include RoHS-restricted materials data, but would then also need to include the source data and authority statements currently required by the DoC format.

- Any of the above forms of certification documentation could be applied to the control of RoHS-restricted materials, but it should be noted that most (if not all) are “paper based”, and that any electronic versions are generally considered to be “for information only” and not to be considered as bona fide “source material” for legal purposes.
- The trust which can be placed in any “certification” is totally dependent on the confidence that can be placed in the organisation and person that issued it, so all of the following questions should always be asked:
  - Where certification markings are visible, does the relevant documentation exist?
  - Is the certification documentation “legal”; i.e. does it provide the information required in relevant legal requirements (e.g. Directives) or commercial contracts?
  - Does the certification documentation give adequate indication of the criteria met and any required related information?
  - Is there any additional evidence not on the certification documents which is required by 2<sup>nd</sup> Parties for the purposes of legal compliance and safety?
  - Is the latter data readily available to whoever needs it in forms that they can easily and correctly apply?

- In particular, if the additional data has been gathered in the form of “chains of evidence” (say from multiple entities which serially handle a certain item, or where there are multiple sources of that item), then:
  - How easy will it be to obtain ALL the information about the effects of those chains?;  
*and / or*
  - How reliable is any certification about those intermediate stages which is being passed to you by your supplier?
- Is the issuing organisation, and the person signing it, capable, competent and authorised to do so and is it reliable, ethical and trustworthy?

The work required for collecting RoHS compliance certificates and any analysis of data would be significant. This is less of a problem to very large manufacturers but SMEs will find this task very difficult, too time consuming and expensive unless a simple compliance demonstration procedure is made available for them to follow.

### 5.2.2 Self-declarations procedure

Producers of electrical equipment will be required to ensure that their products comply with the RoHS Directive and this will be the same for original equipment manufacturers based within the European Union and for importers of equipment into the European Union. The RoHS Directive does not require producers to keep records or to analyse their products to confirm compliance. However, if enforcement authorities discover that a product is not compliant, they will expect the producer to be able to demonstrate that they have taken steps to comply with the Directive and this can be done only with documentary evidence in some form.

The logical first step would be to use materials declarations obtained from suppliers for materials, parts and equipment. The possible format for these is discussed in Section 7.2 but the minimum information required is that the materials, parts or equipment comply with the RoHS concentration limits for all of the individual homogeneous materials of which they are composed. Clearly, it is easier for the original component manufacturers to carry out any analysis that is required to make such a declaration because the analysis of materials used to make components is always easier than analysis of completed components or equipment. Frequently, these manufacturers will be based outside of the EU and it will be difficult for EU based producers to authenticate these claims without themselves analysing these products. It will be a matter of judgement to decide whether any materials declaration are likely to be reliable. There are several criteria that could be used to make an informed decision on this question:

- Usually, the laboratory that carried out the original analysis is not indicated on materials declarations. Accreditation and reputation would influence trust in the data.
- Check reliability of declarations by chemical analysis of a small number of randomly selected parts or materials.

- If, from regular spot checks, past declarations have always been found to be reliable, then producers would have confidence in the accuracy of declarations although this cannot be a guarantee.

Many manufacturers believe that it would be wise to periodically analyse materials or parts even if any one of these criteria apply. If none of these apply but a declaration is provided, for example a material or component is obtained from a new supplier of whom the producer has no experience, then it would be prudent to carry out some analysis to confirm the declarations from this supplier. If no declaration is available or if the producer has any doubts about their reliability, then it would be sensible to analyse more parts or materials. In all of these cases, analysis for all six banned substances may not be necessary. The choice of hazardous material analysis should be made based on knowledge of which of the six materials are likely to be present. For example, the electroplated termination coatings on integrated circuit packages should be analysed for lead, as tin/lead is one of the materials commonly used for these coatings. In this case, cadmium and hexavalent chromium are less likely to be found and the presence of mercury, PBB and PBDE is practically impossible.

Producers would benefit from some guidance on whether analysis is necessary and what should be analysed. This could be provided by the EU using a “decision tree” approach such as is used in the EUROOPEN guide<sup>7</sup> to compliance with the Packaging Directive or by using a checklist. These could define when analysis is recommended.

The amount of work that manufacturers are expected to carry out could in part be related to what would be the effect(s) if the certification were incorrect, unreliable, totally spurious, or just not available.

Without certification or a declaration in some format, analysis will usually be advisable. If certificates are wrong, the risk to the environment depends on the type and quantity of banned substance present. The risk to the producer if the enforcement authorities detect a banned substance is that he may be expected to take these products off the market and prosecution may follow if the enforcement authority can show that the producer has not taken reasonable steps to comply with RoHS legislation.

### **5.2.3 Issues for large, medium and small size manufacturing and distribution companies**

Although producers do not need to prove that their products comply with RoHS, if asked by a customer or challenged by an enforcement authority they may be expected to show that they have taken all reasonable steps to comply with national legislation. It is unlikely that “reasonable steps” will be defined in national legislation and it is not required by the Directive but is a common legal defence<sup>8</sup> for situations where it is impractical or impossible to guarantee compliance under all circumstances. There are three important but separate aspects of RoHS compliance demonstration:

- Firstly, producers will endeavour to obtain only RoHS compliant materials and components and obtain a suitable declaration from their supplier or the original manufacturer.

- Secondly, where there is any doubt and also to check on the reliability of declarations, some components and materials will be analysed.
- The number of components used by a particular producer often bears no relationship to the company turnover or the number of products put onto the market.

Large producers will find it easier to carry out whatever is necessary to comply with the RoHS Directive and to demonstrate this with documentation than small and medium size producers.

### **Issues for Large Producers**

Large manufacturing and distribution companies will generally have the critical mass and organisation to cope with the imposition of RoHS-restricted materials controls on their products, and will also be able, to some degree, to impose requirements on their suppliers and the items received from the latter.

They will be able to implement and maintain these controls because existing business-control systems already go a long way to achieving them, and thus probably only require some updating (albeit substantial and possibly requiring more capable software and hardware). For example:

#### Manufacturing Requirements Planning (MRP) Software requirements:

Software & databases *capable of*

- Extension to cope with input of additional data about RoHS materials
- Including RoHS data in “Bills of Materials” (“BoM”)
- “Industry standard” I/O data formats which will deal with data from a variety of source databases, on-line & off-line
- Outputting in paper & electronic “certificate format” using predefined formats.

*Outgoing Shipping* documentation system capable of:

- Predefined format to include:
  - a) Manufacturer
  - b) Unique manufacturing plant identifier (*or VAT or company registration number?*)
  - c) Shipped material / sub-assembly / complete unit identification code
  - d) Shipment reference
  - e) Batch number
  - f) Batch content (number of containers, bags, units, etc)

- g) Shipped to and sold-to company names & addresses
- h) Customer's Purchase Order number
- i) Shipment date
- j) Company signatory or security code verifying correctness of data
- k) Supplying all above information in electronic form to customer for incorporation into his own system process
- l) Certification of compliance. This needs to be signed by a company director.

### *Goods Inwards and Shipping*

Documentation systems capable of receiving and processing above data a) to k).

The enhancements would be required to cope with the input, processing and output of the RoHS-specific materials data.

REMAT (described in section 7.2.3) may be helpful with handling RoHS data.

### **Issues for Small to Medium Enterprises (SME) manufacturing within the EU**

SMEs often do not have MRP systems and so use systems that are essentially, if not entirely, manual in nature and often poorly documented.

In this case, it will be very labour-intensive task to document the RoHS-critical materials' status for each product, especially where the manufacturer or distributor is providing small quantities, or "one-off's", assembled from components and sub-assemblies from a variety of suppliers – many of which will have themselves imported those items from outside the EU.

Collecting (or attempting to collect!) the RoHS compliance information from suppliers will place a heavy time/cost burden on SMEs since this data would need to be collated in a form, which could be audited by the enforcement authorities if they identify a product that does not comply with the RoHS Directive.

Many SMEs trade on their ability to provide bespoke goods and/or rapid delivery of small quantities (sometimes only one unit), and the cost of collecting and delivering the data could be substantial – and even prohibitive if a simple but effective way of recording data is not available. The main aim of the RoHS Directive is to protect the environment and human health – and therefore the overall benefit of these objectives, which require SMEs to perform these tasks, must be carefully assessed.

The first issue for SMEs is how to handle the large amount of RoHS compliance information in a simple and cost effective way. Compliance certificates provided in the form of paper or pdf documents can be collected and filed but may be difficult and time-consuming to locate for auditing.

Products manufactured in small numbers by small and medium size enterprises (SMEs) are likely to have a much smaller impact on the environment and human health because of the smaller quantities of materials used. The potential risk from hazardous substances being used in error by these producers, and not detected by them, will be less than the risk from the same materials used in products made in very large numbers.

#### **5.2.4 Strategies for product manufacturers based in the EU**

EU based manufacturers need a simple and low cost procedure for ensuring that their products are RoHS compliant.

Electrical equipment imported from outside of the EU would need to pass through customs on entry into the EU and so theoretically RoHS compliance could be checked at this point. In reality, however, most products will not be thoroughly inspected even if all Member States agree to rigorously police RoHS compliance of imported products at the point of entry into the EU. There is no possibility that these requirements could be policed in this way, partly because the RoHS Directive does not apply to components or materials and partly because the effort required for this to be effective would be prohibitive.

Hence, producers will need to ensure that the banned substances are absent from all of the parts, components and materials that they use in products that need to comply with this Directive.

Possible methods that equipment producers could use to ensure that any components that they use do not contain a banned substance include:

- Purchase “RoHS-compliant” components with confirmation from supplier in the form of a materials declaration. Ideally this should be hard copy or electronic and could be downloaded from a website. The format is discussed in Section 7.
- Purchase parts, components and materials which are marked to show that the banned substances are absent sometimes referred to as “RoHS compliant”, or in some cases marked “lead-free”.
- Purchase raw materials to a standard that excludes the six banned substances (at concentrations below the limits to be decided by the TAC).
- Analyse some or all components and materials.

It would be extremely expensive to analyse a random selection from every batch of all components (possibly except by handheld XRF), so producers will need to decide how many analyses are necessary and how frequently. This decision could be based on a strategy defined in a Standard, a check list or in guidance notes issued by the EU. In practice, equipment producers and distributors will not want the enforcement authorities to find a material or component that contains a banned substance as this could prevent sales of these products. Therefore they will tend to carry out more

checks than any minimum recommended by a guide or Standard. Criteria for a decision on analysis could include:

- Very large numbers of products are made and, although the risk of incorporating a part containing a banned substance may be very small, the financial impact of taking this product off the market would be very significant. Here the producer might consider carrying out a relatively large number of checks.
- Components or materials obtained from a new supplier of unknown reliability.
- As a result of reports in the press or information regarding counterfeit or inaccurately defined components.
- Routine checking of materials and components which have a higher risk of containing a banned substance. PVC is a typical example of a “high risk” material because in some parts of the world, lead and cadmium are still used as stabilisers and pigments. Components manufacturers tend to use the lowest cost materials which may be from these sources and each batch may be from a different source. Therefore, in theory, producers should check every batch of PVC whereas other plastics that rarely contain lead or cadmium (these can be used as white and red/orange pigments respectively) could be analysed less frequently. PVC is widely used in electrical equipment for example as wire and cable insulation.
- Past experience of parts supplied containing a banned substance due to an error somewhere in the supply chain.

Equipment producers will want to be able to trust their suppliers to supply components and materials that are RoHS compliant, and any other activities would be secondary. Markings to show RoHS compliance on components, parts and sub-assemblies, such as power supplies, PCBs made by sub-contractors and modules, may be accepted as sufficient evidence of compliance where these are used, but may not always be used as a guarantee of compliance if these have no legal basis. Obtaining material declaration certificates and carrying out analysis will be time consuming and costly, especially for SMEs, and so producers would want to minimise these activities.

Many of the parts (e.g. bulk metal and plastic materials) used in electrical products are more widely used in other industries, and the manufacturers and distributors of these items may not be aware of the RoHS Directive’s requirements or be able to provide material declarations. Equipment producers will sometimes be faced with difficult decisions where these suppliers may be lower cost than those that are able and willing to guarantee RoHS compliance – or where no such supplier exists.

It is equipment producers, however, who have the responsibility for ensuring that their products comply with the RoHS Directive, not the suppliers of components, materials or sub-assemblies, and so the duties on them could be onerous.

## 5.2.5 Strategies for companies importing products into the EU

Companies which import products will face similar issues to those manufacturing in the EU.

Those issues can probably only be dealt with successfully in technical terms “at source”, i.e. in the countries where the goods are manufactured or where final assembly takes place, but such controls are outside the remit of EU based enforcement authorities. Appropriate controls could be applied via “the port(s) of entry” to the EU (as with other controls on such items) as is already used in some EU Member States to prevent the import of products containing banned substances. Random checks are carried out by the Dutch customs authorities who found in 2001 high levels of cadmium in parts of Sony PlayStation games machines<sup>9</sup>. This is banned by the Cadmium Directive (76/769/EEC) and as a result, Sony were not permitted to sell 1.3 million of their products until these parts had been replaced. The UK Customs and Excise (and its equivalents in other States) would need to appoint 3<sup>rd</sup> parties to carry out this work if enforcement authorities use this option.

Equipment is imported into the EU from non-EU manufacturers either direct to distributors and retailers or via locally based holding companies. Under these circumstances, the RoHS Directive defines these as the “producer” who is responsible for RoHS compliance and would need to use the same procedures as EU based manufacturers.

In addition, there are some other options which have precedents for the transportation of goods that could be considered. These are not requirements of the RoHS Directive but could be used by enforcement authorities to prevent the importation of non-compliant products:

- Non-EU shipper to provide certificates that items contain no RoHS restricted materials at all, as part of Shipping documents and Customs Declarations - with substantial penalties for false-declarations. This would be a requirement on organisations or individuals that are not based in the EU and so will be difficult to enforce if there is no EU based importer.
- Airlines and shipping lines that “import” illegal aliens can be fined and are required to transport them away again, also, drivers of road transport are fined for carrying illegal immigrants. Although it is producers who are responsible for RoHS compliance, there are precedents for carriers to bear some responsibility for the transportation of “hazardous” goods. Some existing legislation and other related requirements already apply and are policed under the various implementation of the international UN, EU and national legislation covering the transport of dangerous goods, viz:
  - RID "Transport of dangerous goods by rail " = 98/37/EC
  - ADR "Transport of dangerous goods by road" = 94/55/EC
  - IMDG "International Maritime Dangerous Goods Code"
  - International Civil Aviation Organisation "Technical Instructions for the Safe Transport of Dangerous Goods by Air".

Extending this responsibility to RoHS compliance could be considered but shipping companies will be very reluctant and would find it very difficult to guarantee “RoHS compliance”.

Importers (who will be the “producers”) should hold Technical Files on all imported items – regardless of the requirements of other Directives – including details of the presence or absence of RoHS-restricted materials as declared by the non-EU suppliers.

Such Files might be needed anyway for other purposes, such as to support CE Marking under New Approach Directives – and the task of acquiring and preparing RoHS data could then theoretically be passed back to those non-EU suppliers in the majority of cases.

The veracity of the data would be open to question, however, unless supported by appropriate supplier qualifications akin to EN 14001. That said, it might be possible to enforce the requirements by rigorous market policing and testing – but that will be a more expensive process than for traditional safety and EMC issues.

It should be noted that it is relatively easy to conduct traditional electrical and mechanical (etc.) safety tests on samples of a product, but it is far more difficult to identify all the materials (especially “trace elements”) in all incorporated components without the use of expensive and potentially destructive chemical and/or physical analysis techniques.

### **5.3 Achieving a balance between environmental benefit and demonstrating compliance**

Any compliance procedure should aim to protect the environment and human health as that is aim of the RoHS Directive. It would be reasonable, therefore, to limit what is expected from producers to meet this aim. Any additional activities beyond this that have an insignificant benefit would be a waste of resources and could themselves have a negative environmental and health impact.

