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Ministry of Information Industry of the People's Republic of China Industrial Standard

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Testing Methods for Regulated Substances in Electronic Information Products

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Testing Methods for Regulated Substances in Electronic Information Products

Contents

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Foreword

This Standard has been prepared as a technical supporting Standard for *Method of Control and Management of Pollution from Electronic Information Products* (hereinafter referred to as the 'Management Methods'), and specifies the methods of measuring six toxic substances in electronic information products, namely lead (Pb), cadmium (Cd), hexavalent chromium (Cr VI), mercury (Hg), polybrominated biphenyls (PBBs) and polybrominated diphenyl ethers (PBDEs).

This Standard was submitted by the Information Industry Products Pollution Prevention Working Group.

The China Electronics Standardization Laboratories accept responsibility for this Standard.

This Standard referenced the relevant IEC standard for the measurement of toxic substances in information industry products (IEC 62321,1-CD).

The bodies with primary responsibility for the drafting of this Standard were the China Saibao Laboratory/Ministry of Information Technology No. 5 Electronics Laboratory (Sections 1 to 4, 8 and Appendix, as well as editing of other parts), SGS Communications Standards Services Ltd (Sections 1 to 5 and 7), Shanghai Tianxiang Quality Technology Ltd (Sections 5 and 6), Agilent Scientific Ltd (Sections 6 and 7), Beijing Puni Physical and Chemical Analysis and Measurement Centre (Section 7), Shenzhen Hua Testing Technology Ltd (Sections 7 and 8), the Ministry of Information Technology Special Materials Testing Centre (Sections 7 and 8) and Huawei Technology Ltd (Appendix A) (arranged in section order).

The individuals principally concerned with the drafting of this Standard were Wang Xiaohan, Luo Daojun, Zhou Cuifeng and Xie Chengping (China Saibao Laboratory), Xue Jian (SGS Communications Standards Services), Gao Zixiang (Shanghai Tianxiang Quality Technology), Tu Zhuanhong and Chen Yuhong (Agilent Scientific), Li Shuili and Dun Bing (Shenzhen Hua Testing Technology), He Xiukun (Ministry of Information Technology Special Materials Testing Centre), Luo Chaoyun and Yang Rongxiang (Huawei Technology) and Song Wei (Beijing Puni Physical and Chemical Analysis and Measurement Centre) (arranged in order of principal drafting unit).

This Standard was issued on the … 2006.

The Electronic Products Pollution Prevention Standards Working Group of the Ministry of Information Technology is responsible for the interpretation of this Standard.

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Introduction

The widespread use of electronic information products has prompted people to be concerned about the effects of these products on the environment. This has led a number of countries around the world to introduce regulation covering the materials, energy use and disposal of electronic information products. In China, this resulted in six ministries and commissions including the Ministry of Information Industry drafting the *Method of Control and Management of Pollution from Electronic Information Products* and *Required Limits for Toxic Substances in Electronic Information Products*, which establish requirements for limits on six toxic substances in electronic information products: Pb, Cd, [Cr (VI)], Hg, PBBs and PBDEs.

With the issue of *Method of Control and Management of Pollution from Electronic Information Products* and *Required Limits for Toxic Substances in Electronic Information Products*, various laboratories and testing centres have developed protocols for testing for these toxic substances, but there has been no uniformity in the methods until now. This has led to the implementation of the *Method of Control and Management of Pollution from Electronic Information Products*, and in particular to specification of standards for the testing for the six toxic substances in electronic information products, Pb, Cd, [Cr (VI)], Hg, PBBs and PBDEs, in order to provide uniformity in the methods.

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Testing Methods for Regulated Substances in Electronic Information Products

1. Scope

This Standard specifies the method of testing for six toxic substances, namely lead (Pb), cadmium (Cd), hexavalent chromium (Cr VI), mercury (Hg), polybrominated biphenyls (PBBs) and polybrominated diphenyl ethers (PBDEs), in electronic information products.

This Standard applies to the electronic information products (EIPs) regulated by *Method of Control and Management of Pollution from Electronic Information Products.*

2. Normative references

The following document contains provisions which, through reference in this text, constitute provisions of this Standard. At the time of publication, the edition was valid. All standards are subject to revision, and users are encouraged to investigate the possibility of using the most recent editions of the standard indicated below.

SJxxxx-200x, *Required Limits for Toxic Substances in Electronic Information Products*

See Sections 5 and 8 for other cited documents.

3. Definitions of technical terms

The following terms and definitions are employed in this Standard.

3.1 Electronic Information Products

Electronic radar products, electronic communication products, television broadcasting products, computer products, domestic electronic appliances, electronic measuring instruments, specialized electronic products, electronic component products, electronic applications products and electronics materials products and combinations of such that are manufactured through the use of electronic technology

3.2 Dismountable unit

Machine, component, electronic instrument or assembly that can be dismounted through the use of normal tools

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Testing Methods for Regulated Substances in Electronic Information Products

3.3 Substance

Indicates a single chemical element or compound occurring in nature (For example, lead is a single chemical element, and lead oxide is a compound.)

3.4 Screening

A type of method of analysis in which the levels of elements are initially measured in the objec! to be tested

3.5 Polymeric materials

Synthetic or semi-synthetic organic concentrated material for optical fibre products either cast or extruded as thin films. Polymeric materials include polyvinyl, polyvinyl chloride, epoxy resins, polyamides, polycarbonates, ABS resins and polystyrene etc.

3.6 Metallic materials

Metallic materials are compounds of metallic elements, including iron and steel, the base metals and alloys. (Note: Metallic materials may be iron alloys, nickel alloys, tin alloys, aluminium alloys, magnesium alloys, copper alloys, zinc alloys or rare earth alloys etc.)

3.7 Special electronic materials

This indicates a number of special materials that are used in electronic information products, and refers to compounds of two or three or more of metallic materials, organic materials and inorganic non-metallic materials. These are used in circuit board substrates and conducting rubbers.

3.8 Inorganic non-metallic materials

These are materials consisting of oxides, carbonates, nitrates, chlorides, borides and silicates of elements, and aluminates, phosphates and borates. A general term for materials excluding polymer and metallic materials. Includes glasses and ceramics etc.

3.9 Matrix

Material or substance of the objec! to be analyzed, or includes the shape or form of the material or substance to be analyzed

3.10 Homogeneous material

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Indicates one or multiple substances composed of uniform materials; theoretically homogeneous materials cannot readily be distinguished from normal materials. Such materials can be divided by mechanical means (such as cutting, grinding shearing etc) to reveal the homogeneous nature of the parts or the material.

Other technical terms and definitions are defined under the specific sections dealing with testing methods.

4. General descrip!ion of testing methods

4.1 Contents of test methods

The test methods described in the following sections consist of the seven parts below:
- Scope
- Normative references
- Technical terms and definitions
- Instruments and equipment
- Reagents
- Chemical sampling
- Measurement procedures

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4.2 Flowchart of test methods

Figure 1 illustrates the sequence of testing methods for controlled substances in electronic information products.
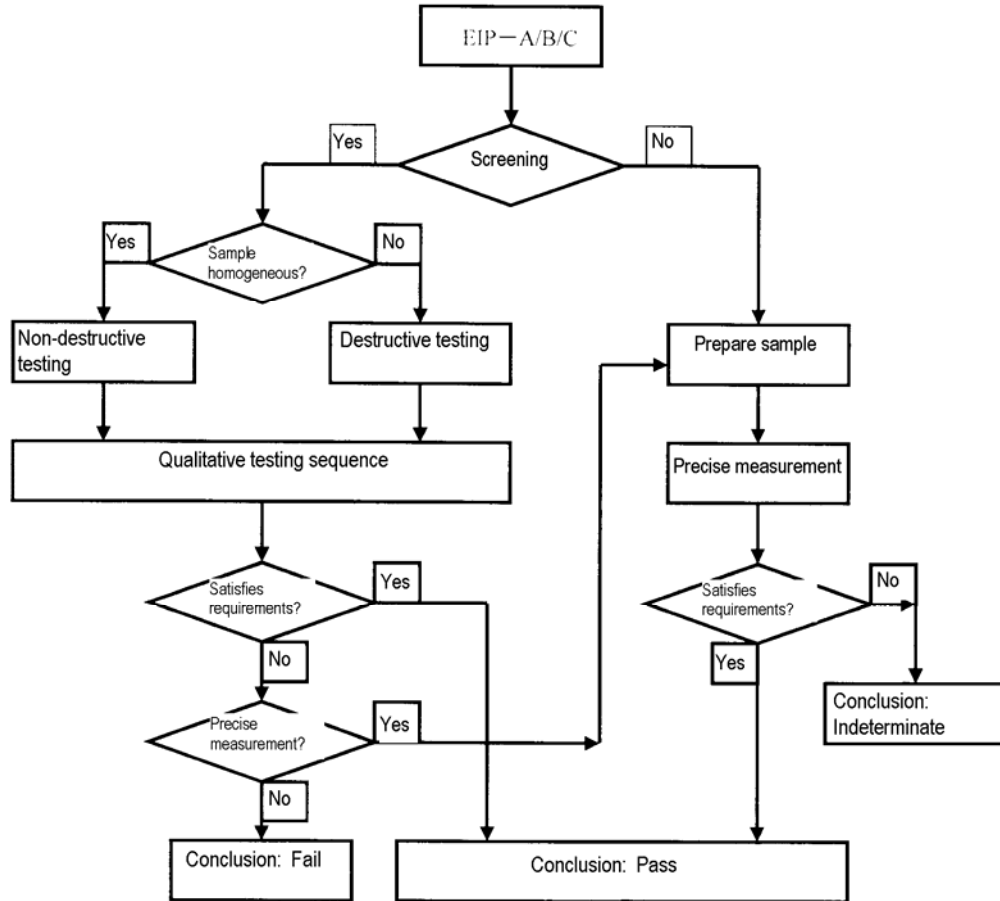
Figure 1 Flowchart of testing methods

4.3 Screening test method

The screening test methods make use of energy-dispersive X-ray fluorescence (EDXRF) and wavelength-dispersive X-ray fluorescence (WDXRF) instruments to perform the initial qualitative testing step for the target substances in the sample. The screening tests can directly measure the sample (in which the samples remain whole), or can involve crushing the sample to form a homogeneous substance (samples pulverized mechanically) which is then measured. Where there are very many typical and homogeneous samples (such as plastics, alloys or glass etc), the samples may be sent directly to screening testing without being pulverized, while composite material samples (such as printed circuit boards) that consist of many different types of compounds should normally be prepared by being mechanically pulverized. Mechanical pulverizing to prepare samples is broadly applicable to screening testing, and is also applicable to precise measurement methods. Refer to

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Appendix A for full details of the procedures and steps involved in the preparation of mechanically pulverized samples.

