14 November 2003



Dear members of TAC on WEEE & RoHS

We, Japan Business Council in Europe (JBCE)¹, are the organisation representing Japanese companies with significant operations in Europe. Our members are among leading multinational corporations in the world.

We strongly called for the Europe-wide uniform test method in the context of RoHS maximum concentration values in our previous position paper dated 11, June 2003. This paper is mainly focused on our proposal of the harmonized test protocol for the governmental market surveillance.

We would be more than happy if this paper could be of any assistance to your discussion in the next TAC meeting .

If you have any questions, please feel free to contact our secretariat or myself.

Sincerely yours,

Takashi Sugiyama Chairman, Environmental Committee Japan Business Council in Europe

¹ For details, including a list of its member companies, please refer to the following website: http://www.jbce.org

Testing compliance with the RoHS substance ban

Japan Business Council in Europe(JBCE)

The Japan Business Council in Europe (JBCE) is convinced of the importance of carrying out as much random testing as possible of electric and electronic products that enter or are made available on the European market. Enforcement of the RoHS substance ban should not remain just a statement of good intentions, but should be put in practice through systematic testing of EEE which should be shared with all EU member states governments.

The RoHS directive prohibits lead, mercury, cadmium, hexavalent chromium (chrome VI), and brominated flame retardants PBB and PBDEs from being used in EEE. Testing methods to determine presence and levels of these banned substances differ from each other.

There are, however, a few concrete issues that need to be dealt with to enable testing to be carried out in an efficient and meaningful way. In general, there are two types of testing methods: non-destructive and destructive one. The former require much less cost and time than the latter, and show enough accuracy for the concentration range higher than a few 10 ppm. Therefore, JBCE would like to encourage authorities to use the non-destructive methods only, unless it is not possible to identify the banned substances such as the case of chrome VI. A detailed explanation is given below.

1. Basic concept of inspection method for enforcement

For the inspection we propose to use the Energy Dispersive X-ray Fluorescence device (EDXF) in general. The basic strategy is described below:

- (1) Screening : Hand-held type EDXF inspection focuses on screening products into three groups: non-compliant, gray area and compliant.
- (2) Final test : desk-top type EDXF inspection is conducted to divide the products belonging to the gray area into either non-compliant or compliant.

An example (see attachment 1):

If the measuring error of the hand-held EDXF is 20 % around the specified threshold concentration range of 1000 ppm (0.1%), products with a banned substance with the measured value below 800 ppm can be clearly considered as compliant, and those above 1200 ppm as non-compliant with the RoHS substance ban. Products in the gray area (800 ppm – 1200 ppm) should be considered as compliant. If authorities would like to narrow the gray area, they can use the desk-top EDXF. If the desk-top EDXF has a margin of error, let us say 5 %, then products with 1050 ppm or below can be considered as compliant.

Concerning two brominated flame retardants (PBB and PBDE) and chrome VI, the EDXF detects different kinds of bromine as a total and different kinds of chromium as a total, respectively. For distinguishing the banned substances from others, a further step is necessary (see Chapters 3 and 4).

Please note that the various factors such as the shape, thickness and matrix of the target item affect the measured values. Therefore, each detail should be discussed and standardized by **a standard committee** that consists of specialists of this field.

2. Lead, mercury, and cadmium

For measuring these three substances the EDXF method is appropriate. There are the hand-held type EDXF and the desk-top type EDXF. The hand-held EDXF can easily be used on site to determine these substances in the concentration range around 100 ppm and/or 1000 ppm within a certain measuring error.

The desk-top EDXF is a little more expensive, but more accurate than the hand-held EDXF. For measurements of small items it is more convenient. Furthermore, most of this type equipment does not require any person authorized for using X-ray.

JBCE would like to propose that for the large items the first rough screening with the hand-held EDXF should be followed by the more accurate measurements with the desk-top EDXF. For small items the desk-top EDXF can be used from the beginning.

Attachments 2 and 3 show flow charts of inspection for lead in plastic and lead in metal, respectively. These are typical examples which deviate from the general scheme shown in attachment 1.

JBCE would like to encourage the authorities to consider the desk-top EDXF as the final testing method in practice, allowing for a relatively low margin of error whilst still avoiding destructive sample preparation using chemicals and much more expensive testing.

As mentioned in Chapter 1, any details should be discussed by specialists. We recommend further to use a calibration curve with standard samples for determining the concentration. This can compensate the individual differences of equipment.