It is not unusual for individual items of electrical equipment to contain 1000 or more individual parts. If only one of these parts contains a banned material at a concentration above the limit, then this equipment does not comply with the RoHS Directive and should not be put onto the market. However, the effort required to find non-compliant materials or components should not be disproportionate to the resultant effect on human health or the environmental. Many variables influence this including:

- Number of product put onto the market
- Weight of product put onto the market
- Weight of banned substance in product, weight of same substance in exempt forms or as an impurity at a concentration below the permitted level
- Significance of the quantity of these six materials in comparison with the amounts entering the environment from other sources

- Quantity of these six materials used in EEE either in exempt applications or in products not covered by the RoHS Directive.

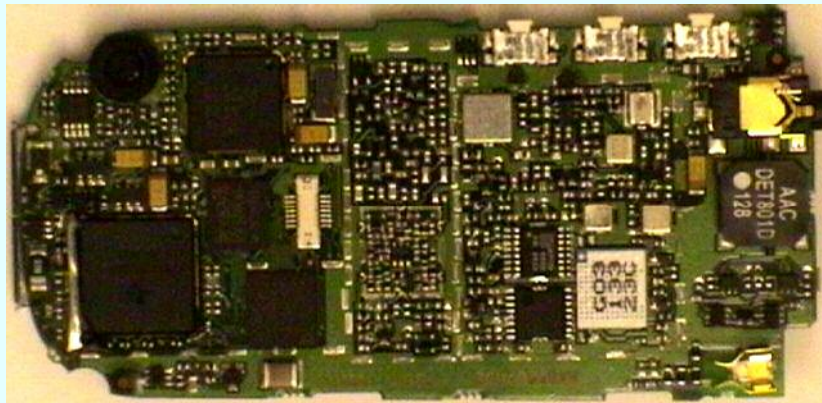
### 5.3.1 Quantity of EEE put onto the market

Some types of consumer electronics equipment are sold within the EU in very large numbers whereas some specialist commercial products may be produced in numbers of a few hundred units or less. In 2001, 110 million mobile phones and 30 million personal computers were sold in Western Europe. Some companies have sold many millions of one type of product; for example, Sony had sold over 12 million Play Station 2 in Europe by September 2002.

Clearly the effect on the environment from a several million non-compliant consumer products will be considerably larger than the effect of a few hundred products containing the same non-compliant components.

The number, size and type of parts that contain banned substances used within a particular product will also affect the environmental impact. This is illustrated by the following examples:

**Example 1:** A mobile phone contains several hundred individual parts, most of which are on the PCB. Most components are attached to the laminate using surface mount technology (SMT) and so all components are attached using one type of solder. Published figures for the amount of solder used within a mobile phone are typically 0.8 g which contains 36% lead and so each phone will contain about 0.3 g lead, most present in solder<sup>10</sup>.



**Figure 1. One side of a typical mobile phone PCB from the late 1990's**

After the PCB is produced but before components are attached, a protective coating is used to prevent oxidation of the underlying copper. Many types of PCBs are protected with a tin/lead solder coating applied by a process called Hot Air Solder Levelling (HASL). Mobile phones however use different types of coatings. The other potential source of lead in a solder joint is the coating used on the terminations of components. These are usually electroplated coatings of tin or tin alloy although other materials are used on a relatively small percentage of components. Tin containing up to 20 weight % lead is commonly used although many manufacturers are gradually phasing out and replacing the lead alloys.

To comply with RoHS, all components should not use tin/lead alloy termination coatings. The quantity of lead used in the coating on one component however can be very small. Each mobile phone can contain over 100

**Example 1 (continued):** MLCCs. These are produced in various sizes but mobile phones usually have only the smallest. One "0603" MLCC (this is not the smallest) has been calculated to contain only 0.05 mg of lead in the termination coating.

Philips Semiconductors published the chemical content of a wide range of their products in 2002<sup>11</sup>. Many of these will have since changed as RoHS compliant products are developed but the data in this publication is useful as the quantity of lead in each component is given. Table 2 gives the relative quantities of lead from various sources and the quantity of lead that could potentially enter the environment.

**Table 2. Relative environmental impact for different sources of lead in a hypothetical mobile phone**

| Source of lead   | Quantity in 1 mobile phone weight (percent in brackets <sup>a</sup> ) | Quantity of lead in 1 million mobile phones |
|--|---|---|
| Solder used on PCB   | 300 mg (0.3%)   | 300 kg                                      |
| One 0603 MLCC  | 0.05 mg (0.00005%)  | 0.05 kg                                     |
| One Quad Flat Pack IC with 64 terminations (Sn15%Pb plated) <sup>b</sup> | 10 mg (0.01%)   | 10 kg                                       |

a. Assumes mobile phone total weight 100 grams

b. Data from Philips Semiconductors

**Example 2:** Most of the lead in a desktop computer is used in the cathode ray tube - one of the listed exemptions in the RoHS Directive. The quantity of lead depends on the screen size and can be 2 kg of lead per screen. Lead may also be used as solder, in component terminations and in HASL coatings. One type of solder and the HASL coating is used to produce all solder bonds on a PCB whereas each component termination coating can be different. Miniaturisation is not as important as in a mobile phone and so larger components with more solder in bonds are used. A typical PC with monitor will contain well over 1000 electronic components plus various plastic and metallic parts. A similar calculation as that for the mobile phone gives the data in Table 2.

**Table 3. Relative environmental impact for different sources of lead in a desktop computer**

| Source of lead  | Quantity in 1 computer (weight and % in brackets <sup>c</sup> ) | Quantity of lead in 1 million computers |
|---|---|---|
| SMT solder (37% lead)   | 48,000 mg (0.18%)   | 48,000 kg                               |
| HASL solder <sup>d</sup>  | 17,000 mg (0.06%)   | 17,000 kg                               |
| One 0805 MLCC   | 0.15 mg (0.000006%)   | 0.15 kg                                 |
| Power transistor <sup>e</sup><br>die attach   | 18.5 mg (0.00007%)  | 18.5 kg                                 |
| SnPb plating on terminations  | 7 mg (0.00003%)   | 7 kg                                    |
| Plastic leaded chip carrier with 44 terminations (Sn15%Pb plated)                         | 2.5 mg (0.00001%)   | 2.5 kg                                  |
| Lead impurity from lead-free SMT and HASL solders assuming that these contain 0.03 % lead | 19.5 mg (0.00007%)  | 19.5 kg                                 |

c. Assumes one personal computer with CRT monitor weighs 27 kg. Typical weight PCBs in processor unit is 1 kg and in monitor about 1.4 kg. In both, solder content is 6% (so 2 weight % lead).

d. In this example, assume that HASL coatings are used on all PCBs. Ref. 14 states that HASL provides 25% of the lead in a solder bond.

e. From ST Microelectronics. Die attach alloy has >85% lead so is exempt from the RoHS Directive.

**Example 2 (continued):** An additional point that should be considered is the amount of the banned substances that could be present as impurities in the alternative materials. Lead-free solders are predominantly tin. Commercial tin used for solders is not totally lead-free, it would be both impractical and expensive to remove all traces of impurities in tin. The maximum lead content in tin used for production of solders (BS3252:1986) is 0.05% lead and a lead content of about 0.03% is typical. The quantity of lead from impurities in solder are included in the Table 3.

These figures help to put the effect of each material into a realistic perspective. A single MLCC that is not RoHS compliant due to lead being used in the termination coating is relatively insignificant in comparison with the quantity of lead that will be present as an impurity in lead-free solder. In fact there would need to be 130 non-RoHS compliant MLCCs to equal the amount of trace impurity lead.

## 5.4 Analysis requirements

There will be occasions when analysis would be advisable which will depend on a variety of issues:

- **Reliability of information from suppliers.** This will inevitably vary and producers will, as a result of experience, determine whether materials declarations from individual suppliers can be trusted. Periodic checks or audits of their suppliers will be advisable, especially when the financial effect on the equipment producer would be very large if the enforcement authorities find a non-compliant product. Checks could take the form of analysis of randomly selected components or materials (with an emphasis on higher risk items) or a formal audit of the procedures used or both. Auditing could be difficult where there are long supply chains especially if these originate from very distant locations.
- **Higher risk materials.** There will be a higher risk in certain materials that a banned substance could be present than in others (e.g. lead and cadmium in PVC). These would require more frequent analysis.
- **Source of analysis data.** If the source of materials declaration data is not known and the producer has no previous experience with a supplier, this will result in some doubt over the suitability of materials or components which can be resolved by independent analysis of all or some randomly selected parts.
- **Components from grey market.** Some manufacturers are forced to obtain components from the so-called “grey market”. These are often components that are no longer produced by the original producer and so to continue manufacturing without going to the trouble and expense of a complete redesign, the equipment manufacturer resorts to obtaining components from other sources. These components are unlikely to have documentary evidence of compliance with RoHS and this may be unreliable even if it is available.
- **Counterfeit components.** There has been a relatively large number of counterfeit components in circulation for many years. These are outwardly very similar to the genuine parts but internally may be very different and there is no guarantee as to their RoHS compliance. Individual components and assemblies are forged. In 2002, counterfeit Compaq memory

boards and hard drives were seized in the USA and there are many reports of counterfeit semiconductors, capacitors and other components. Some of these are clearly defective but many function correctly albeit with limited performance. The organisations that produce counterfeits are criminals who would have no difficulty providing false materials declarations.

#### **5.4.1 Analysis issues and competent authorities**

##### **Frequency of testing**

Repeat analysis will be needed if it is likely that the composition of components, assemblies or materials have changed. If the production location changes (some manufacturers make individual components in more than one factory), the design, sub parts or source of raw materials may also be altered. None of these occur on a regular or predictable basis but it is known that component compositions can vary. Producers ideally should be aware of any changes in their supply chain but often these variations will not be predictable and repeat analysis will be required to ensure continued compliance. This will be increasingly important when the quantity of recycled materials used increases as this is likely to create a larger variation in composition and increase the risk of banned substances being present.

Over time, producers will develop analysis histories for their suppliers. Several years experience without problems would suggest that the frequency of analysis be reduced. Also, as the effect of the RoHS Directive spreads, the six banned substances will be used in decreasing quantities and the need for analysis will fall.

##### **Competent authorities for carrying out tests**

The approach used for some New Approach Directives is for Member States to appoint Notified Bodies who will be organisations that are deemed to be competent who are able to test products to demonstrate that they comply with the requirements of Directives. Notified Bodies should be independent third parties and should not be used by Member State Governments as the enforcement authority as this would create a conflict of interest. Some New Approach Directives require equipment manufacturers to use Notified Bodies, usually where there is a high risk, but it is optional for other Directives and not a stated requirement for the RoHS Directive.

Analysis can be carried out in-house by equipment producers or sub-contracted to a competent organisation. Analysis carried out by an independent third party on behalf of component or material suppliers may provide more confidence to equipment producers than if this is carried out by their supplier in-house. Accreditation of test laboratories can give confidence in test results but may not guarantee that data is more reliable than if it is produced by laboratories without accreditation. Technicians with appropriate skills and good motivation will produce accurate results whether or not they are working in an accredited test house. It is more important that analysts fully appreciate the limitations of analytical techniques and to know what is required to obtain accurate results. Auditing by an independent organisation (such as Underwriters Laboratory for plastics) may be considered as a means of maintaining accuracy and for customers to have greater confidence in results.

## 5.5 Views of the electronics industry on the need for analysis

The views of the electronics industry have been promoted by trade associations including EICTA, ESIA, AEA and JBCE and have been taken into account in producing this report. Soldertec recently carried out a survey of about 70 organisations, mostly working on an international basis on lead-free markings<sup>12</sup>. This included several questions on the need for analysis, in particular:

Q. How should materials and products be certified as lead-free?

| Scenario   | Percent |
|--|---------|
| Material supplier certifies compliance and purchaser carries out random checks   | 32%     |
| Material supplier certifies compliance but purchaser does not check  | 26%     |
| Material supplier certifies compliance and purchaser carries out checks on every batch (Simple screening tests such as with handheld ED XRF) | 15%     |
| Official certificate required  | 8%      |
| Other  | 14%     |
| Don't know   | 10%     |

At least 47% of those surveyed, which include equipment producers, distributors and component manufacturers, intend to carry out at least some analysis.

Q. What type of test procedure would be used?

| Scenario – test methods   | Percent |
|---|---------|
| Material and components must satisfy simple colour reaction "litmus" test     | 14%     |
| Material and components must satisfy simple XRF type check                    | 27%     |
| Material and components must satisfy simple SEM-EDX type check                | 18%     |
| Material and components must satisfy full chemical analysis (ICP etc.) check  | 41%     |
| Scenario - procedure  |         |
| A standard company test procedure is carried out by own company and suppliers | 45%     |
| A standard company test procedure is carried out by external test agency      | 10%     |
| An industry standard test method is required                                  | 45%     |

Soldertec found a surprisingly high percentage that was prepared to carry out full analysis of components and materials. Many planned to use a combination of techniques for example a simple XRF check followed by full chemical analysis if the check was not definitive. Although many would like to see industry standard test methods developed, the larger manufacturers prefer to use their own standard analytical methods.

JBCE believes that as much random analysis as possible should be carried out. Component manufacturers whose main markets are in Europe, Japan or USA will be familiar with the RoHS Directive and are in the process of developing products that do not contain the banned substances. Those whose main markets are in Africa, South America, etc. may not know of the RoHS Directive and so will not be eliminating the banned substances in their products. This view regarding awareness of RoHS is supported by the findings of a recent DTI mission to South East Asia<sup>13</sup>.

## 5.6 Conclusions regarding self-declaration

1. Self declaration is used as the basis for compliance for many other EU Directives and is suitable for the RoHS Directive.
2. Any product put onto the market would be presumed to comply with the RoHS Directive and enforcement authorities will carry out market surveillance to detect non-compliant products.
3. If the enforcement authority finds a non-compliant product, the action they take will depend on the extent to which the producer has taken reasonable steps to prevent non-compliant products from being put onto the market. Most producers will want to be able to provide evidence to show that they have taken reasonable steps although what is reasonable will not be defined and is open to interpretation.
4. Significant challenges face all equipment producers who will need clear guidance as to what they will be expected to do to ensure that their products comply with the Directive's requirements.
5. Most producers are conscientious and will endeavour to ensure that their products are RoHS compliant by using two complementary approaches:
  - o Obtaining Declarations: Identifying components and materials that do not contain banned substances and obtain materials declarations from their suppliers. Some equipment producers prefer these as formal certificates, which can be shown to enforcement authorities if necessary. Some producers currently believe that these will be sufficient and that analysis is not necessary.
  - o Supporting analysis: Many equipment producers plan to, and some already do, check supplier materials declarations by analysis. This is a sensible precaution, especially where no declaration is available, but also as a random check to audit suppliers. The extent to which analysis is necessary will depend on the risk arising from the use of a banned substance.
6. The criteria for analysis will depend on the quantity of product put onto the market (less for small producers than for large producers), the relationship with suppliers and the risk of a banned substance being present.
7. Producers and enforcers should be permitted to use any appropriate analytical technique (as with other EC Directives). They should be aware of the limitations of some of these, however, in particular the low cost screening analysis methods.
8. The extent to which analysis is carried out will depend on many variables. Producers will consider the financial implications but the potential impact on the environment should be another factor for both producers and enforcement authorities. The environmental impact of a non-compliant product being placed on the market is a complex issue affected by many different factors. The enforcement authorities could, for example relate penalties to environmental and health impact. If, for example, the producer has taken reasonable steps to comply with RoHS but a banned substance has been included inadvertently, and if the impact on the environment is insignificant, the authorities may not require these products be removed from sale, only that the defect is be

corrected or taken into account in new products. **This policy, however, needs to be harmonised in all Member States.**

## 6. Standards for Compliance Testing

### 6.1 Development of Standards and present status

At present, no Standard exists that producers or enforcement authorities can use to determine whether products comply with the requirements of the RoHS Directive. This is not surprising as the Directive was adopted by the EU in February 2003 and some final details have not yet been resolved. As a result of the procedures followed to produce EN and CEN standards, the time taken to produce a suitable Standard would be at least two years. Hence, it is unlikely that a Standard will be available by 1<sup>st</sup> July 2006 when the material bans come into force.

When previous self-declaration Directives such as the Packaging Directive (94/62/EC) were introduced, there were no Standards that covered the entire compliance procedures. The Packaging Directive has some similarities with the RoHS Directive.

- Both are non-CE mark Directives
- Both include specific substance bans.