XRF spectrometers (EDXRF or WDXRF) can be used for qualitative screening tests of samples. Chapter 5 of this Standard describes the method of screening using XRF spectrometers. While XRF spectrometers provide a fast and economical means of analysis, there are limits to the scope and application of the results obtained with the method of analysis.

There are three basic categories of samples in qualitative screening:
- Pass (white zone): The levels contained in the sample are clearly below the limits.
- Fail (black zone): The levels contained in the sample are clearly above the limits.
- Indeterminate (grey zone): The levels in the sample are close to the limits, and therefore are neither clearly pass or fail, and require further testing.

Samples that have been found to have passed qualitative screening meet the requirements; samples that have failed do not meet the requirements, and samples that are found to be indeterminate must undergo the following further precise measurement procedures in order to determine whether or not the samples meet the requirements.

4.4 Precise measurement method

The precise measurement method involves a number of testing methods for the levels of restricted materials in organic materials, metallic materials, inorganic non-metallic materials and special electronic materials. After a sample has undergone precise measurement testing, it is possible to determine whether or not it has reached the limits in the Standards for restricted substances.

Table 1 Simplified overview of the precise measurement method

Procedure	Substance	Metallic materials	Polymer materials	Inorganic non-metallic materials	Special electronic materials
Mechanical sample preparation method	-	Direct pul-verization and meas-urement	Direct pul-verization and measurement	Pulveriz-ation	Pulverization
Preparation of chemical samples	-	Dissolve in acid	Dissolve with micro-waves Dissolve in acid Dry burn method Solvent extraction	Dissolve with microwaves Dissolve in acid Solvent extraction	Dissolve with microwaves Dissolve in acid Solvent extraction

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Methods of analysis	PBB/ PBDE	No	Gas chromatography – Mass spectrometry (6)	No	Gas chromatography – Mass spectrometry (6)
	Pb/Cd	Inductively coupled plasma atomic radiation spectrum method, Inductively coupled plasma mass spectrometry, Atomic absorption spectrometry (7.1)			
	Hg	Inductively coupled plasma atomic radiation spectrum method, Inductively coupled plasma mass spectrometry, Cold vapour atomic absorption spectrometry, Atomic fluorescence spectrometry (7.1)			
	Cr (VI)	Coating development method (8.1), Alkali digestion – colorimetry (8.2)	Alkali digestion – colorimetry (8.2)	Alkali digestion – colorimetry (8.2)	Alkali digestion – colorimetry (8.2)

5. Method of screening measurement of toxic substances in electronic information products by the use of the X-ray fluorometer

5.1 Scope

This section specifies the method for the screening of electronic information products for the following toxic materials, elemental lead, cadmium, mercury, chromium and bromine by X-ray fluorimetry.

This method is used to measure elemental lead, cadmium, mercury, chromium and bromine in parts and materials of samples of electronic information products prepared according to the requirements of Appendix A.

5.2 Normative references

GB/T 16597-1996   *Method of Analysis of Metallurgical Products*, *X-ray Fluorescence Spectrum Measurement*

5.3 Terms and definitions

5.3.1 X-ray fluorescence spectrum (XRF)

A type of relative analytical technique. X-ray or low power light-emitting samples are caused to emit characteristic X-rays under strictly controlled conditions. The characteristic X-rays correspond to specific elements, and the concentrations of the elements in the samples directly determine the strength of the X-ray signals. The process of emitting characteristic X-ray signals is known as X-ray fluorescence.

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5.3.2 Wavelength-dispersive X-ray fluorescence (WDXRF)

The atoms of the elements to be measured are subjected to high energy radiation and stimulated to cause the inner layer of electrons to transition, at the same time emitting X-rays at a characteristic wavelength. The elements to be analyzed are measured qualitatively and quantitatively from the wavelength and strength of the spectrum.

5.3.3 Energy-dispersive X-ray fluorescence (EDXRF)

The atoms of the elements to be measured are subjected to high energy radiation and stimulated to cause the inner layer of electrons to transition, at the same time they emit X-rays of fixed energy levels. An X-ray detector with a fixed energy level resolution is used to detect the X-rays at characteristic energy levels for the individual elements in the sample to be analyzed; the elements are analyzed are measured qualitatively and quantitatively from the relative energy levels of the signals that are output from the detector.

5.3.4 Background

Continuous spectrum that overlies the analyte lines, caused principally by the scattering of the incident radiation by the sample

5.3.5 Analyte lines

Characteristic spectrum lines for the content of an element being analyzed for the strength required to measure and distinguish the element. In X-ray fluorescence, the analyte lines are generally the high strength, low interference low background spectrum lines.

5.3.6 Limit of detection

Minimum amount of contained material that can be detected to a specified level of confidence

5.3.7 Interference lines

Overlying or partially overlying spectrum lines that therefore affect the definite measurement of the strength of analyte lines

5.3.8 Matrix effects

Effects on the strength of the X-ray fluorescence of the chemical composition or physical – chemical state of the sample; principally manifested as increased absorption effects, particle size effects, surface fineness effects and chemical state effects etc

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5.3.9 Test portion for calibration curve

Sample of known composition and levels of contents used in order to yield a calibration curve or for calibration

5.3.10 Test portion for standardization of instrument

Sample used for the calibration of drift in instruments. The basic requirements for a standardization sample are that the elemental analyte lines should be of a suitable strength and should remain constant over an extended period of time.

5.4 Instruments and equipment

5.4.1 X-ray fluorometer (wavelength dispersive X-ray fluorescence (WDXRF) or energy dispersive X-ray fluorescence (EDXRF): Consist principally of a stimulation source, a probe, a sample chamber and a digital analyzer

5.4.2 Ancillary equipment: The usual ancillary equipment consists of an automatic sampling device, a sample cutting device, a grinder, a pulverizer, a homogenizer, a sample compacting device and a furnace that must meet requirements when necessary.

5.4.3 Selection of parameters: Recommended X-ray analyte lines

Element	First analyte line	Second analyte line	Organic sample analyte line	Metallic analyte line
Lead	Lβ	Lα	Lα	Lβ
Cadmium	Kα	-	Kβ	Kα
Mercury	Lα	-	Lα	Lα
Chromium	Kα	-	Kα	Kα
Bromine	Kα	Kβ	Kα	Kα

5.5 Reagents

5.5.1 Boracic acid (HBO$_3$): Analytical purity, heated at 105° C for 1 hour and placed in a dry container

5.5.2 Standard samples containing the five elements, lead, cadmium, mercury, chromium and bromine

5.6 Preparation of samples

There are very strict requirements for samples for analysis by X-ray fluorescence; these relate to the different forms of the samples.

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5.6.1 Homogeneous solid samples

Test samples of fixed dimensions may be formed through the use of cutting and grinding devices from samples of solid, sheet or cast samples of irregular shapes. The surface that is irradiated must be representative of the entire sample.

5.6.2 Film samples

When preparing test samples from thin film samples, great care must be exercised to ensure the consistency and homogeneity of structure of the samples. When measuring thin film, spread it out smoothly; a backing material may be used to support the film, but the backing material should be selected for a low background effect.

5.6.3 Special electronic materials

Special electronic materials are generally not homogeneous. Samples may be cut with cutting equipment and cut into small pieces and crushed; they are then powdered with a grinder and are formed into powder not exceeding 1 mm in size. This is mixed uniformly and then boracic acid is used to coat a sample slide to a thickness not exceeding 1 mm.

5.6.4 Liquid samples

When testing liquid samples, take a specified amount of the liquid and place it in a suitable container. When making the measurements, ensure that the liquid is not agitated, does not spill, and does not form bubbles or form a sediment etc. Liquid samples may also be dripped onto suitable media (such as filter papers) and may then be dried and measured.

5.6.5 Contamination of samples

Contamination is the cause of error in analysing samples. In X-ray fluorimetry, particular care is required to prevent the contamination of the surface of the sample. In preparing samples for X-ray fluorimetry, pay particular attention to the following avenues for contamination:
a) Contamination from materials from pulverizing and grinding devices
b) Contamination from the vessels used in dissolving and melting samples
c) Contamination from the working environment in the laboratory
d) Contamination by reagents
e) Contamination from the hands when samples are touched
f) Contamination from inner backing materials
g) Contamination when samples are pressure moulded

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5.7 Measurement procedures

5.7.1 Preparation of the instruments

Install the instrument according to the manufacturer's instructions; the instrument must be capable of operating continuously and maintaining optimum consistency.