3 PBB and PBDEs

The EDXF (both the hand-held and desk-top type) can only detect the presence of brominated substances, and cannot distinguish between the banned PBB/PBDEs and other brominated flame retardants such as TBBPA(Tetrabrombisphenol A). TBBPA is widely used, for instance, in printed circuit boards that can be found in practically every electronic appliance. Therefore, it would appear at first sight that only the expensive and time consuming destructive method by chemicals with GCMS (Gas Chromatography/Mass Spectroscopy) is applicable.

However, JBCE has come across an easy non-destructive testing method that can distinguish between those flame retardants that are banned and those that are not: the Fourier Transform Infrared Spectroscopy (FTIR). FTIR can identify the brominated flame retardant used, if it is added to plastics with a concentration higher than 3 %.

In order to be effective and perform its function of actually preventing fire for a certain period, at least 5 % (50 000 ppm) of a brominated flame retardant is needed in plastics. It does not make any sense to use such flame retardants below this level, and EEE manufacturers indeed never do so. Furthermore, two different flame retardants are usually not used together in one plastic material, and PBB/PBDEs are hardly contained as an impurity in other brominated flame retardants.

Therefore, if the brominated flame retardant is identified with FTIR as TBBPA(or other not-banned brominated flame retardants), we can conclude that neither PBB nor PBDEs are present in the measured plastics at more than the specified threshold concentration.

If the identification of different PBDE's such as penta-, octa- or deca-BDE with the concentration range of 1000 ppm are required, the GCMS method should be applied.

Attachment 4 visualizes the inspection strategy of brominated flame retardants.

Thus, to simplify the inspection JBCE would like to argue for generalised use and acceptance of the FTIR test method in addition to the EDFX. Since FTIR is very sensitive to the condition of the surface of the materials, any details should be discussed by specialists.

4 Hexavalent Chromium (Chrome VI)

Chrome VI is banned by the RoHS directive because of its hazardous properties. Chrome III, on the other hand, has no toxic properties and is considered to be safe for use. Here, no non-destructive methods seem to be available that can distinguish among chrome VI, chrome III and metallic chrome.

For the screening, the EDXF can be used to know whether any chromium is present. If the concentration of chromium is significantly low, the product will definitely comply with RoHS. If the concentration of chromium detected is higher than the specified threshold, a further test is necessary to distinguish whether chrome VI is involved.

For this purpose JBCE would like to propose the diphenyl-carbazide method (a kind of colour test), which is one of the simplest methods among the destructive tests. The suspected part should be dipped in water and one should wait long enough to dissolve chromium ions. If the chrome VI is contained in the solution, it reacts with diphenyl-carbazide and shows red colour. The concentration can be measured with a spectrophotometer.

Attachment 5 illustrates the inspection method for Chrome VI.

Since chrome III can be changed to chrome VI during elution, the condition of elution is very critical. Details should be discussed by specialists.

5 Conclusion

As a summary we propose:

- 1. In principle, only non-destructive methods should be applied.
- 2. For lead, cadmium and mercury EDXF is sufficient.
- 3. For PBB/PBDE's the combined use of EDXF and FTIR is practical.
- 4. For hexavalent chromium, the diphenyl-carbazide method (a colour test) is the simplest test after screening with EDXF.

We would like to emphasize the importance for setting **a standard committee** to discuss any details, because there are many factors which should be taken into account. Furthermore, it should be discussed how to treat, during inspection, exempted applications in the Annex of RoHS directive

JBCE would be happy to collaborate with you for establishing efficient inspection methods as a member of the standard committee.

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General flow chart

Attachment 1



Abbreviations

EDXF: Energy Dispersive X-ray Fluorescence TC: Threshold Concentration

(Remarks)

- 1. The factors which define the values a and b depend on the measuring errors of the EDXF equipment. As examples factors of 0.2 and 0.05 for the simple measurements and for the more accurate measurements, respectively, are used.
- 2. If the shape and/or matrix of the target item are not ideal, the detected value is usually lower than the real concentration.
- 3. If the measurements with EDXF are not accurate enough, or not specific enough, other methods should be applied.
- 4. The basic chart shows a very general strategy only. Each elements with different environments require different scheme.

Example: Lead in plastics



Example: Lead in metal (except soldering alloy)



Example: PBB, penta-BDE, octa-BDE in plastics

Attachment 4



Note *: If the plastic is acrylic, FTIR method can not be applied. Go to GC/MS.

**: This is only the case when penta-/octa-BDEs are banned and deca-BDE is not banned.

Abbreviations:

FTIR: Fourier Transform Infra Red Spectroscopy GC/MS: Gas Chromatography/Mass Spectroscopy



* Document check necessary: on which materials it is galvanized? (For example, stainless steel contains Chromium metal.)
** During water elution some Cr³⁺ are converted to Cr⁶⁺