The Packaging Directive is more complex as there are other requirements and the substance bans are only one aspect of this Directive. The Packaging Directive was adopted in 1994, a guide designed to help companies to establish a self-assessment procedure was published in 1999 and the first of a series of CEN standards were published in 2000.

This is clearly too long a period for electrical and electronic equipment producers to wait for Standards or guidance as they need start work towards compliance as soon as possible. To do this they will need to use a combination of existing standards and well established analytical methods to assess materials, components and products.

### 6.2 Existing standards

Few standard analytical procedures exist for determination of the composition of materials within electrical components and equipment. There are existing Standards for the sampling and analysis of specific materials - mainly metals.

A search for Standards used worldwide has identified a small number of European Standards that may be directly applicable as well as other related Standards including some US Standards that will be useful as a basis for new Standards. These Standards are given as Table 4.

Table 4. Existing Standards for analytical procedures

|                     | Standard   | Suitability  |
|---------------------|--|--|
| Lead                | EN 12402:1999, "Lead and lead alloys – methods of sampling for analysis"   | Detailed procedure for sampling bulk lead and its alloys in the form of ingot. Not suitable for components or solders in other forms. Can be used to analyse high lead content solders   |
|                     | BS 6534:1984, "Method for quantitative determination of lead in tin coatings"  | Suitable for analysis of tin coatings on component terminations and on unpopulated PCBs. The procedure will need to be modified for analysis of lead in tin alloy coatings to account for the additional elements  |
|                     | EN 12441-3:2001, "Zinc and zinc alloys – chemical analysis – part 3. Determination of lead, cadmium and copper – Flame atomic adsorption spectrometric method"   | Suitable for analysis of bulk zinc and zinc alloys   |
|                     | BS 6721-9:1989, ISO 4749-1984, "Sampling and analysis of copper and copper alloys. Method for determination of lead in copper alloys by flame atomic absorption spectrophotometry"                       | Suitable for checking lead content of copper and copper alloys used to make parts for EEE. Analysis by dissolution then AAS. Measures lead from 0.002% to 5%. (lead in copper permitted as alloying element up to 4%)  |
|                     | BS 3338-5:1961 Methods for the sampling and analysis of tin and tin alloys. Method for the determination of lead in ingot tin and tin-antimony solders (photometric method)                              | Suitable for materials (e.g. as ingot). BS 3338-21:1983 is for cadmium in soft solders   |
| Cadmium             | EN 1122:2001, "Plastics. Determination of cadmium. Wet decomposition method"<br>(DD ENV 1122:1995, "Determination of cadmium in plastics with the method of the wet decomposition" (withdrawn, revised)) | EN 1122:2001 is for analysis of cadmium (from 10mg/kg to 3 g/kg) in non-fluorinated plastics. The plastic is decomposed and the cadmium dissolved. This solution is analysed by AAS. Suitable for plastics used to construct electrical equipment  |
|                     | BS 3900-B9:1986, ISO 3856-4:1984, "Methods of test for paints. Tests involving chemical examination of liquid paints and dried paint films. Determination of 'soluble' cadmium content"                  | Specific test procedure for cadmium in paints. Cadmium could be used as a pigment  |
| Hexavalent chromium | BS3900-B10:1986, ISO 3856-5:1984, "Method of test for paints, tests involving chemical examination of liquid paints and dried paint films. Determination of hexavalent chromium content of solid matter" | Procedure for determination of Cr(VI) content of dried paint films in concentrations from 0.05 to 5% Cr. The paint film is first dissolved and this solution is analysed   |
|                     | BS 6068-2.47:1995, ISO 11083:1994, "Water quality. Physical, chemical and biochemical methods. Determination of chromium (VI). Spectrometric method using 1,5-diphenylcarbazide"                         | One of a series of water analysis standards. Unsuitable for electrical components but could be adapted for use for the analysis of solutions of coatings   |
|                     | BS EN ISO 3613:2001, "Chromate conversion coatings on zinc, cadmium, aluminium-zinc alloys and zinc-aluminium alloys. Test methods"  | Colorimetric method based on diphenylcarbazide. Used for detection of CrVI and for determination of mass/unit area of coatings applied more than 24 hour previously and less than 30 days previously. This is a limitation for analysis of coatings on components that might be much older. The method described determines the concentration of only water soluble CrVI |

|  | Standard   | Suitability   |
|--|--|---|
| Less relevant standards for analysis of water, air, etc.         | BS EN ISO 5961:1995, BS 6068-2.21:1995, "Water quality. Determination of cadmium by atomic absorption spectrometry"                            | Series of standards for analysis of cadmium and other elements in water and other materials using various techniques. New standards could be produced for cadmium in electronic component materials based on these  |
|  | BS EN ISO 11885:1998, BS 6068-2.60:1998 Water quality. Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy |   |
|  | EN 13346:2000, "Characterization of sludges. Determination of trace elements and phosphorous. Aqua regia extraction methods"                   | Could be adapted for analysis of components. Aqua regia will dissolve most metals including gold. The extract solution can be analysed by AAS or ICP  |
|  | EN 12338:1998, "Water quality. Determination of mercury. Enrichment methods by amalgamation"   | Not directly useful for electrical components. Method applicable to water containing very low mercury concentrations  |
|  | CR 10299:1998, "Guidelines for the preparation of standard routine methods with wavelength dispersive X-ray fluorescence analysis"             | This is a different technique to energy dispersive XRF which is used in handheld and desktop XRF that are usually used for identification of non-RoHS compliant materials. Wavelength dispersive XRF gives more accurate analysis than ED XRF but is not easy to use with components or thin coatings |
|  | National Environmental Methods Index (NEMI), USA   | Large number of methods for analysis of trace levels in environmental samples including lead, mercury, cadmium and hexavalent chromium in water   |
|  | Occupational Safety and Health Administration (OSHA), USA  | Analysis methods for hexavalent chromium in air   |
|  | National Institute for Occupational Safety and Health (NIOSH) – manual of analytical methods   | Analysis methods for hexavalent chromium in air   |
|  | EPA Methods  | Comprehensive list of standards for sampling and analysis of environmental samples such as water, soil and air. Not directly suitable for components  |
| Japanese Standards Association, JIS H 1141 1993, Analysis of tin | Measurement of metals including lead in bulk tin   |   |

### 6.3 Analysis methods

Methods for analysis of electrical equipment were suggested in responses to the UK Government's discussion paper published March 2003<sup>14</sup>. These, and others, are listed in Table 5.

**Table 5. Analytical methods**

| Method  | Materials analysed                      | Method   | Limitations   |
|---|---|--|---|
| <b>Atomic absorption spectroscopy (AAS)</b>   | Pb, Cd, (Hg if cold vapour method used) | Material for analysis is first dissolved   | Analysis of solutions   |
| <b>Inductively coupled plasma spectroscopy (ICP)</b>  | Pb, Cd                                  | Material for analysis is first dissolved   | Analysis of solutions   |
| <b>UV/Visible spectroscopy</b>  | Hexavalent chromium (CrVI)              | Material for analysis is first dissolved   | CrVI needs to be in solution  |
| <b>Energy dispersive X-ray analysis with a scanning electron microscope (SEM/EDX)</b>                 | Pb, Cd, Hg compounds, Br, Cr            | Surface analysis technique. Area analysed typically 1 µm diameter with depth <1µm. | Detection limit close to 0.1%<br>Cannot determine oxidation state of Cr<br>Identifies Br but does not identify compounds  |
| <b>Spark emission and DC arc emission spectroscopy</b>  | Pb, Cd, Hg                              | Analysis of metals   | Sample preparation not required if material to be analysed is at the surface.   |
| <b>Glow discharge optical emission spectroscopy</b>   | Pb, Cd, Hg, Br, Cr                      | Analysis of thin coatings  | Can be used to analyse multi-layer coatings   |
| <b>Polarography</b>   | Pb, Cd, Hg and CrVI                     | Analysis in aqueous solution   | Copper interferes with CrVI analysis  |
| <b>High performance liquid chromatography (HPLC)</b>  | Brominated flame retardants             | Carried out on liquids   |   |
| <b>Ion chromatography (IC)</b>  | Brominated flame retardants             | Material for analysis is first dissolved   |   |
| <b>Gas chromatography mass spectroscopy (GCMS)</b>  | Brominated flame retardants             | Complex multi-step procedure   |   |
| <b>Energy dispersive X-ray fluorescence analysis (ED-XRFA)</b><br><b>Handheld and desktop methods</b> | Pb, Cd, Hg, Br, Cr                      | Surface analysis, can be non-destructive.<br>Accurate on flat surfaces             | Handheld instruments have limited accuracy. Desktop instruments have limitations. Widely promoted as low cost reliable technique for electronics but recent research shows that accuracy can be poor if not used correctly.<br>Neither is reliable for whole PCBs |
| <b>Wavelength Dispersive X-ray fluorescence analysis (WD-XRFA)</b>                                    | Pb, Cd, Hg, Br, Cr                      | For analysis of homogeneous materials  | Surface analysis so not suitable for components   |
| <b>Fourier transform infra-red (FTIR) spectroscopy</b>  | Brominated flame retardants             | Can be used with a plastic or an extract   | Identification of flame retardants at high concentrations (>3% Br) but has limitations  |
| <b>"Litmus" test</b>  | Lead present at surface                 | Simple screening test  | Designed for identification of metals that contain lead at concentration >1%.   |

The techniques listed in Table 5 have been assessed in more detail for their suitability for materials and component analysis and this is described in Annex 1.

Before the methods listed in the above table can be used, some sample preparation is essential. The way this is carried out depends on the choice of analytical technique as well as whether the RoHS concentration limits apply to single materials or to individual components as discussed in section 4. The main sample preparation techniques for components and for materials are listed in Table 6.

Enforcement authorities and some producers will be presented with complete electrical products or assemblies for assessment. Some dismantling is required for reliable and accurate analysis although some parts can be analysed without dismantling including:

- Flat surfaces of area greater than 1 cm<sup>2</sup> (plastics or metals for Pb, Cd, Hg, Cr and Br) using handheld XRF (calibration using representative standards essential for accurate results).
- Undersides of single sided PCBs (relatively flat surface) using desktop XRF. This will identify where there is Pb, Cr, etc which may correspond with particular components. Unpublished research by one manufacturer has shown that scanning the populated side of a PCB is very unreliable as this technique does not detect elements on vertical surfaces, in shadowed areas or located underneath components.
- Connector plugs, cables by handheld XRF – screening plastics for Pb, Cd, Br and Cr (but the accuracy is not sufficient if the substance present is close to the concentration limit).
- Components on PCBs. SEM/EDX can be used to analyse solder alloy, termination coatings if these have not been wetted by solder (e.g. this is not possible after wave soldering surface mount components), upper surfaces of components such as plastic encapsulations, plastic bodied connector blocks etc.

For any other situation, the equipment will need to be dismantled prior to analysis. In most cases, components present on PCBs will need to be removed before analysis and some components broken down further for analysis. In some cases, one material can be dissolved selectively from a component. The banned substance content of this material can be calculated from:

$$\text{Concentration} = \frac{\text{Mass of substance in extract solution}}{\text{Total mass of material dissolved determined from the weight loss of the component}}$$

**Table 6. Sample preparation techniques**

| Analytical method   | Material analysed                       | Within single materials (metals, plastics, ceramics)  | Within whole components (capacitors, resistors, transistors, etc.)  |
|---|---|---|---|
| <b>Atomic absorption spectroscopy (AAS)</b>   | Pb, Cd, (Hg if cold vapour method used) | Dissolve material then analyse solution. For some components, it is possible to selectively dissolve one surface material only (e.g. lead in tin coatings by BS 6534), then analyse this solution. Concentration is determined either from weight loss of component and amount of element present in extract solution or by analysis of all elements that were extracted. | Dissolve the whole component. This is not always possible in one step as plastics and metals may not both dissolve in a single solvent and so the complete analysis may require several process steps for complete dissolution. |
| <b>Inductively coupled plasma spectroscopy (ICP)</b>                                  | Pb, Cd                                  | Same as for AAS.  | Same as for AAS.  |
| <b>Ultraviolet/visible spectroscopy</b>   | Hexavalent chromium (CrVI)              | Selective extraction of surface coating (e.g. with carbonate buffer). Also need to measure the coating weight to determine concentration in coating. Usually this involves dissolving passivation coating then analysis of the solution for total chromium. CrVI in paint coatings by BS 3900-B10 / ISO 3856-5.   | Dissolve the whole component. Same problems present as with AAS but also need to prevent change in oxidation state of Cr so can be difficult.   |
| <b>Energy dispersive X-ray analysis with a scanning electron microscope (SEM/EDX)</b> | Pb, Cd, Hg compounds, Br, Cr            | Analysis of exposed surfaces. Can examine very small areas but these should ideally be flat. Materials within components can be analysed by first exposing these by cross-sectioning.   | Best done by grinding to power and homogenising. Analyse area of powder as a flat layer.  |
| <b>Spark emission and DC arc emission spectroscopy</b>                                | Pb, Cd, Hg                              | For surfaces of metals.   | Not possible.   |
| <b>Glow discharge optical emission spectroscopy</b>                                   | Pb, Cd, Hg, Br, Cr                      | For analysis of individual layers of multiple layer coatings.   | Not possible.   |
| <b>Polarography</b>   | Pb, Cd, Hg and CrVI                     | Analysis of solutions (see AAS).  | Analysis of solutions (see AAS).  |
| <b>High performance liquid chromatography (HPLC)</b>                                  | Brominated flame retardants             | Method uses liquids so plastic needs to be dissolved or components of plastics extracted with a suitable solvent.   | Not applicable. Plastic analysed separately to rest of component.   |
| <b>Ion chromatography (IC)</b>  | Brominated flame retardants             | Method uses liquids so plastic needs to be dissolved or components of plastics extracted with a suitable solvent.   | Not applicable. Plastic analysed separately to rest of component.   |
| <b>Gas chromatography mass spectroscopy (GCMS)</b>                                    | Brominated flame retardants             | Uses either solvent extract from plastic or small pieces of plastic can be analysed directly with appropriate technique.  | Not applicable. Plastic analysed separately to rest of component.   |

| Analytical method   | Material analysed           | Within single materials (metals, plastics, ceramics)  | Within whole components (capacitors, resistors, transistors, etc.)  |
|---|-----------------------------|---|---|
| <b>Energy dispersive X-ray fluorescence analysis (ED-XRFA)</b><br><br><b>Handheld and desktop methods</b> | Pb, Cd, Hg, Br, Cr          | Handheld instruments can be used for screening materials and components (lead-free or tin/lead) but users must appreciate the severe limitations and periodically compare results with those from an accurate analysis method. Not able to accurately measure 0.1% lead in component termination coating. Desktop instruments can be used with some components to analyse materials present at the surface or within components after cross-sectioning but accuracy can be compromised if the instrument is not calibrated correctly. | <p>Handheld instruments can be used for screening components (lead-free or tin/lead) but cannot measure the concentration in a whole component as only the exposed upper surfaces are analysed.</p> <p>Grind whole components to homogeneous powder for analysis with desktop instrument. Calibration using representative standards needed for accurate results.</p> |
| <b>Wavelength Dispersive X-ray fluorescence analysis (WD-XRF)</b>   | Pb, Cd, Hg, Br, Cr          | Suitable only for homogeneous materials. Calibration is important.  | Grind whole components to homogeneous powder for analysis. Calibration is important   |
| <b>Fourier transform infra-red (FTIR) spectroscopy</b>  | Brominated flame retardants | Suitable only for plastics with flat surface for reflectance technique or as solution for transmission technique.   | Not suitable.   |
| <b>"Litmus" test</b>  | Lead present at surface     | Will only detect lead present at the surface.   | Will determine if lead solder or tin/lead plating is used.  |

## 6.4 Analysis procedures for electrical equipment

Detailed instructions for the analysis of homogeneous materials within electrical equipment is beyond the scope of this investigation but clearly some guidelines will be useful. These methods can not be finalised until the concentration limits values and definitions are finalised by the TAC but could include the following examples.