5.7.2 Preparation of standard curves

Take sets of samples of different known concentrations (not less than three concentrations) of each of the five elements, lead (Pb), cadmium (Cd), mercury (Hg), chromium (Cr) and bromine (Br), and place them in the instrument sample chamber. Measure the compositions of the samples for the times recommended by the manufacturer, measuring each concentration of each element at least four times, and then take the average of four measurements as the spectrum line strength. Finally, prepare the standard curves from the spectrum line strengths and concentrations. The standard curves may be prepared automatically is a computerized analyzer is available.

5.7.3 Calibration and checking

Before each analysis of a sample, perform a calibration and check to check the correctness of the standard curves for each of the five contained elements, lead (Pb), cadmium (Cd), mercury (Hg), chromium (Cr) and bromine (Br).

5.7.4 Measurement of samples

Place the properly prepared sample in the sample chamber, and perform X-ray analysis on the sample for the same time as was used for the preparation of the standard curve. Measure each sample at least twice and take the average value as the spectrum line strength.

5.7.5 Analysis of results

5.7.5.1 Calculation of results

Input the measurements of the spectrum line strengths for the five elements into the standard curve equation to calculate the concentrations of the elements in the samples.

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5.7.5.2 Screening elements

The results of the screening measurements of the restricted substances in the X-ray fluorescence testing of electronic information products yield three states:

Pass (P): The sample measurements pass if the values for all elements are below the specified minimum value.

Fail (F): The sample measurements fail if the value for any one element is above the specified maximum value.

Indeterminate (X): The sample measurements are indeterminate, and more precise measurement is required, if the values for the elements are between the specified minimum and maximum values.

Table 2 Screening limits for different elements in different electronic information product samples

Element	Polymer materials	Metallic materials	Inorganic non-metallic materials	Special electronic materials
Cadmium	P≤70<X<130F	P≤70<X<130F	P≤70<X<130F	P≤70<X<130F
Lead	P≤70<X<130F	P≤70<X<130F	P≤70<X<130F	P≤70<X<130F
Mercury	P≤70<X<130F	P≤70<X<130F	P≤70<X<130F	P≤70<X<130F
Bromine	P≤300<X	-	-	P≤250<X
Chromium	P≤700<X	P≤700<X	P≤700<X	P≤500<X

5.7.6 Application[1]

While the X-ray fluorescence spectrometer is a fast, non-destructive and convenient instrument, it has limitations in regard to XRF; when using this instrument give careful consideration to the applicability of the results obtained. X-ray fluorescence analysis can provide information as to whether an element is present or not. Thus special care is required if the test results are *positive* for the presence of chromium or bromine, as it is not certain that the sample contains the toxic hexavalent chromium or polybrominated biphenyls. On the other hand, if the test results are *negative*, it is possible to say that the sample does not contain hexavalent chromium or polybrominated biphenyls. When analysing samples consisting of different materials, such as samples with coated plastic surfaces, operators should consider the effects on sensitivity of the thickness of the film when performing X-ray fluorescence testing on the coated surface; it is also necessary to consider whether the X-rays will be able to penetrate the thin film sample and reach the substrate, causing error in the results.

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[1] Section numbering repeated

5.7.6 Precision

5.7.6.1 Repeatability

The variation in the results when the same sample is tested by the same operator on the same instrument under the specified conditions must not exceed the value indicated by formula (1):

$$R = 0.029(S + 0.6) \qquad \qquad \dots (1)$$

Where,

S: Average value from the two measurements

5.7.6.2 Reproducibility

The variation in the two results when the same sample is tested by a different operator by the same method in a different laboratory must not exceed the value indicated by formula (2):

$$R = 0.063(S + 0.6) \qquad \qquad \dots (2)$$

Where,

S: Average value from the two measurements

5.7.7 Reporting of measurements

Report the average of the two measurements in terms of mg/kg.

6 Testing Methods for PBBs and PBDEs in Electronic Information Products

6.1 Scope

This document specifies the procedure for the determination of content of PBBs and PBDEs in Electronic Information Products by gas chromatography/mass spectrometry (GC/MS).

This method is suitable for the determination of content of 100-20000 mg/Kg PBBs and PBDEs in polymer materials, special electronic materials from Electronic Information Products. Sample with even lower concentration can be tested if suitable concentrating and purifying steps are carried out.

6.2 Normative References

ASTM D3421 Recommended Practice for Extraction and Determination of Plasticizer Mixtures from Vinyl Chloride Plastics (Withdrawn 1987).

EPA 3540C Soxhlet Extraction

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6.3 Terms and Definitions

None

6.4 Apparatus/Equipment

6.4.1 Apparatus

a) Laboratory fume hood
b) Balance (precision 1 mg)
c) Glassware
d) Soxhlet apparatus with condenser
e) Heating device to fit the round flask adopted at soxhlet apparatus
f) Crushing device: include equipments such as cutter or scissors, grinder, cryogenic grinding device, mainly for sample reduction
g) Furnace (400° C or higher)
h) Oven (capable of maintaining a constant temperature (105° C – 250° C)

i) 18 # standard screen.

6.4.2 Equipment

a) Gas chromatography – temperature programmable with all the necessary accessories such as injector (either manual or automatic) and capillary column.

b) Capillary column: 10 ~ 30 m (L) × 0.25 mm (ID) × 0.1 μm (film thickness), fused silica column (DB-5 or equivalent);
c) Mass spectrometry: be capable of generating 70 eV electron for analyte ionization. The scan range shall cover 50m/z~1000.0m/z.
d) Data analysis system: be capable of collecting, recording, storing, and processing MS data.

6.5 Reagents
a) Solvents for extraction: acetone, toluene, cyclohexane, normal hexane, methanol, dichloromethane, isooctane and nonane
b) Standard stock solution (1000 mg/L): can be either prepared using pure standard materials or purchased from concerned agencies. The mixed standard isomer solution of common PBBs or PBDEs also can be purchased from relevant suppliers.
c) Calibration standard: prepare at least 5 calibration standards of various concentrations with the lowest concentration being slightly higher than the detection limit of this method. The other four concentrations shall fall into the range of actual samples.
d) Internal standard solution: Labelled 13C12 BDEs or 4,4'-dibromoonctafluro biphenyl (f) or Penathrene D10 as recovery standard for the analysis of PBDEs. Labelled 13C12 PBB or 4,4'-dibromoonctafluro biphenyl (f) or Penathrene D10 as recovery standard for the analysis of PBBs).
e) Liquid nitrogen: industrial grade

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6.6 Sample Preparation

6.6.1 Sample Reduction

Disassemble the electronic information products into various material samples according to annex A. Cut the samples into small pieces of 0.1 cm×0.1 cm using scissors or cutter. After being cooled down, grind the samples to a size of no more than 1.0 mm by a miller (or in an equivalent manner) and then mix them well.

6.6.2 Extraction of Sample solution

Take 0.1 g - 0.2 g of above well mixed sample and measure its weight accurately to 0.0001 g. Transfer it into a cellulose mantle and placed in the Soxhlet extractor. Add 50 ml-200 ml of extracting toluene or other solvents (6.5 a) as well as the internal standard solution (6.5 d), followed by 1-2 grains of zeolite. Complete the Installation of the Soxhlet extractor and heat for 4-24 hours for extracting. After cool the solution down, transfer it to an appropriate volumetric flask and fill the flask up with water to the mark. The solution may be used in test steps directly. If the concentration of the determined substance in the sample is above the range of the concentration calibration curve, appropriate dilution is necessary.

6.7 Test Procedure

6.7.1 Gas chromatographic conditions

a) Injection port temperature: 250° C - 320°C
b) Initial temperature and holding time of column oven: 100° C, held for 1-3min
c) Temperature programming conditions of column temperature: temperature programmed from 100° C - 320° C at 5° C - 20°C/min and constant for 5 min;
d) Carrier gas: Helium, flow rate: 1.2 ml/min;
e) GC/MS interface temperature: 320° C;
f) Injection mode: (pulse) splitless injection;
g) Sample size: 1 µl-2 µl.

6.7.2 Mass spectrometry conditions

a) Ionizaton: EI
b) Electron energy: 70eV;
c) Ion source temperature: 250-300°C; mass separator temperature: 150°C (recommended);
d) Resolution: larger than 800 (preferably larger than 1000);
e) Analysis mode: Selected ion monitoring (SIM), ions monitored are shown in Table 3 and Table 4, respectively.

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6.7.3 Sample Analysis

a) Power on the instrument and let the instrument stabilize;

b) Calibrate the instrument using instrument calibration standards according to the requirement of the instrument;

c) Develop standard curve: inject standards of various concentrations (6.5 c) into GC/MS in turn. Develop the standard curve of peak area vs concentration of the standards;

d) Sample determination: inject sample extracts of the same size in standard curve developing into GC/MS, and determine the PBBs and PBDEs according to the ion peaks given in below Table 3 and Table 4. Calculate the corresponding PBB and PBDE contents on standard curve by the peak areas.

Table 3 Molecular weight and Identification ion peaks of PBB

Number	Chemical name	Molecular formula	Molecular weight	Quantitative ion	Identification ion peaks		
1	Mono-BBs	$C_{12}H_9Br$	233.1	234.0	236.0	232.0	152.2
2	Di-BBs	$C_{12}H_8Br_2$	312.0	312.0	314.0	310.0	152.2
3	Tri-BBs	$C_{12}H_7Br_3$	390.9	391.8	393.8	389.8	230.0
4	Tetra-BBs	$C_{12}H_6Br_4$	469.8	469.8	467.8	309.9	307.9
5	Penta-BBs	$C_{12}H_5Br_5$	548.7	549.6	547.6	389.8	387.8
6	Hexa-BBs	$C_{12}H_4Br_6$	627.6	627.6	625.6	467.7	465.7
7	Hepta-BBs	$C_{12}H_3Br_7$	706.5	705.5	703.5	545.6	543.6
8	Octa-BBs	$C_{12}H_2Br_8$	785.4	785.4	783.4	625.5	627.5
9	Nona-BBs	$C_{12}HBr_9$	864.3	703.4	863.2	705.4	703.4
10	Deca-BBs	$C_{12}Br_{10}$	943.2	781.3	943.2	783.3	781.3

Table 4 Molecular weight and Identification ion peaks of PBDE

Number	Chemical name	Molecular formula	Molecular weight	Quantitative ion	Identification ion peaks		
1	Mono-BDEs	$C_{12}H_9BrO$	249.1	250.0	252.0	248.0	141.0
2	Di-BDEs	$C_{12}H_8Br_2O$	328.0	328.0	330.0	325.9	168.0
3	Tri-BDEs	$C_{12}H_7Br_3O$	406.9	405.8	403.8	246.0	123.0
4	Tetra-BDEs	$C_{12}H_6Br_4O$	485.8	485.7	483.7	325.9	162.9
5	Penta-BDEs	$C_{12}H_5Br_5O$	564.7	564.6	562.6	403.8	201.9
6	Hexa-BDEs	$C_{12}H_4Br_6O$	643.6	483.7	643.5	483.7	241.9
7	Hepta-BDEs	$C_{12}H_3Br_7O$	722.5	561.6	721.5	561.6	563.6
8	Octa-BDEs	$C_{12}H_2Br_8O$	801.4	641.5	801.4	641.5	320.8
9	Nona-BDEs	$C_{12}HBr_9O$	880.3	719.4	881.3	719.4	360.7
10	Deca-BDEs	$C_{12}Br_{10}O$	959.2	799.3	959.2	799.3	400.3

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6.7.4 Calculation of Analytical Results

Polybrominated Biphenyl (PBB) and Polybrominated Diphenyl Ether (PBDE) contents in the sample are calculated from the equation:

$$X_i = \frac{C_i \times V_i \times d}{m_i} \times 10^6$$

(3)

Where:

X_i— PBB and PDBE i contents in the sample in ppm (mg/kg);

C_i - PBB and PDBE i contents in the sample extracts in mg/L;

V_i - Volumetric size of n the sample extracts in L;

d - Dilution rate of the sample extracts;

m_i - Sample size in mg

6.7.5 Precision

The absolute deviation of two results from duplicate testing shall not exceed 20% of the arithmetic average.

7 Testing Methods for Lead (Pb), Cadmium (Cd) and Mercury (Hg) in Electronic Information Products

7.1 Testing Methods for Lead (Pb) and Cadmium (Cd) in Electronic Information Products

7.1.1 Scope

This document specifies the procedure for the determination of content of Lead (Pb) and Cadmium (Cd) in Electronic Information Products.

This method is suitable for determination of content of Lead (Pb) and Cadmium (Cd) in polymer materials, metal materials, special electronic materials and inorganic non-metal materials.

7.1.2 Summary of Method

An appropriate mass of cryogenically milled and homogenized sample is digested in an acid solution under fixed temperature or pressure conditions. The elements lead (Pb) and cadmium (Cd) in the Digestion solution are determined by Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP - AES), Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) or Atomic Absorption Spectrometer (AAS).

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7.1.3 Normative References

GB/T 15337-1994 General rules for atomic absorption spectrometric analysis

GB/T 10725-1989 Chemical reagent General rules for inductively coupled plasma atomic emission spectrometry

GB/T 3260.9-2000 Methods for chemical analysis of tin Determination of lead, copper and zinc content

GB/T 5121.3-1996 Copper and copper alloys-Determination of lead content

GB/T 8647.6-88 Nickel-Determination of cadmium、cobalt、copper、

manganese、lead and zinc contents-Flame atomic absorption spectrophotometric method

GB/T 15555.1-1995 Solid waste—Determination of total mercury—Cold atomic absorption spectrometry

ISO 17294-1 Water quality - Application of inductively coupled plasma mass spectrometry (ICP-MS) - Part 1: General guidelines

EPA 3050 Acid Digestion of Sediments, Sludges and Soils

EPA 3052 Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices

EPA 7000 Series Method for determining Pb, Cd, Cr and Hg

Battery Industry (EPBA, BAJ and NEMA), 1998, Standard Analytical Method for the Determination of Mercury, Cadmium and Lead in Alkaline Manganese Cells using AAS, ICP-AES and "Cold Vapor"

7.1.4 Terms and Definitions

 a) ICP-AES (-OES), Inductively coupled plasma atomic emission spectroscopy

Method of determining the target element contained in the sample by means of atomization and ionization of the sample with high-frequency plasma. The energy emitted by excited atoms or ions is measured. The wavelengths of the emitted energy are specific to the elements present in the sample.

b) ICP-MS, Inductively Coupled Plasma Mass Spectroscopy:

Method of determining the target element contained in the sample by means of ionizing the sample with high-frequency plasma. The generated ion is measured with mass spectrometer for the number of ions in mass-to-charge ratio (m/z) of the target element for analysis of the element or its isotope.

c) AAS, Atomic Absorption Spectrometry:

Method of determining the chemical elements content in the sample by means of converting the target element into free atom through flame or chemical reaction and measuring the characteristic electromagnetic absorption by the ground state atom of the element in vapour phase.

Testing Methods for Regulated Substances in Electronic Information Products

7.1.5 Apparatus and Equipment

a) Atomic Absorption Spectrometer (AAS)

b) Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP-AES/OES)

c) Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

d) Various Glass ware for laboratory

e) Heating device

f) Muffle furnace

g) Microwave digestion system equipped with Polytetrafluoroethylene - tetrafluoroethylene high pressure digestion vessel

h) Electronic analytical balance capable of accurate weighing to 0.1mg

i) Crucible, 50ml or 150ml

j) Hydrofluoric acid resistant holder

k) Bunsen burner, or similar type of gas burner

7.1.6 Reagents

Unless otherwise stated, only approved high purity reagents and 18 MΩ ASTM Type 1 water or equivalent purity water shall be used.

a) Nitric acid: ρ = approx. 1.4g/ml, 65%

b) Hydrochloric acid: ρ = approx. 1.16g/ml, 37%

c) Hydrogen peroxide: ρ = approx. 1.10g/ml, 30%

d) Sulphuric acid: ρ = approx. 1.84g/ml, 95%

e) Hydrofluoric acid: ρ = approx. ISO 40 ≈ 42%; JIS 46 ≈ 48%

f) Hydrobromic acid: ρ = approx. 1.48 g/ml, 47 ~ 49%

g) Perchloric acid: ρ = approx. 1.67 g/ml, 70%

h) Phosphoric acid: ρ = approx .1.69 g/ml, above 85%

i) Boracic acid

j) Mixed acid 1 (hydrochloric acid: nitric acid, 3:1)

k) Mixed acid 2 (hydrochloric acid: hydrogen peroxide, 3:1)

k) Mixed acid 3 (nitric acid: hydrofluoric acid, 1:3)

l) Mixed acid 4 (hydrofluoric acid: 1nitric acid, 2:2)

m) Lead standard solution, 1000 μg/ml

n) Cadmium standard solution, 1000 μg/ml

o) Scandium standard solution, 1000 μg/ml

p) Yttrium standard solution, 1000 μg/ml

q) Rhodium standard solution, 1000 μg/ml

7.1.7 Sample Preparation

7.1.7.1 Sample Reduction

Disassemble the electronic information products into the various material

samples according to annex A. Cut the samples into small pieces of 0.1cm×0.1cm using a shear or cutting machine (or in other manners). Metal materials and inorganic non-metal materials can be used directly in the next step, while polymer materials and special electronic materials require to be pulverized into particles or powder no more than 1mm in diameter and mixed uniformly for the next step.

7.1.7.2 Preparation of Metal Material Sample

a) Direct dissolving

Take about 0.1 g to 0.2 g of sample into a beaker and measure its weight accurately to 0.0001 g. Add appropriate portion of acid (or suitable proportion of mixed acid) according to the metal material matrix, and heat the beaker and its contents until sample is dissolved completely.

In general, hydrochloric acid, nitric acid or their mixed solutions are recommended. Use for example hydrofluoric acid, perchloric acid or sulphuric acid for further dissolving when the sample is found indigestible. A PTFE/PFA beaker is required in case that hydrofluoric acid is used.

Note: There may be some precipitate (lead sulphate, barium sulphate, silver chloride, alumina or aluminium hydroxide etc.) during sample dissolution. Therefore, it shall be determined that the selected dissolving method will not result in the loss of target element. Another method is required for sample digestion (for example, method of alkali fusion or sealed pressure vessel) if target substances are included in the residues.

1) General Alloy Method

Take about 0.20 g of sample into a beaker and measure its weight accurately to 0.0001 g. Add 10ml of mixed acid solution of hydrochloric acid and nitric acid slowly (typical ratio of hydrochloric acid: nitric acid is 3:1, depending on the composition of matrix materials). Heat the beaker and its contents under low temperature until sample dissolves completely. Gradually cool the solution to room temperature, transfer the contents to a 50 ml volumetric flask. Then fill the flask up with water to the mark. Dilute the sample to desired concentration according to the method used.

It is necessary to add another 1ml of hydrofluoric acid in order to assure a complete digestion of the sample when the sample contains Si, Zr, Hf, Ti, Ta, Nb or W (See Section 5 'Screening Tests').

Execute procedure identical to preparation of test sample solution concurrently without sample.