**Table 7. Sample preparation techniques**

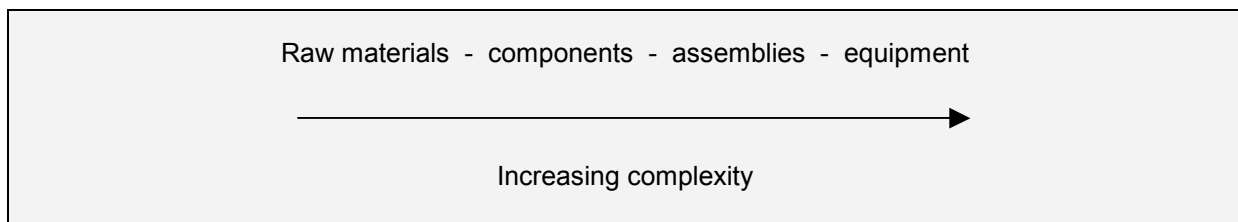
| Component or material   | Procedure  |
|---|--|
| Plastic mouldings and other parts   | <p>XRFA for Pb, Cd, Br, Cr (note limitations).</p> <p>SEM-EDX for Pb, Cr, Br (note limitations).</p> <p>Acid digestion followed by AAS for Cd and Pb. Handheld XRF for bromine. If bromine is detected, then identification and quantification of PBB and PBDE will be required.</p>   |
| Wire and cable insulation   | <p>Remove insulation, dissolve in acid, analyse by ICP for Pb and Cd.</p> <p>Handheld ED-XRFA suitable only to identify cable with high levels of Pb or Cd or that they are absent.</p> <p>Desktop ED-XRFA. For routine checking but should be calibrated using insulation with known Pb and Cd content (e.g. determined using ICP).</p>   |
| Solder and other metals as single bulk materials                            | Any suitable method can be used (AAS, ICP-AES, ED-XRFA, SEM/EDX, spark emission and DC arc emission spectroscopy).   |
| Solder on populated PCB   | <p>SEM/EDX of solder surface will determine whether lead is present at &lt;0.1%.</p> <p>ED-XRFA is suitable if the lead content is high or very low and only if there is a large enough flat surface for analysis. Results can be unreliable for the reasons discussed in this report. The size of errors depends on many factors and can be larger than those quoted in recent publications.</p>  |
| Tin alloy coating on component terminations, determination of lead content. | <p>SEM/EDX.</p> <p>Desktop ED-XRFA if flat surface area is large enough.</p> <p>Method defined in BS6534:1984.</p>   |
| Electric contact surface layers   | <p>Dismantle component to expose contact surface then:</p> <ul style="list-style-type: none"> <li>- Desktop EDXRFA of contact surface.</li> <li>- Cross-section through substrate/braze and contact material then analyse all three separately using SEM/EDX. Note detection limit for cadmium by EDX is not low enough so if this is suspected to be present at &lt; 0.1% but &gt;0.01%. (NB. Cadmium deliberately added will be &gt;0.1%).</li> <li>- Dissolve the parts, analyse by AAS or ICP-AES for cadmium then estimate its concentration from the proportions of each material. This method will be difficult to carry out accurately.</li> </ul> |
| Passivation coating on galvanised steel screws                              | <p>SEM/EDX or ED-XRFA analysis of surface will identify Cr but not oxidation state.</p> <p>Extraction with carbonate buffer solution then use a colorimetric methods to confirm CrVI. Need to know coating thickness (as mass/cm<sup>2</sup>) to calculate Cr VI concentration.</p>  |
| Parts made with alloys containing lead for free-machining                   | RoHS Directive Annex, item 6 permits limited concentrations of lead in steel, aluminium and copper alloys. To analyse these by AAS or ICP, these need to be separated and any coatings removed before analysis. Alternatively, the lead content could be determined by quantitative SEM/EDX of a cross-section.  |

## 6.5 Multiphase component analysis issues

It is possible to analyse individual components either as a whole or by analysis of each material separately after either physical or chemical separation using the techniques listed in Table 7. It is also theoretically possible to analyse most complex components and assemblies but interpretation of the results becomes more difficult as the complexity increases.

### 6.5.1 Complexity of items versus ease of analysis

Analysis of a single homogeneous material is easier to carry out than analysis of a component that may contain many different materials. Hence, it is easiest for the original component or material manufacturer to analyse their incoming raw materials to ensure that none of the banned substances are used so that their products can be used in RoHS compliant equipment. Components contain relatively few individual materials in comparison with assemblies. Further along the supply chain, products become more complex with many more different homogeneous materials present so that analysis is more difficult.



Analysis is easiest and most accurate for materials at higher concentrations. Therefore it is easier to analyse the incoming raw materials than a finished piece of equipment. The following table illustrates this by showing the concentration of the lead present in the coating of one component calculated as a concentration in the component, as a concentration of a PCB and as a concentration in the final product assuming that there is no other lead present.

**Table 8. Lead concentration originating from one QFP device determined as a percentage of different parts of a hypothetical desktop personal computer**

| Lead from Philips QFP device measured as the concentration in: | Typical concentration of lead |
|--|-------------------------------|
| SnPb termination coating                                       | 15%                           |
| Philips QFP device (1.12 grams)                                | 0.09%                         |
| In typical PC motherboard (1kg)                                | 0.00015%                      |
| In computer with CRT monitor (27 kg)                           | 0.000006%                     |

Ideally, producers of assemblies and equipment should be able to rely on their suppliers for an assurance that incoming components and assemblies (such as PCBs and power supplies) are suitable for use in “RoHS compliant” equipment. They will also rely on materials declarations from component and materials suppliers. However, these may not always be available or trustworthy and so producers would want to analyse selected parts within finished products, assemblies or certain

individual components. Components could be analysed individually if these are available, otherwise after removal from assemblies and in some cases *in-situ* within assemblies (by SEM-EDX or some desktop ED-XRFA). The aim of the analytical procedure would be to determine if any of the homogeneous materials that make up the product contain any of the six banned substances at concentrations higher than the upper limits that will be defined by the TAC. A simple screening test using a handheld ED-XRFA will be suitable in many cases but this does have limitations (see Annex 1).

There are several complicating factors that could occur during the chemical analysis of electrical equipment. Analysis of a large sample of a homogeneous material is relatively straightforward. Producers need to know whether this contains one of the banned substances at a concentration above the specified limit, which is likely to be 0.1 % (except for cadmium at 0.01%). This concentration can be measured easily. It is more difficult to measure very low concentrations accurately, which could result from analysis of an assembly; it will be easier to measure 0.1% of lead than 0.0001% of lead. The following example and those given in section 6.5.2 – 6.5.4 assume that the concentration limits will apply to materials and not to whole components as defined in Section 4.

**Example 3:** A typical mobile phone constructed using tin/lead solder contains about 0.3 % by weight of lead, most of which is in the form of solder <sup>10</sup>.

If the entire phone is dissolved in acid and the solution analysed, this will show that the phone contains 0.3% lead. Assuming the concentration limit for lead in homogeneous materials to be 0.1 weight %, the phone does not comply with the requirements of the RoHS Directive. This analysis however does not show where the lead is present within the phone.

If the same phone is produced using lead-free solder, but all of the components on the PCB have tin/lead termination coatings, then the total lead content of the phone will be approximately 0.02 weight % lead. (This estimate is based on the relative quantities of lead in a typical solder joint that has come from the termination coating and from the solder <sup>15</sup>).

Analysis would give a figure of 0.02% lead. This is much less than the lead concentration limit in a homogeneous material but there is no way of knowing from this figure how much lead is present in the termination coatings or in the components. Tin/lead coatings on components will typically contain between 5 and 30% lead and so are not likely to be suitable for RoHS compliant products, however analysis of the whole phone has not shown this.

It is impossible to determine the lead content of the component termination coatings from analysis of the whole phone. Taking this example further, analysis of the whole phone could give a lead content of 0.00005% if either:

- there is only one MLCC with a termination coating of tin with 20% lead (a non-compliant component), or
- there are 100 components with a lead concentration of 0.05% in their termination coatings.

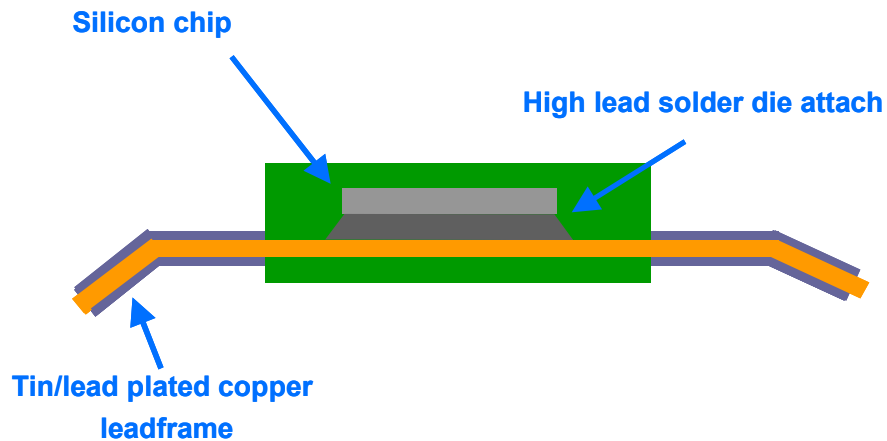
**Therefore, it is not possible to interpret from analysis of the whole phone if it is RoHS compliant.**

Measurement of a concentration as low as 0.00005% is very difficult and the accuracy is likely to be low. Mobile phones are very small and light-weight so these difficulties will be worse where there are many small components used within a large and heavy piece of electrical equipment. The only way to obtain a definitive result is to analyse individual components using an appropriate technique.

## 6.5.2 Substances present in both banned and exempt forms

The RoHS Directive provides exemptions for use of the banned substances in certain cases. In some cases this means that exempt and banned forms of the same substance may be present in the same component. This needs to be taken into account when considering how to analyse for the banned substances. The following are examples of device technologies where this issue arises.

### Semiconductors



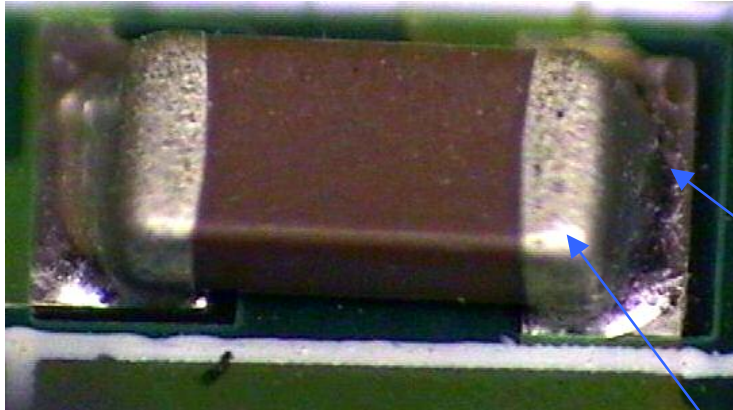
**Figure 2. Diagram of a power transistor showing both banned and exempt forms of lead**

High melting point solders containing more than 85% lead are exempt. These are typically used for die attachment. Solderable termination coatings, on the other hand, often consist of tin with between 5 and 40 % lead and are not exempt. Hence, analysis of the whole component is meaningless and the RoHS compliance status can be determined only by analysis of the termination coating.

**Example 4:** ST Microelectronics make a Schottky diode containing a total of 1.16% lead by weight of the whole device. The lead in the die attach comprises 1.07% and lead from the termination coating 0.09%.

Multi-layer ceramic capacitor (MLCC)

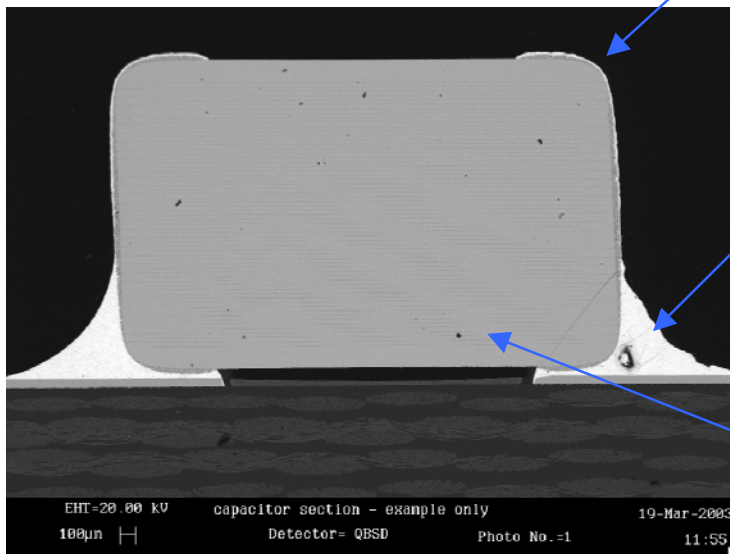
The MLCC is another common example of where lead is used in an exempt form and as a material that is banned. Item 7 (4) of the Annex to the RoHS Directive provides an exemption for lead (but not cadmium) in ceramics used in electrical components.



**Figure 3. Optical image of MLCC**

Solderable termination coating – tin/lead

Soldered to PCB



Dielectric ceramic layers. Lead or cadmium are used as binders

**Figure 4. Scanning electron image of cross-sectioned MLCC**

Again, analysis of the total lead in this type of component (e.g. by dissolving in acid then use of ICP) would not provide an indication of whether it is suitable for RoHS compliant products. Compliance status can be determined only by separate analysis of the individual homogeneous materials on the surface of and within components. ED-XRFA and SEM/EDX are both surface analysis techniques and so will be suitable for the analysis of lead present only at the surface but not if this is present

within the component. However, materials present internally can be analysed by first exposing these by preparation of a cross-section.

### 6.5.3 Low level impurities

Analysis of whole components could lead to incorrect interpretation of analysis results because of trace impurities that are present in most materials. For example, commercial tin that is used to make lead-free solders contains typically 0.03% of lead.

Taking an example of a hypothetical component, which contains two homogeneous materials both with a lead concentration below the RoHS Directive limit of 0.1%. Theoretical concentrations in a component are shown in Table 9.

**Table 9. Example of possible lead distribution in a hypothetical component, weight 10 g**

| Percentage of total mass                         | Lead concentration (%) homogeneous material | Mass of lead in each homogeneous material | Total mass of lead per component | Actual % within component |
|--|---|---|----------------------------------|---------------------------|
| All of component except termination coating, 95% | 0.01%                                       | 0.00095 g                                 | 0.00135 g                        | 0.0135%                   |
| Termination coating only, 5%                     | 0.08%                                       | 0.0004 g                                  |                                  |                           |

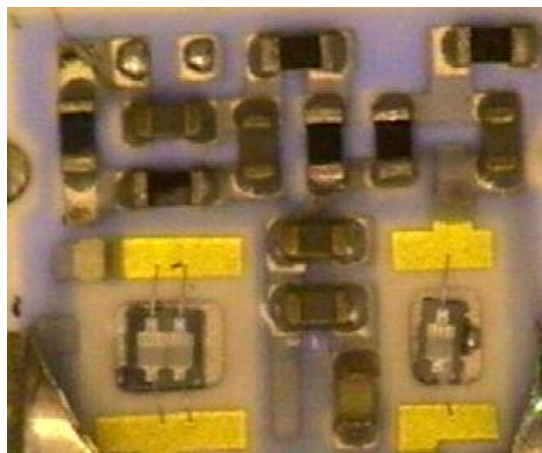
In this hypothetical example, if the component were analysed as a whole, the overall lead concentration would be found to be 0.0135%.

If it is known that the termination coating accounts for 5% of the total mass of the component then if all of the lead was incorrectly assumed to be present in this one material, the lead concentration would be calculated at 0.27% which is above the RoHS concentration limit. If the concentration within the other 95% of the component was known, it might be possible to estimate the true lead content in each of the two materials but in reality, components have many more separate materials and so this will not be possible. Analysis of each individual homogeneous material including the termination coating itself however, will give a clear definitive answer and would show that the component used for this example is RoHS compliant whereas analysis of the whole component could have resulted in it being rejected.