2) Tin Alloy Method

Take about 0.20 g of sample into a beaker and measure its weight accurately to 0.0001 g. Add 10ml of mixed acid solution of hydrochloric acid and nitric acid slowly (typical ratio of hydrochloric acid: nitric acid is 3:1, depending on the composition of matrix materials). Cover the beaker with watch glass to allow the violent reaction to be finished, heat the beaker slightly until its contents until sample is dissolved completely. Allow the beaker be cooled down for a while, remove the watch glass. Add 10 ml of sulphuric acid along the beaker wall and heat the beaker until white fume of SO_3 is generated. Allow the sample be cooled down. Add 20 ml of hydrobromic acid along the beaker wall and mix the contents well. Then heat the beaker until white fume of SO_3 is generated. Repeat above steps for 3 times. Allow the beaker and its contents to be cooled to ambient temperature. Add 10 ml of nitric acid to dissolve salts. After cooling, transfer the solution to a 50 ml volumetric flask and then fill the flask up with water to 50 ml. Dilute the sample to desired concentration according to the method used.

Or, take about 0.20 g of sample and add 10ml of hydrochloric acid and hydrogen peroxide to dissolve the sample (typical ratio of hydrochloric acid: hydrogen peroxide, 3: 1, depending on the composition of matrix materials).

It is necessary to add another 1ml of hydrofluoric acid in order to assure a complete digestion of the sample when the sample contains Si, Zr, Hf, Ti, Ta, Nb, W (See Section 5 'Screening Tests').

Execute procedure identical to preparation of test sample solution concurrently without sample.

b) Microwave digestion (mainly for insoluble alloys)

Take about 0.10 g of sample and measure its weight accurately to 0.0001 g. Transfer it into a PTFE/PFA digestion vessel, and add 5 ml of mixed acid solution of hydrochloric acid and nitric acid with appropriate proportion (typical ratio of hydrochloric acid and nitric acid, 3: 1, depending on the composition of matrix materials). It is necessary to add another 1ml of concentrated hydrofluoric acid in order to assure a complete digestion of the sample when the sample contains Si, Zr, Hf, Ti, Ta, Nb, W (See Section 5 'Screening Tests').

After a period of reaction of the sample, put the whole vessel in a microwave digestion device. Digest the sample according to the operation instruction until it is dissolved completely. Take out the digestion vessel, and after the vessel is cooled down to room temperature, add proper amount of boric acid to permit the complexing of excessive hydrofluoric acid (boracic acid is not required in case that a hydrofluoric acid resistant atomizer is used). Transfer

the solution to a 50ml volumetric flask and fill the flask up with water to 50ml. Dilute the sample to desired concentration according to the method used.

Execute procedure identical to preparation of test sample solution concurrently without sample.

7.1.7.3 Preparation of Inorganic non-Metal Material

Hydrofluoric acid resistant vessels must be used, because some non–metal elements such as silicon are typically contained in inorganic non–metal samples and extremely corrosive hydrofluoric acid is required during the preparation of sample.

Take 0.10 of sample and measure its weight accurately to 0.0001 g. Transfer it into a PTFE/PFA digestion vessel, and add 3 ml of mixed acid solution of concentrated hydrochloric acid and concentrated nitric acid with appropriate proportion (typical ratio of hydrochloric acid and nitric acid, 3: 1) and 3 ml of concentrated hydrofluoric acid. After a period of reaction of the sample, place the whole vessel in a microwave digestion device. Digest the sample according to the operation instruction until it is dissolved completely. Take out the digestion vessel, and after the vessel is cooled down to room temperature, add proper amount of boric acid to permit the complexing of excessive hydrofluoric acid (boracic acid is not required in case that a hydrofluoric acid resistant atomizer is used). Transfer the solution to a 50 ml volumetric flask and fill the flask up with water to 50 ml. Dilute the sample to desired concentration according to the method used.

Execute procedure identical to preparation of test sample solution concurrently without sample.

7.1.7.4 Preparation of Polymer Material Sample

a) Dry Ash method

1) When the sample does not contain halogen compounds (See Section 5 'Screening Tests'):

Take 0.20 g of sample into a beaker and measure its weight accurately to 0.0001 g. Mount the crucible in the hole in the heat resistant thermal insulation board and then heat it gently. When the sample has decomposed to a charred mass, heating is gradually increased until the volatile decomposition products have been substantially expelled and a dry carbonaceous residue remains. Then transfer the crucible and its contents to the muffle furnace at 450 ± 25 °C, with the door left slightly open to provide sufficient air to oxidize the carbon. Heating is continued until the carbon is completely oxidized and a clean ash is obtained. Then remove the crucible

and its contents from the furnace and allow it to be cooled to ambient temperature. Add 5 ml of nitric acid, and transfer the resulting solution to a 50 ml volumetric flask and fill the flask with water to 50 ml. Dilute the sample to desired concentration according to the method used.

Execute procedure identical to preparation of test sample solution concurrently without sample.

2) When the sample contains halogen compounds (See Section 5 'Screening Tests'):

Take 0.20 g of sample into a crucible and measure its weight accurately to 0.0001 g. Add 10 ml to 15 ml of sulphuric acid and heat the crucible and its contents slowly on a hot plate or sand bath until the plastic melts and blackens. Then add 5 ml of nitric acid and heating is continued until the plastic degrades completely and white fumes are generated. After cooling, place the crucible in a muffle furnace maintained at 450° ± 25° C and make the sample evaporated, dried, and burnt until the carbon has been completely incinerated. Then remove the crucible and its contents from the furnace and allow them to be cooled to ambient temperature. Add 5 ml of nitric acid, and transfer the resulting solution to a 50 ml volumetric flask and fill the flask with water to 50 ml. Dilute the sample to desired concentration according to the method used.

Execute procedure identical to preparation of test sample solution concurrently without sample.

b)   Wet Acid Digestion Method

This method is used to determine cadmium only. It is not suited for determining lead, because the use of sulphuric acid can lead to a loss of lead in the sample due to the formation of lead sulphate.

1) General Dissolving Method

Take 0.20 g of sample into a flask and measure its weight accurately to 0.0001 g. Add 5 ml of sulphuric acid and 1ml of nitric acid, and heat the flask and its contents until the sample is reduced to ash and white fumes are generated. Stop heating and add nitric acid in small quantities (approx. 0.5 ml), and resume heating until white fumes are generated. Repeat above heating and decomposition with nitric acid until the decomposed solution turns pale yellow.

Then allow the sample to be cooled down for several minutes. Add hydrogen peroxide in small quantities, several milliliters at a time, and heat the sample once again until white fumes are generated. After cooling, transfer the

solution to a 50ml volumetric flask and fill the flask up with water to the mark. Dilute the sample to desired concentration according to the method used.

Execute procedure identical to preparation of test sample solution concurrently without sample.

2) When general digestion is inadequate or when the sample contains silica, titanium, etc. (See Section 5 'Screening Tests'):

Take 0.20 g of sample into a flask and measure its weight accurately to 0.0001 g. Add 5 ml of sulphuric acid and 1ml nitric of acid, and heat the flask and its contents until the sample is reduced to ash and white fumes are generated. Stop heating, add nitric acid in small quantities (approx. 0.5 ml) and resume heating until white fumes are generated. Repeat the above heating and decomposition with nitric acid until the decomposed solution turns pale yellow.

Then allow the sample to be cooled down for several minutes. Add hydrogen peroxide in small quantities, several milliliters at a time, and heat the sample once again until white fumes are generated. After cooling, transfer the solution to a fluorocarbon resin vessel. Add 5 ml of hydrofluoric acid and heat the vessel until white fumes are generated. Then remove the digestion vessel and its contents from the furnace and allow them to be cooled to ambient temperature. Add the proper amount of boric acid to permit the complexing of excessive hydrofluoric acid (boracic acid is not required in case that a hydrofluoric acid resistant atomizer is used). Transfer the solution to a 50 ml volumetric flask and fill the flask up with water to 50 ml. Dilute the sample to desired concentration according to the method used.

Execute procedure identical to preparation of test sample solution concurrently without sample.

c) Microwave digestion

Take 0.10 g of sample and measure its weight accurately to 0.0001 g. Transfer it into a PTFE/PFA digestion vessel, and add 5 ml of nitric acid and 1 ml of hydrogen peroxide. Add another 1 ml of concentrate hydrofluoric acid when sample contains Si, Zr, Hf, Ti, Ta, Nb, W (See Section 5 'Screening Tests'). Put the whole vessel in a microwave digestion device. Digest the sample according to the operation instruction until it is dissolved completely. Take out the digestion vessel, and after the vessel is cooled down to room temperature, add proper amount of boric acid to permit the complexing of excessive hydrofluoric acid (boracic acid is not required in case that a hydrofluoric acid resistant atomizer is used). Transfer the solution to a 50 ml volumetric flask and fill the flask up with water to 50 ml. Dilute the sample to desired concentration according to the method used.

Execute procedure identical to preparation of test sample solution concurrently without sample.

Note: Hydrogen peroxide may react rapidly and violently on easily oxidizable materials and should not be added when the sample might contain large quantities of easily oxidizable organic constituents.

7.1.7.5 Preparation of Special Electronic Materials Sample

Take 0.10 g of sample and measure its weight accurately to 0.0001 g. Transfer it into a PTFE/PFA digestion vessel, and add 5 ml of mixed acid solution of concentrated hydrochloric acid and concentrated nitric acid with appropriate proportion (typical ratio of hydrochloric acid and nitric acid, 3: 1, depending on the composition of matrix materials). Add another 1ml of concentrated hydrofluoric acid when the sample contains Si, Zr, Hf, Ti, Ta, Nb, W (See Section 5 'Screening Tests'). After a period of reaction of the sample, put the whole vessel in a microwave digestion device. Digest the sample according to the operation instruction until it is dissolved completely. Take out the digestion vessel, and after the vessel is cooled down to room temperature, add proper amount of boric acid to permit the complexing of excessive hydrofluoric acid (boracic acid is not required in case that a hydrofluoric acid resistant atomizer is used). Transfer the solution to a 50 ml volumetric flask and fill the flask up with water to 50 ml. Dilute the sample to desired concentration according to the method used.

Execute procedure identical to preparation of test sample solution concurrently without sample.

7.1.8 Test Procedure

7.1.8.1 Preparation of Calibration Solution of Lead and Cadmium

Because of the different extent of matrix effects existing in various analyzers, method of standard solution preparation shall be selected from calibration method (matrix matching method), internal standard method, and standard-addition method according to the tested materials.

Prepare calibration blank and at least three calibration standards as calibration solutions.

When internal standard method is employed, internal standard element may be added in the solution either in preparation or added inline by apparatus during testing. As to ICP-AES, scandium and yttrium can be selected as internal standard elements; and as to ICP-MS, rhodium can be used as an internal standard element. The concentration of the internal standard element is equivalent to that of tested element.

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7.1.8.2 Development of Calibration Curve of Lead and Cadmium

a) ICP/AES (-OES)

Determine the readings for the emission intensity of the target elements (and, if required, that of the internal standard element) of calibration solution series from low to high concentration.

In the calibration curve method, the curve showing the relationship between the emission intensity of the target elements and their concentration is developed as the calibration curve.

In the internal standard method, the curve showing the relation between intensity vs. concentration of the target elements with respect to that of the internal standard elements is developed as calibration curve.

Selected spectral lines and potential interferences in the analysis are shown in Table 6.

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Table 6 **Spectral Line of Cadmium and Lead and Potential Interferences**

Interference Element Spectral Line	Cd	Cd	Cd	Cd	Pb	Pb	Pb	Pb
(nm)	214.439	226.502	228.802	361.051	217.000	220.353	261.417	283.305
Ag	+	+	+	+	+	+	+	+
As	+ +	+	+ + +	+	+	+	+	+
Au	+	+	+ +	+	+	+	+	+ + +
B	+	+	+	+ + +	+	+	+ +	+
Ca	+	+	+	+	+	+	+	+
Co	+	+ +	+ + +	+ + +	+ +	+ + +	+ + +	+ +
Cr	+	+	+	+	+	+	+ +	+
Cu	+	+	+	+	+	+	+	+ +
Eu	+	+	+	+ + +	+ +	+	+ + +	+ + +
Ga	+	+	+	+	+	+	+	+
Ge	+	+	+	+	+	+	+	+
In	+	+	+	+	+	+	+	+
Ir	+ +	+ +	+ +	+ +	+ + +	+ + +	+ + +	+ + +
Mg	+	+	+	+	+	+	+	+ +
Mn	+	+	+	+ + +	+	+ +	+ + +	+
Mo	+ +	+	+	+ + +	+ +	+	+ +	+ + +
Ni	+	+	+ +	+ + +	+ + +	+ +	+	+
Pd	+	+	+	+	+	+ + +	+	+
Pt	+ + +	+	+ +	+	+	+	+	+
Re	+ +	+ +	+	+ + +	+ +	+ + +	+ +	+ + +
Ru	+ +	+	+ +	+	+ +	+	+ + +	+
Sb	+ +	+	+	+	+ +	+	+	+
Sc	+	+	+ + +	+ +	+ +	+ +	+ + +	+ +
Sn	+	+	+	+	+ +	+	+	+ +

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V	+	+	+ +	+ + +	+ +	+ +	+ +	+
W	+ +	+ +	+ +	+ +	+ + +	+	+ + +	+ +
Zn	+	+	+	+	+ + +	+	+	+
Al	+	+	+	+	+ + +	+ + +	+	+ +
Ti	+	+	+	+ +	+	+ + +	+	+ +
Fe	+ + +	+ + +	+	+ +	+ + +	+ +	+ + +	+ + +
Nb	+	+	+			+		+ + +
Hf						+		+ + +
Ta						+		+ +
Pb	+	+	+	+				-
Cd					+	+	+	+
+ +	no or small interference (Strength of interference by 1000 mg/kg matrix elements is typically no more than 0.05ppm)							
+ +	medium interference (Strength of interference by 1000 mg/kg matrix elements is about 0.05~0.2ppm)							
+ +	strong interference(Strength of interference by 1000 mg/kg matrix elements is above 0.2ppm)							

In the case of interference from co-present substances, either a wavelength that does not interfere with the calibration range has to be selected or a suitable method has to be adopted to eliminate the interference.

b) ICP/MS

Determine the isotope counting of the target elements (and, if required, that of the internal standard element) of calibration solution series from low to high concentration.

In the calibration curve method, the curve showing the relationship between the emission intensity of the target elements and their concentration is developed as the calibration curve.

In the internal standard method, the curve showing the relation between intensity vs. concentration of the target elements with respect to that of the internal standard elements is developed as calibration curve.

Selected isotope and potential interferences in the analysis are shown in Table 7.

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Table 7 Isotopes of Cadmium and Lead and Potential Interferences

Element	Isotope	Potential Isotope Interferences	Potential Interference of Molecules and ions
Cd	111	/	MoO, MoOH, ZrOH
	112	Sn	MoO, MoOH
	113	In	MoO, MoOH, ZrOH, RuO
	114	Sn	MoO, MoOH, RuO
Pb	204	/	/
	206	/	PtO
	207	/	IrO
	208	/	PtO

In the case of interference from co-present substances, either an isotope that does not interfere with the calibration range has to be selected or adjustments in interference volume have to be made using a suitable method.

c) AAS

Determine the readings for the emission intensity of the target elements of calibration solution series from low to high concentration.

Analytical line: cadmium (Cd) 228.8nm; lead (Pb) 217.0nm or 283.3nm;

Absorbance reading range: The absorbance readings typically vary between 0.1～0.6. If necessary, adjust the concentration of solution and the length of light path, or extent the measuring range;

In the case of interference from co-present substances, either a wavelength that does not interfere with the calibration range has to be selected or adjustments in interference volume have to be made using a suitable method.

7.1.8.3 Sample Analysis

After the calibration curve is plotted, measure the calibration blank, sample solution and spiked samples solution. The corresponding concentration is available from calibration curve based on the signal reading of each sample. Every sample should be determined twice and the relative standard deviation should be no more than 10% and the recovery of spiked samples should be between 90% and 110%.

As to AAS, if the sample concentration is above the range of the concentration curve, the solution shall be diluted to the range of the calibration standards.

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The standard substance or calibration solution is used as quality control
sample and the measurement precision is checked with them (such as once
every 10 samples). If necessary, a calibration curve has to be developed again.

7.1.8.4 Calculation of Analytical Results

The content of tested element in %, which is represented by mass fraction Wm,
is calculated from the equation:

$$W_M = \frac{(c1 - c2) \times V \times d \times 10^{-6}}{m} \times 100$$

Where:

c1 — Value of tested element concentration of sample solution that read from
the calibration curve, in µg/ml;

c2 — Value of tested element concentration of blank solution that read from the
calibration curve, in µg/ml;

V — Volume of the solution, in ml;

d - Dilution rate of the sample solution;

m — Sample size, in g

7.1.8.5 Precision

The absolute deviation of two results from duplicate testing shall not exceed
10% of the arithmetic average.

7.2 Testing Methods for Mercury (Hg) in Electronic Information Products

7.2.1 Scope

This document specifies the procedure for the determination of content of
mercury (Hg) in Electronic Information Products.

This method is suitable for determination of content of mercury (Hg) form
Polymer Materials, metal materials, special electronic materials and inorganic
non-metal materials.

7.2.2 Summary of Method (CVAAS, AFS, ICP - AES and ICP-MS)

An appropriate mass of cryogenically milled and homogenized sample is
digested in an acid solution under fixed temperature or pressure conditions.
After digestion, sample solution should be stored at 4 °C to minimize
evaporation. For longer term storage of mercury, it is recommended to use the
medium of 5.0% nitric acid + 0.05% potassium permanganate solution.

The element mercury in the obtained digestion solution is determined by
Cold Vapour Atomic Absorption Spectrometry (CV-AAS), Atomic

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Fluorescence Spectrometry (AFS), Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP-AES), Inductively Coupled Plasma Mass Spectrometry (ICP-MS) or Atomic Absorption Spectrometer(AAS) . When using CV-AAS and AFS, the mercury is reduced to the elemental state before it is analyzed.

7.2.3 Normative References

GB/T 15555.1-1995 Solid waste—Determination of total mercury—Cold atomic absorption spectrometry
EPA 3050 Acid Digestion of Sediments, Sludges and Soils
EPA 3052 Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices
EPA 7471A, Mercury in Solids using CVAAS (Manual)
EPA 7470A, Mercury in Water using CVAAS (Manual)
EPA 7474, Mercury in sediment and tissue by AFS

BCR - 680, BCR - 681: Plastics packaging and packaging material; certification of mass. fractions of As, Br, Cd, Cl, Cr, Hg, Pb and S in polyethylene.

7.2.4 Terms and Definitions

a) CVAAS, Cold Vapour Generation Atomic Absorption Spectrometry

Method of determining mercury content by means of reducing the mercury ion in the sample to be analyzed to free atom and measuring the characteristic electromagnetic absorption by the ground state atom of the element in vapor phase

b) AFS, Atomic Fluorescence Spectrometry

Method of qualitative and quantitative analysis by means of wavelength and intensity of atomic fluorescence spectral lines.