### 6.5.4 Multilayer coatings

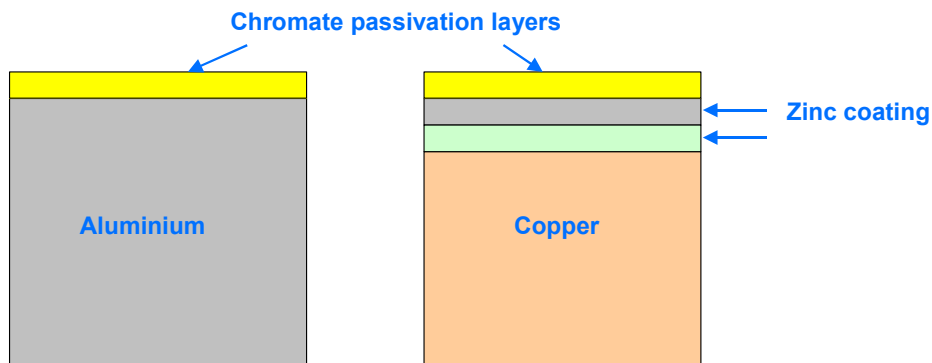
Components often have coatings which consist of several layers. For example, copper IC leadframes are first electroplated with nickel and then over-plated with tin. Some coatings are treated with a hexavalent passivation treatment solution which deposits a thin coating containing CrVI on the surface. The analysis procedure will depend on whether the maximum concentration limits are in components, homogeneous materials or in this case the multiple layer coating. Analysis of individual coatings can be carried out but this can be more difficult if the layer thickness is much less than 2µm, which is not uncommon. Some examples include:

- Integrated circuits contain a semiconductor (usually silicon) with several layers deposited on one surface. Analysis of these separately can be carried out only with difficulty due to their very small size. However, none of the six banned substances are likely to be present
- Thick film circuits contain many layers containing a variety of materials. These are produced in a way that results in these layers being strongly bonded to each other so that physical separation will be difficult. Both lead and cadmium are used in this type of circuit and so analysis is likely to be necessary. Analysis of the unfired pastes or inks would be the easiest and most accurate but these may not be available to equipment producers and would not be available to enforcement authorities.



**Figure 5. Part of a hybrid circuit which is produced using thick film pastes as well as some discrete components**

- Electroplated coatings often have more than a single metal layer. Copper is usually electroplated first with a barrier layer of nickel and then with a tin coating. These can be analysed separately, for example by SEM/EDX but this is more difficult if any individual layer is very thin ( $< 2\mu\text{m}$ ). Selective dissolution such as the method described in BS 6534 can be used but the choice of method must be chosen specifically for the type of coating and the composition of the material on which it is deposited.
- Hexavalent chromium passivation is used on various metals including aluminium as castings or machined parts and on thin electroplated coatings such as zinc. The passivation process is a chemical reaction that occurs on the metal surface between the metal and chromate ions in the passivation solution. The product is a thin layer of a mixture of materials which includes some hexavalent chromium as illustrated below.



**Figure 6. Schematic of layers on aluminium casting (left) and zinc electroplated copper (right) (not to scale)**

In the this instance, the chromate passivation coating on the aluminium parts is clearly a different composition to the bulk aluminium and can be removed by physical methods (e.g. abrasion with emery paper). Chromate coatings are usually very thin and, as a result, the aluminium and chromate coating cannot be separated cleanly from each other by physical methods. It is possible, however, to analyse the chromate coating by selectively dissolving this coating and analysing the extract solution. It is also possible to chemically analyse each of the layers on the chromate passivated zinc/nickel electroplated copper if the correct procedure were used but it would be very difficult to separate each layer by physical means. The aim of the RoHS Directive is to restrict the use of harmful substances including hexavalent chromium and this will be achieved if the passivation coating is defined as one homogeneous material. If the combination of layers including the passivation coating is assumed to be one homogeneous material, then it would be possible for manufacturers to use hexavalent chromium passivation treatments.

**Example 5:** One producer has reported that analysis of the chromate passivated zinc coating gives a CrVI concentration of <0.1%, whereas the CrVI content of the passivation coating itself would be >0.1%

## 6.6 Requirements for new Standards

Producers will be able to demonstrate that materials, components, assemblies and equipment comply with the requirements of the RoHS Directive by using the analytical procedures described in this report.

Procedures exist for all of the banned substances present in bulk materials, but the analysis of individual phases within complex components and equipment requires a good knowledge of the construction of electronic components as well as a high level of analytical expertise. ERA and other organisations around the world have many years' experience carrying out failure analysis of electrical equipment and some of the techniques used to analyse components for defects are suitable for demonstration of RoHS compliance. Analysis Standards exist for a small number of materials as listed in Table 4.

Analytical results are usually more acceptable and trusted where the analysis is carried out according to a standard procedure although, in reality, accurate results can usually also be obtained by non-standard but suitable methods. The existence of Standards would enable many more organizations to carry out component analysis accurately and it should be a medium term goal to develop standards for the analysis of at least some of the more difficult components. Suggested examples for new Standards include:

- A Standard to cover demonstration and testing of RoHS compliance (there could also be subsidiary Standards to cover specific aspects). Guidance on sampling methods and the definition of concentration limits and homogeneous materials (with examples) would be useful. This approach is already used for New Approach Directives although RoHS is not a New Approach Directive.
- Conformity assessment procedure (as described in the “Guide to the Implementation of Directives Based on the New Approach and Global Approach”<sup>16</sup>). This should give clear guidance, especially to small and medium size producers, of what enforcement authorities will expect if they detect a non compliant product.
- A Standard covering the use of desk-top ED-XRFA for analysis of electronic components and materials, individually and within electrical equipment. This would avoid errors that could result from not appreciating the limitations of this technique. There are CEN guidelines for wavelength dispersive X-ray fluorescence analysis (CR 10299:1998)<sup>17</sup>. This is not written specifically for any particular material but is useful for the prevention of analytical errors that can occur with both ED-XRFA and WD-XRFA.
- Analytical procedures for identification and quantitative analysis of PBB and PBDE flame retardants in plastics.
- A Standard covering the use of SEM/EDX for analysis of electronic components and materials, individually and within electrical equipment. This would avoid errors that could result from not appreciating the limitations of this technique.
- Analysis procedure for passivation coatings.

At each stage of the supply chain from component manufacturer to an EU importer, an ability to purchase materials, components and even finished products to meet a composition specification will simplify purchasing requirements. Standards already exist for a wide variety of materials, especially metals which specify the maximum and minimum concentrations of all constituents.

Some existing Standards could be modified, for example:

- Product Standards that specify tin/lead solder must be used.
- Standards for free-machining alloys exist which specify lead content but the upper limits are higher than those permitted in item 6 of the Annex to the RoHS Directive.

It should be possible for new Standards for RoHS compliant materials, components and assemblies to be developed although these are unlikely to be available by the 1<sup>st</sup> July 2006.

## 6.7 Conclusions regarding Standards for compliance testing

1. The exact meaning of the maximum concentration values is currently not clear and different interpretations are being assumed. This is in part due to some of the terms used in the proposed definition having been used in other Directives, some of which have different intended meanings (in particular “unit”). Difficulties with precise translation may also be a complicating factor.
2. The maximum concentration values must be clear, in particular it should be understood whether these values apply to “materials” such as metals, plastics and ceramics or to individual components. There will be some “grey areas” most of which can be clarified by use of a list of indicative examples.
3. The definition that is chosen will have a direct impact on the quantities of the banned substances manufacturers will be permitted to use.
4. A small number of Standards exist that can be used for analysis of components and materials from electrical equipment but are not sufficient for equipment manufacturers or enforcement authorities to use to assess materials, components or products.
5. Analytical methods are available with sufficient accuracy for almost all materials and components used in electrical products. In many cases, there are several techniques that could be used. Some items will be difficult to analyse although this will depend in some cases on the precise interpretation of the maximum concentration values.
6. ED-XRFA has been widely promoted as a simple low cost analysis technique but research has shown that accuracy can be poor if the limitations are not understood and taken into account. It is possible for these instruments to give the wrong assessment of products; that is passing a material containing >0.1% lead or failing a material containing <0.1% lead.
7. Some components present difficulties for analysis, for example if one contains lead in an exempt form and in a banned form as discussed in section 6.5.2.
8. New Standards would be helpful to the electronics industry as these will give guidance to how equipment should be tested and ensure that the same results are obtained in all Member States. However it is unlikely that these will be available in time for the 1<sup>st</sup> July 2006 deadline.
9. It is reasonable that component and material manufacturers and equipment producers are permitted to use any suitable analytical technique but they should be aware of the limitations.

## **7. Standards on Reporting Formats**

### **7.1 Current status**

At present there is no standard for materials declarations or reporting RoHS status. This has led to a situation in which manufacturers and distributors are receiving an increasing number of requests for materials declarations. These requests take a very wide variety of forms and the information requirements are very varied and are required in many different formats. This is creating a great deal of work for manufacturers and distributors whereas a simple system that uses a single format and requires the minimum amount of information required to show RoHS compliance would be much easier and lower cost to manage.

Producers of equipment need assurances from their suppliers that products do not contain banned substances. A simple phrase stating that a component or material complies with the RoHS Directive substance bans would in many cases be sufficient and some manufacturers are already using wording to this effect. The legal status of these documents is at present unclear and individual producers may need to consider changing the terms and conditions of their purchase contracts to protect themselves from errors on the part of their suppliers.

### **7.2 Existing declaration formats and databases**

Several organisations are working on material declaration formats that component and equipment manufacturers can use to provide composition information for their customers. These were not originally developed for the RoHS Directive but may be a useful starting point for a simple information system. Web based systems for easier data handling are also being developed and research into simple low cost database software is also being carried out. Some manufacturers will already have their own in-house systems for handling incoming data although these were not designed specifically for RoHS.

#### **7.2.1 Materials declaration formats**

An internet search was carried out to identify suitable ways of providing compliance information including materials declaration formats. Information was also provided by various individuals and organisations including ESIA and Tin Technology.

The main purpose of all of the material declaration procedures discussed here is to obtain data from the supply chain which is required for the end of life of equipment (WEEE Directive) or for vehicles (ELV Directive). The data is also used for improving product design and to meet legal and regulatory requirements. None are being developed specifically for the RoHS Directive but some may be suitable for this purpose.

The amount of data required for each format varies but all require information on more materials than just those banned by RoHS. These range from 29 materials in a EICTA/EIA/JGPSSI procedure

(others are optional) to at least 142 in the DKE (Deutsche Kommission Elektrotechnik) draft standard on which the ZVEI<sup>①</sup> system is based.

### **EIA (USA), EICTA (Europe), JGPSSI(Japan) – Material Composition Declaration Guide<sup>18</sup>**

EICTA<sup>②</sup>, EIA<sup>③</sup> and JGPSSI<sup>④</sup> have been collaborating to develop a materials composition declaration procedure and recently published a draft guide to this process. This initiative has wide support from industry and other organisations.

The aims of their approach are to satisfy legal requirements, drive product design improvements and respond to enquiries from customers, recyclers, etc. The procedure has not been developed for the RoHS Directive but the published draft guide indicates that it could be used for identifying whether components are RoHS compliant.

This proposed approach uses two lists of substances totalling 29 substances for compulsory inclusion (inclusion of others is optional). This includes

- the six banned by RoHS,
- other hazardous materials,
- substances of economic value such as gold and
- materials that have a negative impact on end of life management.

There is a list of data requirements, many of which are optional but mandatory requirements include the mass of each part or sub-part and of each substance.

Reporting of the form and location of the hazardous substances listed in Annex A of the procedure (which includes all six RoHS banned substances) is mandatory. Figure 7 shows the proposed draft format for declarations. Key points to note are:

- the procedure only requires reporting where a hazardous substance exists above a certain concentration limit or if intentionally added,
- the concentration limits are the same as the proposed limits for RoHS (0.1%) for substances except cadmium where it is 0.0075% (against 0.01% in RoHS),

Consequently, compliance with RoHS would be indicated by the six banned substances not being included.

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① German Electrical and Electronic Manufacturer's Association

② European Information, Communications and Consumer Electronics Technology Industry Association

③ Electronics Industry Alliance

④ Japanese Green Procurement Survey Standardisation Group

| SAMPLE MATERIAL DECLARATION DATA SHEET |                             |                                |
|--|-----------------------------|--------------------------------|
| Date:                                  | 03/09/11                    |                                |
| Company Name:                          | Any Company                 |                                |
| Product Name:                          | Integrated Circuit          |                                |
| Product Number:                        | 001                         |                                |
| Product Total Mass (g):                | 0.717 g                     |                                |
| Material/Substance Name                | Material/Substance Mass (g) | Material/Substance Information |
| Antimony                               | 0.01166                     |                                |
| Silver                                 | 0.05636                     |                                |
| Copper                                 | 0.21834                     |                                |
| Lead                                   | 0.001715                    | Solder Plating                 |
| Gold                                   | 0.0047                      |                                |

**Figure 7. Proposed material declaration data sheet for the EICTA/EIA/JGPSSI procedure**

The final format has not yet been agreed. There are discussions about which materials should be included in the two compulsory lists and one suggestion has been made that the absence of some materials should be positively stated. If this change were made, this procedure would provide the information producers need to determine whether a component contains a substance banned by the RoHS Directive.

#### **International Material Data System (IMDS) <sup>19</sup>**

This is a system set up by automobile manufacturers to build up a database of the full composition of all components and materials that are used in automobiles, principally in response to the End of Life Vehicle (ELV) Directive but also to aid Design for the Environment. Data requirements are very detailed and the full composition of all parts including weight and percentage is required.

IMDS has been running for several years but not all suppliers have been willing to provide individual material data of components. One reason given by ZVEI is that the time and personnel resources required to provide this detail is “huge” and the “electronics industry sees no possibility to enter the individual materials data of components, for our automotive customers and their suppliers, into the IMDS system”<sup>20</sup>. There is also the view that this information is proprietary.

The IMDS system requires information on 90 materials or classes of materials as a minimum. All of the 90 are hazardous and so should be included in materials safety data sheets (MSDS) which are required for substances and materials although not usually for parts or components. The IMDS scheme does permit confidential information to be protected by limiting accessibility.

IMDS is complex, requires training and the data is incomplete as a significant number of component manufacturers, mainly in S.E. Asia, have been unwilling to provide data on their products. This type

of approach would be too complex and expensive for small and medium size equipment manufacturers to use.

### **ZVEI draft guidelines on materials declarations** <sup>21</sup>

ZVEI has published a draft “Information on Substances and Materials in Products” which comprises guidelines and a draft form for materials declarations. The format is similar to that proposed by EICTA/EIA/JGPSSI and requires details from a list of 142 that should be included. The list of substances is the DKE list of declarable substances.

The average mass % in each material is required as well as the component mass. The ZVEI system requires the compulsory inclusion of fewer substances than the EICTA/EIA/JGPSSI system but any of the 142 can be included.

This system is already used by some component manufacturers including ST Microelectronics who publish this information on their website. This approach is not designed for the RoHS Directive and equipment producers may not be able to ascertain whether a component complies with RoHS from this data (this will depend on the definition of the maximum concentration levels to be agreed by the TAC).

### **DKE – Record of Substances in Products** <sup>22</sup>

DKE has produced a draft standard for a record of substances in products. Data is for specific parts or product families.

Substances that are intentionally utilised including consumables are included but materials used in the manufacturing process but not present in the product are excluded. The concentration of substances and weight limit values are required for any of a list of 142 materials and types of material present at a concentration above 0.1% although other substances may be included.

## **7.2.2 Declaration of conformance formats**

### **ISO/IEC DIS 17050 - 2– Suppliers Declaration of Conformity (SDoC)** <sup>23</sup>

The IEC is currently developing a standard procedure for supplier’s “Declaration of Conformity”. Suppliers will be able to use these to demonstrate that their products meet specified legislation or standards including the RoHS Directive. A standard form will be used which will contain only the essential information that is required. In the case of RoHS, only the six banned substances would need to be considered and the declaration would state simply that the product conforms to the requirements of the RoHS Directive. In addition to the standard form, which can be provided in any suitable medium (e.g. hardcopy or electronic), suppliers will be required to maintain supporting documentation so that the declarations can be traceable. Supporting documentation would include test methods, test results and laboratory accreditation status.

### 7.2.3 Systems for handling composition data

There are several web based software tools being developed to simplify materials declaration. Some of the larger electrical equipment manufacturers have their own systems that they require their suppliers to use.