7.2.5 Apparatus and equipment

a) CV-AAS
b) ICP-AES(- OES)
c) ICP-MS
d) AFS
e) Heating and reflux device equipped with reaction flask, reflex condenser and absorption vessel;
f) Various glassware for laboratory
g) Hydrofluoric acid resistant sample holder
h) Heating device
i) Microwave digestion system with Polytetrafluoroethylene - tetrafluoroethylene high pressure digestion vessel

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j) Electronic analytical balance capable of accurate weighing to 0.1mg;

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Note: Because of the sensitivity of the described mercury analytical techniques, each single sampling step should be done with great care. All sampling, storage and manipulation devices have to be mercury-free. Soak all glassware with 50 % (m/m) nitric acid for 24 hours at room temperature, and then rinse thoroughly with grade 1 water, specified in ISO 3696:1987.

7.2.6 Reagents

Unless otherwise stated, only approved high purity reagents and 18 Ω ASTM Type 1 water or equivalent purity water shall be used.

a) Nitric acid: ρ = approx. 1.40 g/ml, 65%;

b) Hydrochloric acid: ρ = approx. 1.19 g/ml, 37%;

c) Hydrogen peroxide: ρ = approx. 1.10 g/ml, 30%;

d) Hydrofluoric acid: ρ = approx. ISO 40% $\approx$ 42%; JIS 46% $\approx$ 48%;

e) Sulphuric acid: ρ = approx. 1.84 g/ml, 95%;

f) Boracic acid (HBO_3);

g) Mercury standard solution, with concentration of 1000 µg/ml;

h) Sodium chloride - hydrochloric acid hydroxylamine solution: 12 g sodium chloride and 12g hydrochloric acid hydroxylamine dissolved in 100 ml water;

i) Potassium permanganate (G.R.): 5% aqueous solution (w/v), Dissolve 5 g of potassium permanganate in 100 ml of reagent water;

j) Sodium hydroxide;

k) Sodium tetrahydridoborate;

l) Potassium borohydride (trace metal grade), sodium hydroxide, G.R. 1 % in 0.05 % NaOH: Add approximately 1000 ml of reagent water to a 1L volumetric flask followed by the addition of 0.05 g sodium hydroxide. Add to 10.0 g potassium borohydride and stir to dissolve. Dilute it to scale with reagent water

7.2.7 Sample Preparation

7.2.7.1 Sample Reduction

Disassemble the electronic information products into various material samples according to Appendix A. Cut the samples into small pieces of 0.1cm×0.1cm using a shear or cutting machine (or in other manners). Metal materials and inorganic non-metal materials can be used directly in the next step, while polymer materials and special electronic materials require to be pulverized into particles or powder no more than 1mm in diameter and mixed uniformly for the next step.

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7.2.7.2 Preparation of Metal Material Sample

a) Direct dissolving

1) General Preparation Method of Samples

Take about 0.1g - 0.2 g of sample into a clean reaction flask and measure its weight accurately to 0.0001 g. Then add 30 ml conc. HNO_3. The flask is furnished with a reflux condenser and an absorption vessel containing 10 ml 0.5 Mol/L HNO_3, before a temperature program is started to digest the samples for 1h at room temperature. Raise the temperature to 90 °C and digest under the constant temperature for 2 hours. After cooling to room temperature, put the content of the absorption tube into the reaction vessel and transfer the obtained solution into a 250 ml volumetric flask and filled the flask up with 5 % (m/m) HNO_3 to the mark.

2) Digestion of materials containing zirconium (Zr), hafnium (Hf), titanium (Ti), copper (Cu), silver (Ag), tantalum (Ta), niobium (Nb) or tungsten (W)

Take about 0.1 g - 0.2 g of sample into a clean reaction flask and add 20 ml of concentrated hydrochloric acid and 10ml of concentrated nitric acid. The flask is furnished with a reflux condenser and an absorption vessel containing 10 ml 0.5 Mol/L HNO_3, before a temperature program is started to digest the samples for 1h at room temperature. Raise the temperature to 95 °C ± 5°C and digest under the constant temperature for 15 minutes. Remove the sample from the heating digester and let it cool down to room temperature.

If the sample is not digested completely, repeat adding aqua regia and heat again, until the sample is digested completely. With each subsequent addition of acid, the sides of the chamber shall be rinsed so that any sample that adheres to the sides of the chamber is reintroduced into the solution.

When the sample is digested completely, add 20ml of reagent water and 15ml of $KMnO_4$ solution to the reaction chamber. Mix thoroughly and continue heating for 30 min at 95° C ± 5° C. After cooling to room temperature the solution is quantitatively transferred over a filter into a 100 ml volumetric flask. Rinse the reaction chamber, condenser and absorber tube with the reagent water, and transfer the rinsing solution into the volumetric flask. Add 6ml of sodium chloridehydroxylamine- hydrochloride to reduce the excess permanganate. Dilute the digested sample solution to the mark with reagent water and mix thoroughly.

Execute procedure identical to preparation of test sample solution concurrently without sample.

b) Microwave Digestion (Mainly for Insoluble Alloys)

Take about 0.10 g of sample and measure its weight accurately to 0.0001 g. Transfer it into a PTFE/PFA digestion vessel, and add 5 ml of mixed acid solution of hydrochloric acid and nitric acid with appropriate proportion (typical ratio of hydrochloric acid and nitric acid, 3: 1, depending on the composition of matrix materials). Add another 1ml of concentrate hydrofluoric acid when sample contains Si, Zr, Hf, Ti, Ta, Nb or W (See Section 5 "Screening Experiments").

After a period of reaction of the sample, put the whole vessel in a microwave digestion device. Digest the sample according to the operation instruction until it is dissolved completely. Take out the digestion vessel, and after the vessel is cooled down to room temperature, add proper amount of boric acid to permit the complexing of excessive hydrofluoric acid (boracic acid is not required in case that a hydrofluoric acid resistant atomizer is used). Transfer the solution to a 50 ml volumetric flask and fill the flask up with water to 50 ml. Dilute the sample to desired concentration according to the method used.

Execute procedure identical to preparation of test sample solution concurrently without sample.

7.2.7.3 Preparation of Inorganic non-Metal Material

Hydrofluoric acid resistant vessels must be used, because some non–metal elements such as silicon are typically contained in inorganic non–metal samples and extremely corrosive hydrofluoric acid is required during the preparation of sample.

Take 0.10 g of sample and measure its weight accurately to 0.0001 g. Transfer it into a PTFE/PFA digestion vessel, and add 3 ml of mixed acid solution of concentrated hydrochloric acid and concentrated nitric acid with appropriate proportion (typical ratio of hydrochloric acid and nitric acid, 3: 1) and 3 ml of concentrated hydrofluoric acid. After a period of reaction of the sample, place the whole vessel in a microwave digestion device. Digest the sample according to the operation instruction until it is dissolved completely. Take out the digestion vessel, and after the vessel is cooled down to room temperature, add proper amount of boric acid to permit the complexing of excessive hydrofluoric acid (boracic acid is not required in case that a hydrofluoric acid resistant atomizer is used). Transfer the solution to a 50 ml volumetric flask and fill the flask up with water to 50 ml. Dilute the sample to desired concentration according to the method used.

Execute procedure identical to preparation of test sample solution concurrently without sample.

7.2.7.4 Preparation of Polymer Materials Sample

Take 0.10 g of sample and measure its weight accurately to 0.0001 g. Transfer it into a PTFE/PFA digestion vessel, and add 5 ml of nitric acid and 1ml of hydrogen peroxide. Add another 1ml of concentrate hydrofluoric acid when sample contains Si, Zr, Hf, Ti, Ta, Nb or W (See Section 5 "Screening Experiments"). Put the whole vessel in a microwave digestion device. Digest the sample according to the operation instruction until it is dissolved completely. Take out the digestion vessel, and after the vessel is cooled down to room temperature, add proper amount of boric acid to permit the complexing of excessive hydrofluoric acid (boracic acid is not required in case that a hydrofluoric acid resistant atomizer is used). Transfer the solution to a 50 ml volumetric flask and fill the flask up with water to 50 ml. Dilute the sample to desired concentration according to the method used.

Execute procedure identical to preparation of test sample solution concurrently without sample.

Note: Hydrogen peroxide may react rapidly and violently on easily oxidizable materials and should not be added when the sample might contain large quantities of easily oxidizable organic constituents.

7.2.7.5 Preparation of Special Electronic Materials Sample

Take 0.10 g of sample and measure its weight accurately to 0.0001 g. Transfer it into a PTFE/PFA digestion vessel, and add 5 ml of mixed acid solution of concentrated hydrochloric acid and concentrated nitric acid with appropriate proportion (typical ratio of hydrochloric acid and nitric acid, 3: 1, depending on the composition of matrix materials). Add another 1ml of concentrated hydrofluoric acid when the sample contains Si, Zr, Hf, Ti, Ta, Nb or W (See Section 5 "Screening Experiments"). After a period of reaction of the sample, put the whole vessel in a microwave digestion device. Digest the sample according to the operation instruction until it is dissolved completely. Take out the digestion vessel, and after the vessel is cooled down to room temperature, add proper amount of boric acid to permit the complexing of excessive hydrofluoric acid (boracic acid is not required in case that a hydrofluoric acid resistant atomizer is used). Transfer the solution to a 50 ml volumetric flask and fill the flask up with water to 50 ml. Dilute the sample to desired concentration according to the method used.

Execute procedure identical to preparation of test sample solution concurrently without sample.

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7.2.8 Test Procedure

7.2.8.1 Preparation of Calibration Solution of Mercury

The standard mercury solution shall be stored in an inert plastic container. The stable period of mercury solution with a concentration of 1000 µg/ml is more than 1 year, and the solution with concentration lower than 1 µg/L shall be prepared in site.