#### **RosettaNet – Material Composition Partner Interface Process** <sup>24</sup>

RosettaNet is developing an Internet based system for the exchange of material composition data between suppliers and customers. RosettaNet is a consortium of over 400 companies within the electronics sector which has developed a variety of business information systems based on the Internet. RosettaNet claim that all of their systems are public, free and no licence is required.

#### **Greenpack – Materials Declaration Tool** <sup>25</sup>

Green Electronics Packaging and Environmental data flow Management (Greenpack) is a research initiative that is developing web based software that aims to simplify materials declaration.

#### **REMAT** <sup>26</sup>

Software is being developed by the University of Surrey, Polymer Research Centre which is being designed to handle the large amounts of data that will be needed for compliance with the WEEE and ELV Directives and may also be useful for the collection and reporting of data for the RoHS Directive.

REMAT is a Resource Flow Management Tool that can be used by large and small manufacturers as well as enforcement authorities. It should be compatible with other software currently in use and be able to accept data in any format. This is in the early stages of development and is not yet commercially available. If this can provide what the developers are hoping for and it is available at a reasonable cost, it would be a useful tool for management of large amounts of data.

#### **Material Declaration Wizard** <sup>27</sup>

Software is already available for handling composition data.

### 7.2.4 Individual company approaches

Ericsson is an equipment manufacturer which has an Excel form on their website which they require their suppliers to complete. This can then be printed or emailed. Ericsson require information on a very long list of substances present above certain threshold concentrations (some as low as 1 ppm) and so could involve their suppliers in significantly costly analysis. This approach requires the concentrations of substances in components, not within each homogeneous material and so may not be suitable for showing whether a component is suitable for RoHS compliant products.

An increasing number of component manufacturers including Philips, ST Microelectronics and Agere Systems publish materials declarations on their websites. These could be incompatible with other

systems being in a different format or having less or different information although this should change if one universally agreed format was agreed.

Some component manufacturers regard the composition of their products as proprietary and would not want to divulge this information. This is why some component manufacturers have refused to provide data for the IMDS database.

The only information that equipment producers need for RoHS compliance is an assurance from their supplier that the products comply with the RoHS Directive. Producers do not need to know the concentrations or quantities of the banned substances in these products and some component producers are adopting the approach that they will provide a statement that the product is suitable for use in RoHS compliant equipment by declaring that the six banned substances are absent. As producers require only confirmation of the absence of the six banned substances (except where there are exemptions), this would appear to be sufficient.

There are plans in China to develop a standard RoHS compliance statement that all Chinese manufacturers would use.

### **7.3 Symbols and markings to show RoHS compliance**

Discussions are taking place within the electronics industry about the use of symbols or identifying marks on individual components, assemblies such as PCBs and on equipment to show, as a minimum that the item complies with the RoHS Directive. Markings have been proposed by JEDEC, NEMI, JEITA, Soldertec and others. Additional information could be included to show the type of solder or termination coating used which would aid repair <sup>28</sup>.

At present, component manufacturers have a variety of policies for indicating RoHS compliance or lead-free:

- No markings or change of part number are used to identify RoHS compliant components. This is fairly common.
- A change of part number is used only during the transition period while both lead and lead-free components are available.
- Various letter codes are used to denote “lead-free”. Also, the definition of lead-free does vary.
- A small number of producers include a code letter that identifies and differentiates “lead-free”, “RoHS compliant” and containing lead (e.g. Philips Semiconductors).
- A small number of producers use a code letter that identifies the termination coating used (e.g. Ricoh).

- Symbols applied to the surface of device plastic encapsulation (e.g. Cypress Semiconductor uses a white square for lead-free).

For showing compliance to the RoHS Directive, these are indicative only as there is no accepted standard marking and so these approaches alone should not be relied on as the only means of ensuring that products are RoHS compliant.

A standard RoHS compliant symbol could be used as a materials declaration so that other formats such as materials declaration certificates, would not be needed. Equipment producers would still need to assess whether their suppliers declarations are reliable though, and carry out periodic checks.

A survey carried out by Soldertec <sup>12</sup> showed that equipment producers would like to see marks on components, assemblies, PCBs etc. but many component manufacturers are not in favour of applying markings to their products.

#### **7.4 Centralised analysis databases**

Databases such as RosettaNet and Greenpack are discussed in Section 7.2.3. These are designed as repositories for data supplied by component manufacturers. For RoHS, equipment producers may need to check by analysis that the components they obtain do not contain any of the banned substances. If all producers analyse a random selection of components, there is likely to be some duplication and, while a large number would be analysed, the results would not be widely available.

Publishing the RoHS compliant status of components at one web based location should

- reduce the amount of work required by component manufacturers and equipment producers,
- avoid the need for producers to contact all of their component suppliers,
- ensure that component manufacturers can avoid having to provide this information in many different formats,
- provide a highly visible location where producers can be made aware of components that are claimed to be suitable for RoHS compliant products but are found not to be.

Electrical equipment manufacturers could collaborate either as small groups or through trade associations to pool resources and carry out random analysis of the more widely used components. This would reduce an individual producer's costs and might ensure that a larger range of components are checked.

Key to any centralised system is accurate analysis data. Where this type of information is shared, it is advisable that the analysis is carried out by an accredited laboratory. This is all the more important since any errors in the data will be less likely to be found in a system where duplication of analysis is minimised.

## 7.5 Conclusions regarding standards for reporting formats

1. An increasing number of electrical equipment manufacturers are asking their suppliers for details of a wide range of hazardous materials that might be either used in the production process or are components of products. This information is used to reduce the impact of their products on the environment by limiting or restricting the use of all hazardous materials and is part of Design for the Environment procedures.
2. This data will be important in the proposed Energy using Products Directive (EuP) but is not required for the RoHS Directive. As far as RoHS is concerned, the only requirement on equipment producers is that materials, components, assemblies or equipment do not contain any of the six banned substances at concentrations above the specified limits in homogeneous materials. A simple statement to this effect may be sufficient as a materials declaration.
3. A variety of materials declarations are being proposed. Most have not been developed specifically for the RoHS Directive and so include data on many more materials than is required for compliance with RoHS. Some, such as the IMDS system, are very complex and not ideally suited for RoHS compliance. The final format of the EICTA/EIA/JGPSSI materials declaration has not been agreed but should be suitable for RoHS. The format from ZVEI may also be suitable but the one being developed by IEC (SDoC) is the simplest requiring only a statement that the product conforms to specified Directives or Standards.
4. A standardised system for marking materials, components, PCBs, assemblies and products is being discussed by several organisations. If these become accepted as Standards, they could be used to declare that the substances banned by RoHS are below the maximum concentration limits.
5. Various methods of disseminating RoHS data have been considered. Web-based databases are being developed for suppliers to publish their product's composition data. Some manufacturers already publish their data on company websites.
6. Many equipment manufacturers intend to carry out at least some analysis to check on their suppliers declarations. They could collaborate to share the cost of analysis but there are potential risks that need to be considered.

## 8. Information Exchange Networks

### 8.1 Overview

Each Member State will enforce its own national legislation and will appoint a body or organisation to carry out the necessary policing of the legislation but it is essential that declarations and evidence produced by producers for one Member State should be suitable for all other states.

Member State enforcement authorities responsible for existing New Approach Directives currently collaborate in a limited way. This is usually when a safety risk is identified. This is reported to the Commission who pass on the information to other Member States.

In practice, enforcement authorities within Member States communicate with their counterparts in other States only when a product that has been put onto the market has been found not to comply with a Directive's requirements. There is no routine collaboration to inform others of equipment that has been tested and found to be satisfactory which would avoid duplication.

### 8.2 Market surveillance and information exchange

The policing organisations within member states are obliged to collaborate with each other in accordance with Article 10 of the EC Treaty. Doing so is to their benefit because this will enable them to be more effective through:

- Avoiding duplication of effort.
- Building up a database of analysis data.
- Preventing the sale of non-RoHS compliant products in all Member States as soon as this is discovered in one Member State.

The approaches used within Member States to enforce existing New Approach Directives have been reviewed and recommendations made by a Council Resolution on 10<sup>th</sup> November 2003 <sup>29</sup>. The review found that the methods used and the resources available vary considerably. Proactive and reactive approaches of various types are used and some of the proactive approaches are judged to be the most effective. It has been recommended that a common set of minimum criteria for market surveillance should be used and the amount of effort for each product category should be defined. Where the market size is small, groups of Member States could pool their resources to ensure uniform and fair enforcement. One recommendation was that information exchange provisions by Member States needs to be included in individual Directives however this is not a requirement of the RoHS Directive.

With the exception of the new General Product Safety Directive (GPSD, 2001/95/EC), currently there are no information exchange networks within the EU for adopted New Approach Directives except under a few specific circumstances, all of which apply only when defects or safety risks are detected.

- RAPEX (Rapid Alert System for Non-Food Products<sup>30</sup>) is an early warning system used for consumer products that are covered by the General Product Safety Directive (92/59/EEC). The procedure is laid down in an Annex to this Directive and is used when a product which has a serious safety risk is identified. In such cases the enforcement authority in a Member State will report this to the Commission. The Commission verifies this information and circulates it to other Member States. The main aims are to prevent unnecessary duplication and ensure that Member States collaborate.

RAPEX is only when a problem is identified, there is no transfer of information on equipment that have been tested and found to be safe. This would be valuable information that would avoid the need to test the same product more than once. RAPEX was set up as a result of Council Decision 84/133/EEC on 2<sup>nd</sup> March 1984.

- Medical Devices Vigilance System<sup>31</sup>. This is a comprehensive system for monitoring medical devices and applies when an incident which might lead to harm to patients occurs. In this case, the manufacturer is responsible for informing the authorities which is followed by an assessment carried out by the surveillance authorities. They then inform the Commission and other Member States. In this case, the Commission takes on a supporting and co-ordinating role.
- Community injury data-collection and information system. This is a Community database for the collection of data on injuries. Member States collect this data by means of surveys and the information is made available to national governments, the Commission and other organisations. This database has the capability of being able to respond rapidly as information placed on the database could be available immediately.
- Safeguard clause procedure<sup>32</sup>. This is used where a risk is identified or non-compliant CE marked products are placed onto the market.

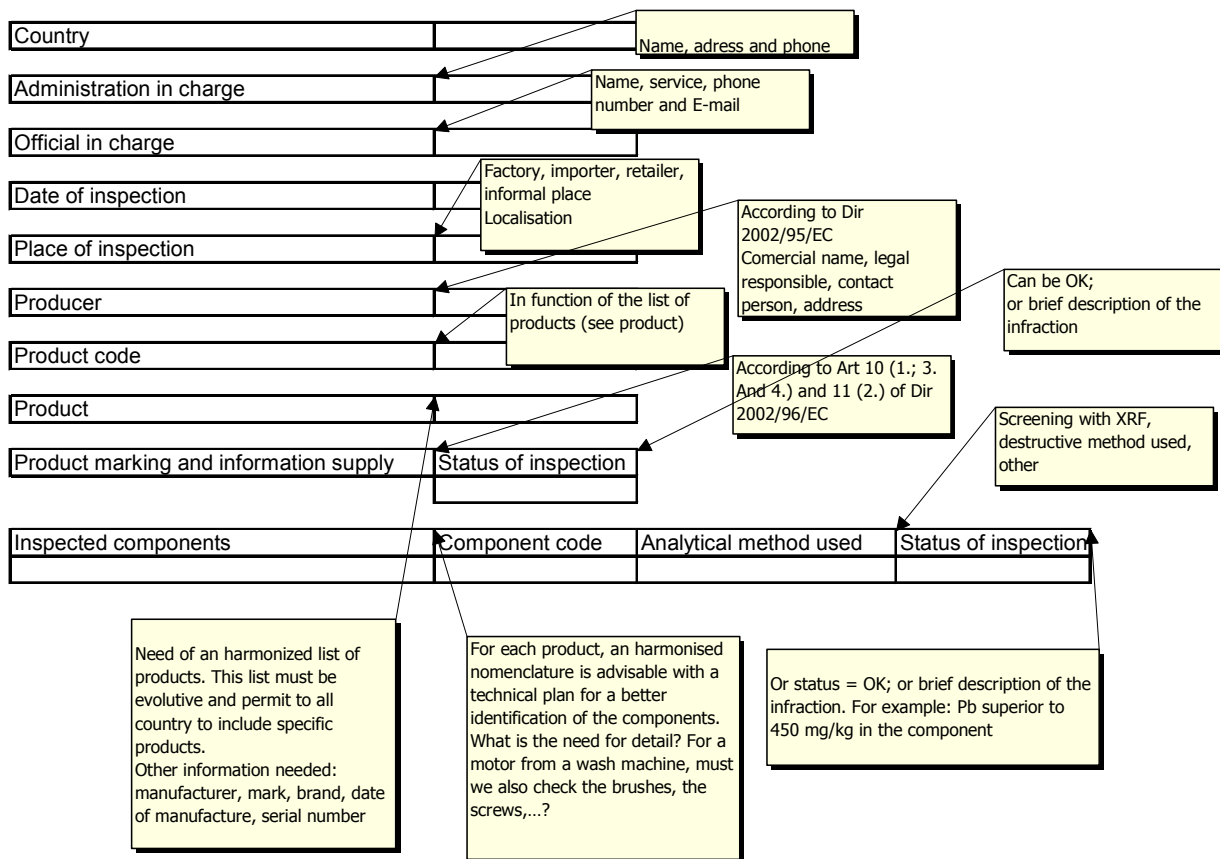
### 8.3 Comparison with New General Product Safety Directive

- The new General Product Safety Directive (2001/95/EC) revises the original Directive and in particular gives more detailed requirements for market surveillance. This includes an indication of how surveillance should be carried out which is absent from the RoHS Directive. Article 10 states that the Commission would set up a European Network of Member State Authorities. With most New Approach Directives, the Commission is reactive but it intends to take on a proactive role with the new GPSD. Enforcement authorities within Member States are already collaborating by targeting specific products.
- The GPSD has several important differences to RoHS. Enforcement of GPSD in most Member States is by National Authorities but this is carried out at a local level in Germany and in the UK (by Trading Standards). The market surveillance authorities for RoHS have not yet been published although this will not be trading standards in the UK and will probably be organised on a national basis. Enforcement of the GPSD in most Member States is by “hit

list” campaigns on product types which have reported safety concerns. Products are obtained from the market which are then tested.

- There are unlikely to be reports to enforcement authorities of non-RoHS compliant products. RoHS compliant and non-compliant products do not appear different and will function identically. It is most unlikely that a problem would be identified during recycling by analysis of scrap to detect unusually high levels of one of the banned substances because the banned substances will be present from pre-July 2006 products, exempt components and materials and from equipment not covered by RoHS. Enforcement authorities will need to carry out random testing of products in all categories and sub-categories to detect infringements. This is a very large task but could be shared between all Member States to pool resources and avoid duplication. In order for this to work effectively, all Member States should agree on the level of funding they will provide to carry out market surveillance in order to distribute the costs in a fair manner.

#### 8.4 Data reporting framework



**Figure 8. A proposed structure of an inspection data report sheet for RoHS and WEEE**

A standard format for enforcement authorities to report their findings from assessment of products would increase the harmonisation between Member States. These should be distributed to other States whether the product is found to be RoHS compliant or not. Circulation of data on RoHS

compliant products would help to avoid duplication of effort. Figure 8 shows a structure of an inspection report sheet which has been proposed by DG Environment <sup>33</sup>.

In many cases only selected components and materials will be assessed. This will be based on market intelligence, checking higher risk items and some random checks. All should be included in the report. As these reports will be shared, they should also include details of accreditation of the test laboratory (if any) and who has carried out the analysis.

Detailed guidance is required to carry out assessment and needs to be used in all Member States and be available to producers so that the same sampling and analytical procedures could be used and nomenclature is clearly understood.

## 8.5 Conclusions regarding Information Exchange Networks

1. Collaboration between enforcement authorities in Member States is mainly limited to passing on information where a safety risk has been identified. Different approaches are used in each Member State and the resources available vary significantly.
2. The New General Product Safety Directive (2001/95/EC) contains measures to encourage collaboration and the pooling of resources and has set up a Product Safety Network to co-ordinate market surveillance. This type of approach would be beneficial for the RoHS Directive as it would allow enforcement authorities to share analytical expertise, co-ordinate targeted campaigns to check the compliance of all types products and to avoid duplication of effort.
3. Most electrical equipment is sold in all EU Member States and if a procedure to ensure compliance is acceptable in one State then this must be recognised in all other States. Methods used by enforcement authorities for assessing products should be based on one standard procedure although any reliable and accurate analysis method could be used. Producers will also be permitted to use any reliable and accurate analysis procedure. This will be possible only if enforcement of RoHS Directive legislation by Member States is co-ordinated and enforcement authorities collaborate such as already occurring with the New GPSD.
4. Suggestions for enabling harmonisation and collaboration include:
  - Producing clear guidelines for product assessment procedures. These could also be used by producers.
  - Setting up a RoHS Network to co-ordinate market surveillance.
  - Sub-contracting analysis to laboratories with the required skills and expertise.
  - Using a standard reporting format and circulate results to all States including data on products found to comply with RoHS legislation.

## 9. Methodology and guidance on demonstrating compliance

It is clear that both industry and enforcement agencies would benefit from clear guidance on how to go about complying with the RoHS Directive. While it is beyond the scope of this project to produce such a guide the following shows how a checklist or decision tree type of approach could be used.

The checklist and decision trees in sections 9.1 and 9.2 respectively are initial ideas only and should not be relied on as procedures for demonstration of compliance.

### 9.1 Check list approach - an example

1. Do you have an assurance or declaration from suppliers that all parts, materials and components comply with the RoHS Directive

- If YES Go to 3, If NO Go to 2

2. Item has unknown RoHS status, either

- Find an alternative supplier or
- Analyse to confirm compliance with RoHS Directive unless type of material or component is known not to contain banned substances

3. Items are from new unknown supplier

- Carry out random analysis on selected items to check accuracy of claims about RoHS status.

4. Items from established supplier used for at least one year (and found to be reliable)

- Carry out random checks, 2 or 3 components or materials per supplier should be sufficient.

5. Item is listed as high risk

- It is likely that producers will want to check the composition of a larger proportion (every batch) of materials and components that have a higher risk of containing a banned substance. Examples of higher risk items listed in Table 10.

**Table 10. Examples of higher risk components and materials**

| Material or component  | Risk element                             |
|--|--|
| Solders (note several different solders may be used)                 | Lead                                     |
| Red, orange, yellow plastics   | Cadmium (pigments)                       |
| Plastics - HIPS and others that use PBB and PBDE as flame retardants | PBDE, PBB                                |
| Plastics - PVC   | Pb and Cd as stabilisers                 |
| Aluminium castings, galvanised steel parts                           | Hexavalent chromium passivation coatings |
| Relay and switching contacts   | Cadmium                                  |
| Light sensors  | Cadmium                                  |
| Paint  | Lead driers                              |

## 9.2 Decision-tree approach - an example

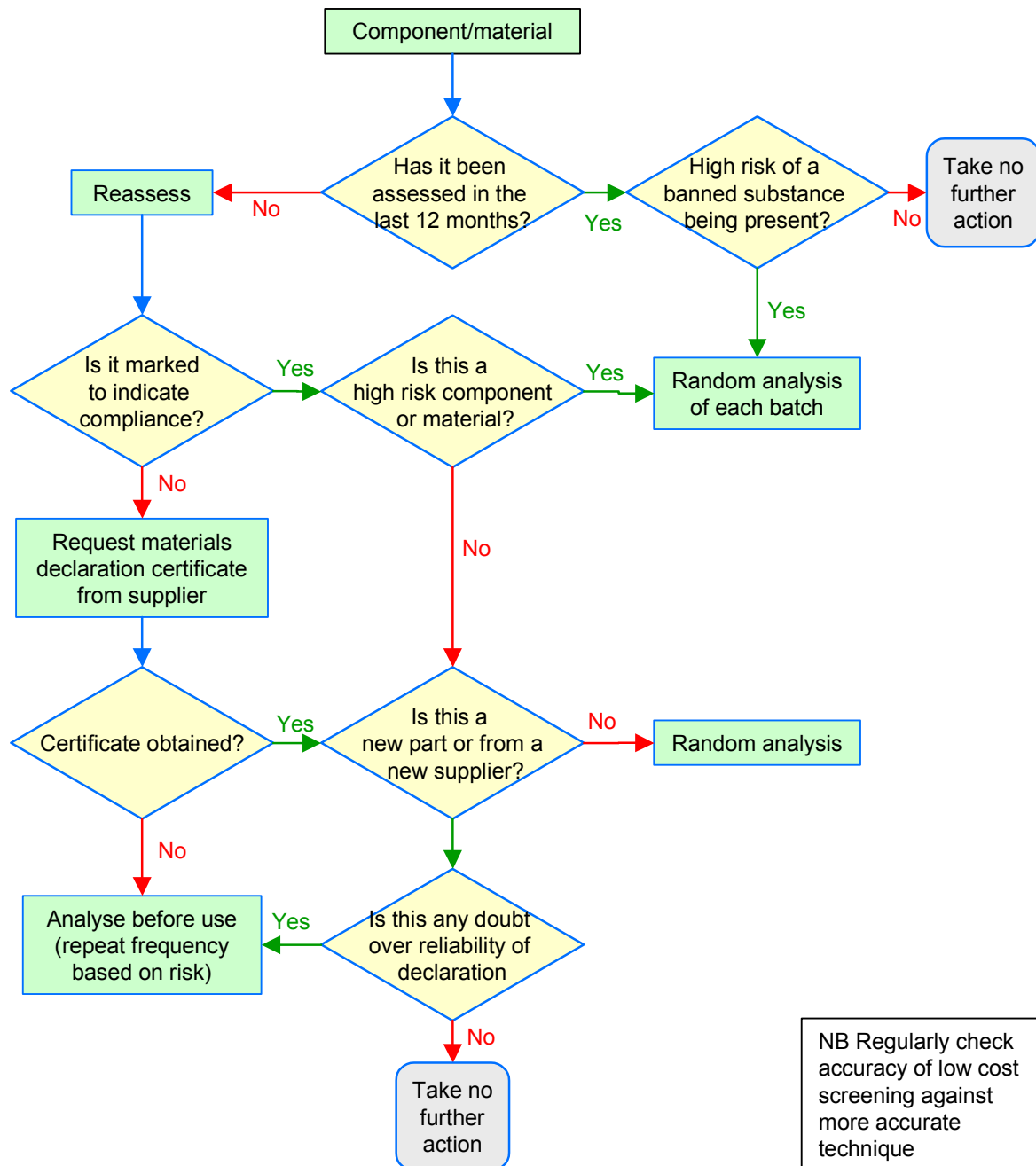


Figure 9. Example of a decision tree approach for demonstrating RoHS compliance

A comprehensive guide could be produced when the definition of the maximum concentration levels has been agreed by the TAC. This should be produced by the TAC in collaboration with representatives of the electronics industry as well as consultants with expertise in component analysis.

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- Tin Technology
- Underwriters Laboratory (UL)
- Weald Components (Component manufacturer and distributor)

## **Annex A - Analysis techniques**

### **A.1 Analysis for lead, cadmium and mercury**

Various analysis techniques can be used for these metals as well as for total chromium. The more frequently used methods are described here.

The first two methods; AAS and ICP-AES, both require the material to be first dissolved. This is possible for single materials or coatings that can be selectively dissolved but the analysis materials located within complex components are best analysed by using either ED-XRFA or SEM/EDX of a cross-sectioned sample although both have limitations which are discussed below.

Mercury in the form of metal is an unusual case as it is a relatively volatile material and so some analysis techniques are unsuitable. The most commonly specified method is cold vapour atomic adsorption spectroscopy. Mercury compounds are not volatile and so can be analysed using more methods.

Certain types of fluorescent lamps are exempt from the RoHS Directive. The exemptions permit the use of mercury in quantities up to certain quantities within each lamp. It will be necessary to determine how much mercury is present in a lamp to ensure that these are RoHS compliant but no reliable methods are known to ERA

Glow discharge optical emission spectroscopy is a specialised technique that is not widely used but would be useful for the analysis of electroplated coatings with little sample preparation required. Multiple layers of different metals can all be analysed separately and with very low detection limits. A variation on this technique can be used for paint or polymer coatings. This does not however distinguish the different forms of Cr or identify bromine compounds. The minimum film thickness is normally 0.1µm and so will not be suitable for very thin chromate passivation coatings.

Analysis of one material within a whole components after complete dissolution has problems which are explained in Section 6.5. This approach is possible only if the proportion of material to be analysed is known as a mass percent of the whole component and the metal being analysed is absent from all of the other phases including as impurities.

#### **A.1.1 Atomic adsorption spectroscopy (AAS)**

AAS is widely used for the quantitative analysis of metals in solution and can be used for the analysis of a wide variety of materials including plastics, metals and ceramics, all of which must be pre-dissolved. The technique has been in common use for many years in across many applications resulting in a thorough understanding of the analytical issues. Standard methods exist which can be applied to most the sample matrix types likely to be encountered and the accuracy and sensitivity will be adequate for most applications assuming that sufficient sample is available to achieve metal concentrations in solution around 10mg/l. Sampling of cadmium at the 0.01% limit is likely to cause

sampling problems however. For chromium, which tends to form refractory oxides, a hotter nitrous oxide/acetylene flame may be required to eliminate chemical interferences. The absolute precision of the method is good with multiple readings from a single solution giving relative standard deviations better than 1%.

Much greater sensitivity and smaller sample volumes are used with graphite furnace AAS which uses thermal atomisation with the use of a flame. The method is prone to interference, however, requiring greater skilled analysis and it suffers from a generally much slower sample throughput. The method is likely to be used in only exceptional cases and would not normally be considered as a standard method for compliance testing.

### **A.1.2 Inductively couple plasma atomic emission spectroscopy (ICP-AES)**

This technique, which is often referred to simply as ICP, uses an inductively coupled plasma rather than a flame to dissociate the sample and excite the atoms to emit light of characteristic wavelengths. The very high temperature of the plasma results in a complete dissociation of the sample molecules and an elimination of almost all chemical interferences. The high-energy source also results in greater sensitivity for almost all elements but particularly those which would tend to form refractory oxides at lower temperatures. The measurement of emission rather than adsorption allows several elements to be determined in rapid sequence or simultaneously. These advantages of a multi-element analysis and higher sensitivity have resulted in the technique replacing Flame AAS in many applications.

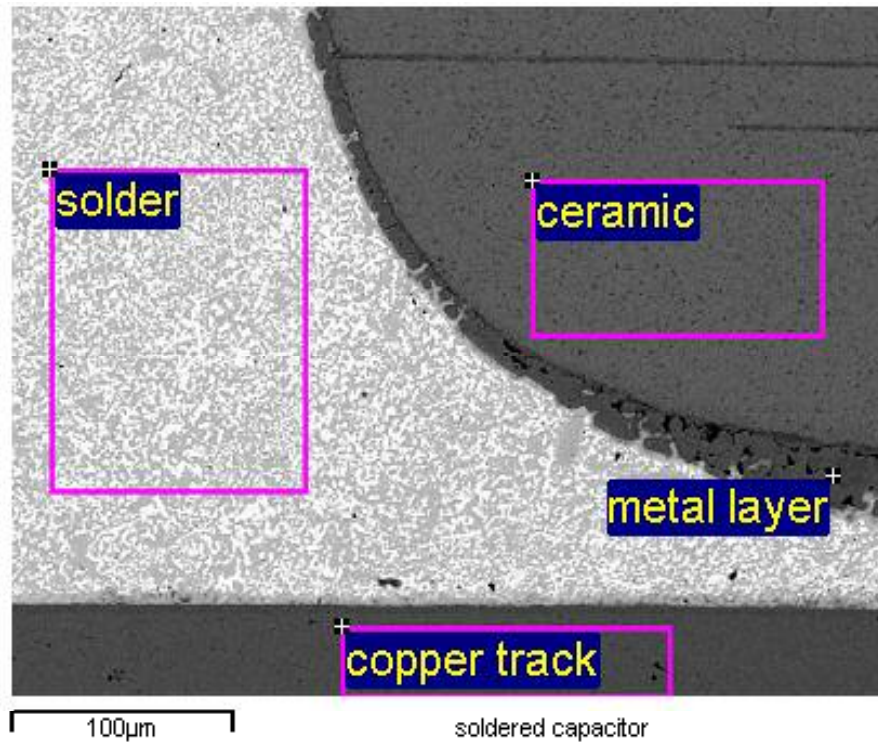
The stability of the plasma is good with precision data as good or better than Flame AAS. The limits of the method are those shared with Flame AAS; that is, the isolation of the homogeneous phase and its complete dissolution.

Real sensitivities will be instrument specific but generally detection limits of around 0.01 µg/ml should be possible for lead, chromium and cadmium making the analysis for these elements at the likely specified limits readily achievable.

### **A.1.3 Energy dispersive X-ray analysis with scanning electron microscopy (SEM/EDX)**

Very small areas of the surfaces of components and materials can be analysed using this technique. It is suitable for termination coatings, for example to determine the lead content of a tin alloy solderable coating, analysis of plastic encapsulation around a component to determine if there is lead above the permitted concentration. It would also determine whether there is more than 0.1% of cadmium but can not be used to determine concentrations below 0.1% and where the limit is likely to be 0.01%. Hence if the presence of cadmium is suspected, a different method will be required. SEM/EDX will detect bromine but cannot determine which brominated flame retardant is present. The detection limit for bromine can be 0.05% or lower if aluminium is absent and so this can be used to determine whether there are any brominated flame retardants present but further work will be required to determine if this is a sensitive enough technique.

One of the main advantages of SEM/EDX is the small analysis area. Some components have very small features, especially internally and these can be analysed after breaking open the component. This is best done with a cross-section as this exposes a flat surface for analysis as shown in the example in Figure 7.



**Figure 10. SEM image of cross-sectioned multi-layer ceramic capacitor. The solder, ceramic, metal layer and copper track can all be analysed separately**

#### **A.1.4 Energy dispersive X-ray fluorescence analysis (ED-XRFA)**

X-ray fluorescence analysis instruments are either energy dispersive or wavelength dispersive. Wavelength dispersive instruments offer the highest accuracy and lowest detection limits but are suitable only for homogeneous materials and can not be used for components or complex assemblies. Energy dispersive instruments are less sensitive and can be used for components as well as materials but these do have limitations.

Two types of commercial energy dispersive instrument are available for the analysis of materials and electrical components:- portable handheld instruments and desktop instruments.

### Handheld instruments

Hand held XRF analysers have been used for many years to obtain quantitative or semi-quantitative analysis of field samples. Sampling for more accurate lab based instruments is deemed impractical or too slow. Applications are found in fields such as geological survey, metal reprocessing and environmental assessment.

These hand held devices will normally be limited to an application specific set of elements and a library of calibration data for those elements within a certain set sample matrixes. Analysis of materials outside the specified range will produce results but these cannot be used for quantitative information.

Traditionally these devices use radioisotopes as the excitation source with more than one source being required to cover the whole of the normal element range which usually covers elements of atomic number 22 (Titanium) and above. Typical gamma sources used in these devices are americium 241, cobalt 57, iron 55 and cadmium 109. Cadmium 109 is probably the most widely used. The recent development of small x-ray tubes suitable for hand-held devices offer the possibility of more flexible systems capable of measuring all elements and without the regulatory complications associated with radioisotopes but the long-term reliability of this new technology is still to be proven.

An ideal sample for examination by a hand held XRF device has the following characteristics:

- Of uniform composition
- A previously known or calibrated matrix
- A flat material that can be pressed directly against the sampling window
- A sample area with dimensions greater than 1cm<sup>2</sup>.

For survey work conducted on electronic goods non-destructively and without prior preparation these conditions will not usually be met and limitations are inevitable. The examination of samples with less than uniform composition and complex geometries will produce results that are qualitative with uncertain detection limits. The sampling depth achieved will depend greatly on the nature of the sample matrix, the excitation energy being used and the element being sought but will typically be a few microns in metals to a few millimetres in plastics. As a result thin paint or polymer layer such as conformal coatings could be effectively transparent although thicker and higher density layers such as electronic packaging will represent an effective barrier to the X-rays.

The limits of detection for any elements will also be affected when the substrate is sub-surface but the magnitude of the effect is likely to be unpredictable. Not all elements are equally responsive to this technique and in an EPA report assessing the use of portable XRF suggests that the chromium response was particularly poor and this technique is inferior to conventional laboratory based methods.

With a radioisotope source typical optimum detection limits will be between 10-20 mg/kg for elements like lead and cadmium and 100 mg/kg for chromium.

This method is suitable mainly as a screening method for importers and manufacturers where many of the sampling difficulties are likely to be reduced, but even here these components are likely to be themselves mixtures of several “homogeneous phases” and quantitative assessment will not be possible without the use separation methods. Despite the difficulties and limitations inherent in the use of this equipment it will undoubtedly find use in some, albeit limited, circumstances.

The technique could be used to detect lead in solder, lead in polymers, cadmium in polymers and coatings. The technique can also determine the presence of bromine in polymers and this could be used to screen for the presence of brominated flame-retardants but with no information about its identity. The inability to distinguish between the various forms of chromium would suggest it will have limited value in assessing the presence of CrVI.

It may be possible to mask parts to be analysed to avoid interference from the surrounding materials. This may be useful but the response could be used only to confirm that something does not comply with the RoHS Directive due to the limited signal from a small area, a negative result may not confirm RoHS compliance.

As with all testing methods the appropriateness will depend on cost and time benefits as well as the nature and the quality of the information required, in most cases the use of hand held XRF should only be used as an adjunct to other more selective and quantitative procedures for screening materials and components to detect those that clearly are not RoHS compliant or materials that have concentrations of the banned substances that are well below the RoHS limits.

This technique is being widely promoted as a simple low cost screening technique. It can be useful for checking incoming materials as long as the results from the instrument are regularly checked by analysis of the same material using a technique such as ICP or AAS which are accurate and the other substances present do not affect their response.

### **Desktop instruments**

X-ray fluorescence spectrometers come in many forms; traditionally accurate analysis of complex mixtures including both metals and ceramic materials has been achieved using wavelength dispersive x-ray analysis. Quantitative analysis usually involves precise sample preparation methods often destructive in their nature. Between this and portable hand-held devices there are many laboratory or bench-top based energy dispersive (ED) devices currently on the market.

ED-XRFA instruments are normally based on X-ray tube excitation with solid-state detectors based on lithium-doped silicon. The resolution of these detectors is around 170eV which, although much poorer than the resolution offered by wavelength dispersive instruments, is good enough to analyse most complex materials. These devices offer better precision and detection limits than the portable types and are primarily designed to offer fast, non-destructive analysis. All machines can analyse

elements with a atomic number greater than 22 (titanium) and some (e.g. from Oxford Instruments) can also analyse lighter elements (from sodium, atomic number 11).

All the elements covered by the Directive are potentially analysable by this technique. The problems inherent in portable devices - geometric effects, analysis depth, spectroscopic and matrix effects are still relevant but hardware and software modifications are available to moderate their impact. Desktop energy dispersive x-ray fluorescence spectrometers have been designed specifically for detection of cadmium and lead in electronic components. An instrument from Horiba analyses a relatively small surface area, diameter 1.2mm. Penetration depth is limited so this instrument can give reliable information but will have limitations. Clearly bulk materials can be analysed, metallic surface coatings on components or PCBs are suitable if their thickness is greater than the instrument's penetration depth and their area is at least diameter 1.2 mm. Accuracy is much lower if the sample size is much less than the instruments minimum sample area although some instruments are able to map surfaces (e.g. Fischer) and so can discriminate and accurately analyse very small areas.

Materials are normally placed on a flat sampling area and are scanned from below, only the flat surfaces in contact with this sampling region are available for quantitative analysis. Instruments that incorporate video imaging and motorised stages can be used show the exact area that is being analysed and can automatically analyse a large number of components or several sites on a single location.

#### Detection limits

Real detection limits will depend on many factors including the complexity of the mixture and the physical form of the sample but for most metallic elements these will be better than 20mg/kg (0.002%) in a flat uniform samples - sufficient to detect all the elements covered in the RoHS Directive at their most likely lower permitted levels.

#### Calibration

For accurate quantitative results a series on matrix specific calibration standards are required, perhaps 10 or more standards to cover a wide concentration range. For screening purposes the use of fundamental parameters based on the response to pure elements may be sufficient. This method uses mathematical methods to calculate and compensate for the effects of inter-element matrix effects. Screening using fundamental parameters could produce useful semi-quantitative information within a few seconds but when the concentration of a banned substance is close to the specified limit then other more accurate quantitative methods may have to be used to confirm the result.

Research carried out recently (Underwriters Laboratory) has shown that ED-XRFA can give very misleading results if fundamental parameters are used for calibration and also if the wrong matrix is used. Inaccuracy occurs even if a different filler is present in a polymer. In one quoted example, the instrument indicated 1000ppm with a polymer containing only 500 ppm. The error can be considerably worse than is often quoted if calibration is also not carried out correctly.

### Analysis of inhomogeneous samples

Meaningful quantitative analysis can only be achieved for homogeneous materials, that is, on layers of uniform composition that are thicker than the analytical depth and which have a size greater than the measurement spot or window. The x-ray penetration depth in metals is likely to be only a few microns and layers thinner than this will be subject to errors. Small sample size is less of a problem as in many modern machines it is possible to change collimators to produce different spots sizes. Analysis areas can be less than 1 mm in diameter and with a fine primary beams areas can be scanned which can provide compositional information on areas much smaller than this. The use of motorised stages and video cameras could in principle select very small areas for analysis and offer the possibility to interrogate complex electronic devices for a particular homogeneous phase of interest and one with geometry favourable to quantitative analysis.

### Conclusion

ED-XRD is still a relatively new technique and there is little experience in the direct examination of electronics goods. However the combination of speed, non-destructive nature (for materials at the surface), and the possibility of the analysis of small sample areas make it a potentially useful analytical or screening technique particularly in combination with other more precise analytical methods. It does however have limitations which need to be understood by analysts. Calibration using representative standards is essential to obtain accurate data otherwise very significant errors can occur.

#### **A.1.5 Wavelength dispersive X-ray analysis (WD-XRFA)**

The same potential problems with ED-XRFA can occur with WD-XRFA. This technique is normally used only with homogeneous materials such as plastic, metals and ceramics. It is not suitable for individual components unless these are ground to a fine homogeneous powder which is analysed as a flat layer.

#### **A.2 Analysis for hexavalent chromium**

Hexavalent chromium is used mainly as thin passivation coatings on metal surfaces or as corrosion inhibiting pigments in paints. If deliberately used, the concentration in a coating or paint will exceed the proposed 0.1% lower limit for a homogeneous material but may not exceed that concentration in a component or a multiple layer electroplated coating.

As chromium in the hexavalent form only is banned, it is necessary to distinguish between chromium in its different oxidation states. Most techniques including AAS, ICP XRF and EDX analysis all analyse quantitatively total chromium but do not indicate the proportion, if any, in the hexavalent state. As with the other banned metals, analysis is carried out by first dissolving the material. It is essential however to ensure that any trivalent chromium is not oxidised to the hexavalent form and also hexavalent chromium is not reduced to the trivalent form. US standards (e.g. OSHA method ID-103) for analysis of air dust samples recommend a carbonate buffer for selective dissolution of CrVI.

This method could probably be modified for hexavalent chromium treated metal surfaces but would need validation. The advantage of the carbonate buffer is that it will dissolve chromates (CrVI salts) without affecting the underlying metals.

Thin surface coatings can be dissolved to determine the concentration but knowledge of the coating thickness will be needed (this could be as mass/unit area). Analysis in paint coatings is specified in BSI and ISO Standards. There are many Standards for analysis of hexavalent chromium in water and in air. These are not directly applicable to electrical equipment but could form the basis of analysis techniques. Most methods rely on the formation of a coloured solution by reaction of the hexavalent chromium with a reagent that produces a characteristic colour. The intensity of the colour is accurately measured with a spectrometer. A simple qualitative method is described in ZVO-012-QUA-02. This requires minimal equipment and a few chemicals. Formation of a pink-purple colour confirms that hexavalent chromium is present but a negative result does not prove that hexavalent chromium is not present.

### **A.2.1 Ion chromatography (IC)**

Ion chromatography is a liquid chromatography widely used in the analysis of ions in aqueous solutions, the technique is particularly suited to the analysis of low-level inorganic anions such as sulphate and chloride. Modern instruments can easily detect and quantify inorganic anions at sub-ppm concentrations.

In regards to RoHS compliance the technique may find use in testing for hexavalent chromium via the analysis of chromate ions in solution. As with all the analytical methods for chromate, the initial problem is the extraction of the chromate ions from the test sample but having done so the technique will provide an accurate and reproducible method to determine quite low levels of chromate in the resulting solutions. The EPA method 218.6 for the analysis of chromate in water uses a post-column reaction with diphenylcarbazide and absorbance detection at 530nm and has a detection limit of 0.4 mg/l.

### **A.2.2 PBB and PBDE flame retardants**

PBB and PBDE are only two of the classes of brominated flame retardants that are used in plastics. Analysis of bromine is not sufficient as this may be present as a permitted compound. XRF or EDX can be used to determine if any bromine compounds are present then if not then it would be safe to conclude that PBB and PDE are not present (i.e. present at >0.1%). Additional analysis will be needed to determine; a) if PBB or one of the banned PBDE compounds are present and b) at what concentration.

There is a lot of research on this topic and several commercial laboratories offer analysis of various materials containing PBB and PBDE. Most work and the techniques used have been based on trace contamination analysis in water, sediments and marine organisms such as fish which contain these compounds at much lower concentrations and are much more complex mixtures that occur in most

plastics. It is likely that simpler techniques to the ones being offered commercially may be suitable but these will first need to be proven.

### **A.2.3 Gas chromatography (GC)**

Gas chromatography is a versatile means for the separation and identification of complex mixtures of volatile organic compounds such as brominated flame-retardants. However the literature concerning the analysis of poly brominated biphenyls (PBBs) and poly brominated diphenyl ethers (PBDEs) is primarily concerned about the estimation of these species within food and other biological materials linked to the recent interest in the fate of these compounds in the environment. The analytical approach for biological samples usually involves an extraction step to obtain the analytes in solution often using lengthy soxhlet extraction steps, a clean-up stage to remove unnecessary or interfering compounds and finally the determination step usually involving mass spectrometry as the detection step. There are many technical problems involved in the quantitative extraction and isolation of this group of compounds often at very low levels and from the very complex matrix represented by biological samples. The analysis is further complicated by the large number of possible isomers for these compounds, with over 200 for PBDEs alone. A combination of high-resolution chromatography and high-resolution mass spectrometry has been used to allow for the complete separation of all these forms. There appears to be no general accepted standard method currently available for the analysis of these substances in polymers however the much higher levels present when used in polymers and the more simple sample matrix make this, in principle, a much simpler analytical task.

### **A.2.4 Sample extraction**

To analyse brominated flame-retardants in a polymer the sample must be dissolved in a suitable solvent or the analyte must be completely extracted. No single solvent or extraction method will work for the wide range of polymers possible in electronics goods. For each polymer a specific solution or extraction procedure will be required. For some of the more resistant and heavily filled polymers accelerated extraction procedures, such as microwave digestion, may be required. In most cases an additional step will be required to remove the bulk of the polymer from the sample prior to injections onto the GC column. In each case a measure of the extraction efficiency or recovery will be required to allow accurate quantification.

The relatively high thermal stability of these brominated compounds is sufficient to allow thermal decomposition methods to be considered. Methods such as pyrolysis-GC have the advantage that no solvents are required and therefore universal methods may be formulated although greater care is required in obtaining representative samples and in the method calibration. Pyrolysis-GC very rapidly heats a small amount of sample to above the decomposition temperatures (450°C) in the absence of air and the volatile compounds are passed directly onto a GC column. The method will need to be carefully optimised to give the maximum separation of the flame-retardant compounds with the minimum decomposition and this may need to be a polymer specific process, however the advantages of avoiding the development of lengthy liquid extraction procedures would be considerable.

### **A.2.5 Detection methods**

Detection can be achieved either by comparison of retention times with known standards, which have recently become available commercially, or by using mass spectroscopy to identify which compounds are present.

Most of the PBBs and PBDEs (except deca-BDE) will be present as a mixture of isomers. These are essentially different geometric forms which all have very similar chemical properties. The legislation does not distinguish these as different compounds and for validation of compliance, the identification and quantification of individual isomers will probably not be required and the complete resolution of all peaks unnecessary. The use of (now standard) high temperature capillary GC columns is likely to provide sufficient resolution in most cases. The low vapour pressure and high boiling point of the more fully brominated forms will require columns to operate at high temperatures (350°C) for extended periods potentially leading to on-column decomposition during the analysis or degradation of the column itself. However many manufactures have now producing specially designed high temperature columns which should allow for the analysis of all the PBDEs including the least volatile deca-brominated form.

The use of high-resolution mass spectroscopy common in environmental samples will not be essential for RoHS testing. The simpler matrix and higher concentrations will mean that modern bench-top mass spectrometers will almost certainly have sufficient sensitivity and resolution. The use of mass spectroscopy for detection provides high sensitivity as well as atomic mass information sufficient to provide unequivocal identification of most the eluted peaks. With modern instruments, direct detection using electron capture will always provide sufficient sensitivity but in this case standards containing know mixtures of isomers will be required to positively identify the peaks. Until recently these standards were difficult to obtain, but due mainly to the interest in environmental issues a full range of isomer standards are now available specifically for GC analysis.

In summary, none of technical issues relating the to the analysis of flame-retardants in plastics appear to be insuperable but as yet there appears be little practical experience. Until the method has become routine and applied to a wide range of materials is impossible to know if the analysis will be more or less difficult then anticipated. There are currently a few laboratories offering this type of analysis using a range of GC based methods with quoted detection limits better than 1 µg/kg.

### **A.2.6 Probe Mass Spectroscopy (probe – MS)**

An alternative technique that is being offered by at least one commercial laboratory is probe – MS. This technique uses solid plastic without the need for extraction or sample dissolution a solid sample of plastic is heated rapidly to volatilise all additives including any PBB or PBDE. The mass spectrometer is used to identify and quantify the evolved compounds. This technique is new for this application and may not be suitable for very complex mixtures of brominated flame retardants.

### **A.2.7 High performance liquid chromatography (HPLC)**

Liquid chromatography represents an analogue to GC in which a liquid rather than a gas is used as the carrier or mobile phase. It has the advantage of higher separation efficiency and can separate compounds irrespective of their volatility. The method could be successfully applied to analysis of PBBs and PBDEs using a range of detection methods including UV adsorption and light scatter detection. Mass spectrometric detection is also possible with HPLC but this is technically more complex than with GC because of the need to separate the analytes from the liquid mobile phase. Being a liquid based technique the method will need to start with extraction and clean-up stages and will not have the advantage of a possible thermal decomposition route.

Despite the lack of technical barriers HPLC is generally regarded as a more difficult technique for routine analysis than GC and the number of literature references to the use of HPLC applied to the analysis of brominated flame-retardants is relatively small. As GC methods are already well developed in the environmental field it will be easier to modify these for polymer analysis than to develop HPLC methods.

### **A.2.8 Fourier transform infra-red (FTIR) spectroscopy**

Organic compounds show strong adsorption in the infrared part of the electromagnetic spectrum due to the many possible modes of vibration within the molecules. The infrared spectra of many polymers can be rapidly obtained using attenuated total reflection (ATR) devices. This method could, in principle, be used to screen for the presence of brominated flame-retardants within polymers but has many limitations:

- Spectra can be difficult to obtain from heavily coloured or filled materials.
- ATR spectra are predominately of the surface and may not reflect the bulk composition.
- Interference from peaks due to the sample matrix will be common in the region where most of the characteristic carbon-bromine absorption will be found.
- The method is of low sensitivity with several percent required to be above the detection limit.
- Without separation it will be difficult to identify if a mixture of compounds is present.

These potential difficulties suggest that this technique will not find use as a generally applicable screening test however they may be certain specific applications where it might find use. The Shimadzu application note A358<sup>34</sup>, for example, demonstrates that 5% wt/wt of DBDPE is detectable by this means in polystyrene but information on other systems is not available.

The screening of plastics for the presence of bromine may be more easily achieved using the Belstein hot wire test.