The stability of mercury standard solutions can be severely affected because mercury tends to be absorbed on the internal wall of the container. Therefore it is recommended to stabilize mercury standard solutions by an addition of a few drops of 5 % $KMnO_4$ solution.

Because of the different extent of matrix effects existing in various analyzers, method of standard solution preparation shall be selected from calibration method (matrix matching method), internal standard method, and standard-addition method according to the tested materials.

Prepare calibration blank and at least three calibration standards as calibration solutions.

When internal standard method is employed, internal standard element may be added in the solution either in preparation or added inline by apparatus during testing. As to ICP-AES, scandium and yttrium can be selected as internal standard elements; and as to ICP-MS, rhodium can be used as an internal standard element. The concentration of the internal standard element is equivalent to that of tested element.

7.2.8.2 Development of Calibration Curve of Mercury

a)   ICP/AES(-OES)

Determine the readings for the emission intensity of mercury (and, if required, that of the internal standard element) of calibration solution series from low to high concentration.

In the calibration curve method, the curve showing the relationship between the emission intensity of mercury and its concentration is developed as the calibration curve.

In the internal standard method, the curve showing the relation between intensity vs. concentration of mercury with respect to that of the internal standard elements is developed as calibration curve.

Selected spectral line of mercury in the analysis: 194.227 nm

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b) ICP/MS

Determine the isotope counting of mercury (and, if required, that of the internal standard element) of calibration solution series from low to high concentration.

In the calibration curve method, the curve showing the relationship between the emission intensity and the concentration is developed as the calibration curve.

In the internal standard method, the curve showing the relation between intensity vs. concentration of mercury with respect to that of the internal standard elements is developed as calibration curve.

Isotope of mercury selected for analysis in calibration curve method and internal standard method: m/z = 202

c) CVAAS:

Determine the readings for the emission intensity of mercury of calibration solution series from low to high concentration, and the curve showing the relationship between the emission intensity of mercury and its concentration is developed as the calibration curve.

Absorbance reading range: the absorbance reading typically varies between 0.1 to 0.6 to minimize the error of absorbance measurement. If necessary, adjust the concentration of solution and the length of light path, or extent the measuring range;
    Light source: Hg electrodeless discharge lamp or hollow cathode lamp
    Wavelength: 253.7 nm
    Slit width: 0.7 nm
    Purge air: N2 or Ar

    Reducer: 3% $NaBH_4$ (dissolved in 1% NaOH)

d)   AFS

Determine the readings for the fluorescence intensity of mercury of calibration solution series from low to high concentration.

The curve showing the relationship between the fluorescence intensity of mercury and its concentration is developed as the calibration curve..

Fluorescence intensity reading range: the fluorescence intensity reads shall fall into the linear range of the instrument to minimize the error of absorbance measurement. If necessary, adjust the concentration of solution;

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Instrument parameters:

Light source: Hg hollow cathode lamp, current: 30 mA, wavelength: 253.7 nm

Minus high-voltage: 360 V

Furnace temperature: 800° C

Argon flow   Carrier gas: 600 ml/min, Screen gas: 1000 ml/min

Reducer: 3%NaBH$_4$ (dissolved in 1% NaOH)

7.2.8.3 Sample Analysis

After the calibration curve is plotted, measure the calibration blank, sample solution and spiked samples solution. The corresponding concentration is available from calibration curve based on the signal reading of each sample. Every sample should be determined twice and the relative standard deviation should be no more than 10% and the recovery of spiked samples should be between 90% and 110%.

As to AAS, if the sample concentration is above the range of the concentration curve, the solution shall be diluted to the range of the calibration standards.

The effect of matrix composition on the redox reaction shall be taken into account when CVAAS or AFS is selected.

The standard substance or calibration solution is used as quality control sample and the measurement precision is checked with them (such as once every 10 samples). If necessary, a calibration curve has to be developed again.

7.2.8.4 Calculation of Analytical Results

The content of tested element in %, which is represented by mass fraction Wm, is calculated from the equation:

$$W_M = \frac{(c1-c2) \times V \times d \times 10^{-6}}{m} \times 100$$

where:

c1 — Value of tested element concentration of sample solution that read from the calibration curve, in µg/ml;

c2 — Value of tested element concentration of blank solution that read from the calibration curve, in µg/ml;

V — Volume of the solution, in ml;

d - Dilution rate of the sample solution;

m — Sample size, in g.

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7.2.8.5 Quality Control

a) Contamination by Mercury

Cautious handling of apparatus and careful technique will minimize this problem. The following precautions contribute to avoiding sample contamination:

Use only distilled or deionized water. Care must be taken that all materials in contact with the water are composed of an inert plastic. Pure water, even when stored in PTFE, can leach impurities from the container in very short periods of time.

Chemicals used for sample preparation can be a major source of contamination. Only reagents that are free of mercury should be used.

It is therefore strongly recommended to measure the blank values of the reducing agents and the other chemicals before using them for sample preparation.

Beakers, pipettes volumetric flasks, etc., are all major sources of metal contamination. It is best to use inert plastics for sample handling.

For measurements by ICP-AES(-OES) and ICP-MS and AFS, the memory effect occurs in cases where high concentrations of mercury are introduced. Therefore when the tested solution is with high concentration of mercury, sampling system with memory effect as low as possible shall be selected, or sample solution shall be diluted, and the sampling system shall be fully cleaned after the measurement.

b) Precision

The absolute deviation of two results from duplicate testing shall not exceed 10% of the arithmetic average.

8 Determination of Hexavalent Chromium (Cr VI) in Electronic Information Products by Colorimetric Method

8.1 Qualitative Testing of Hexavalent Chromium (Cr VI)

8.1.1 Scope

This method is best used for qualitative determination of hexavalent chromium from coating.

8.1.2 Normative References

ISO 3613:2000(E), "Chromate conversion coatings on zinc, cadmium, aluminium-zinc alloys and zinc-aluminium alloys - Test methods"

8.1.3 Terms and Definitions

None

8.1.4 Apparatus and equipment

None

8.1.5 Reagents

All the reagents shall be with a grade of A.R. or above.
    a) Diphenylcarbazide;
    b) Acetone;
    c) Alcohol (95%);
    d) Orthophosphoric acid solution (75%);
    e) De-ionized water.

8.1.6 Sample Preparation

Prior to the test, the sample surface shall be free of all contaminants, finger prints and other extraneous stains. If the surface is coated with thin oil, it shall be removed prior to the test using a clean, soft lab wipe wetted with a suitable solvent, or rinsing the surface with a suitable solvent at room temperature (not exceeding 35º C). The samples shall not be subject to forced drying at temperature in excess of 35º C. Treatment in alkaline solutions shall not be performed as chromate coatings are broken down by alkalis.

If there is a polymer coating on the top of a sample surface, a gentle abrasion with a fine sandpaper, such as a SiC grinding paper with 800 grit size, may be applied to remove the polymer layer, but without removing the chromate coatings on the sample. Other coating removal methods may be applied if they are proven to be more effective.

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Testing Methods for Regulated Substances in Electronic Information Products

Appendix A: Guide to method of preparation of samples in process of testing for toxic substances

The purpose of this Appendix is to provide integrated rules for the dismantling and preparation of samples in the process of testing electronic information products for toxic substances.

A1 Structure of electronic information products

A1.1 Terminology and definitions

A1.1.1 Overall unit: Indicates an electronic product that is capable of providing a given function when supplied with electric power, such as a television set, telephone or electric fan and so forth

A1.1.2 Part: Indicates a structural unit of the overall unit and that can be dismantled by means of simple tools, such as a circuit board, power supply or module and so forth

A1.1.3 Essential electronic component: Indicates a structural electronic component or part of a circuit board, such as a resistor, capacitor, integrated circuit, lighting part or detachable plug board and so forth

A1.1.4 Raw material: Principally indicates a basic structural material of an essential electronic component or structural part, such as a metal, plastic, solder, adhesive, coating or cleaning liquid and so forth, such substances generally appearing to be 'homogeneous substances'

A1.2 Classification of methods of connection

A1.2.1 Physical connection: Indicates all methods of joining or fixing two different materials by means of physical force such as pressure, friction or gravity and so forth. Normally indicates crimping, riveting, bonding, binding, screwing, buttons, covers or hoops and so forth

A1.2.2 Chemical connection: Indicates a method of joining two different materials that requires a metallurgical or chemical reaction to form the connection. Normally indicates welding, electroplating, galvanizing or bonding and so forth

A1.3 Restricted substances in high risk areas and states

A1.3.1 Lead: Additive to coatings, pigments, stabilizers, batteries, solder, cladding, glass, light bulbs, solid lubricants, rubber and so forth

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A1.3.2 Cadmium: Stabilizers in plastics, coatings in electrical contacts, batteries, springs, joints, PCBs, fuse wire, pigments and coatings, semiconductor photoelectric sensors and so forth

A1.3.3 Mercury: Additive in plastics, colorants, fluorescent lamps, thermostats, sensors, relays, metal etching agents, batteries, rust preventive agents, disinfectants and adhesives and so forth

A1.3.4 Hexavalent chromium: Anti-rust coatings on metals, pigments, rust preventive agents, corrosion preventive agents and ceramics and so forth

A1.3.5 PBBs and PBDEs: Fireproofing agents for organic materials, PCBs, joints and outer shells of plastic and so forth

A2 Purpose of dismantling electronic information products, and principles for dismantling

A2.1 Purpose

In order to accurately measure the concentrations of regulated substances in electronic information products and to provide effective controls over the use of toxic substances in electronic information products, the products (to be tested for toxic substances) must be dismantled into the basic structural parts and units prior to measurement. (See **Table 2** for details.)

Table A1 Purpose of dismantling electronic information products (EIP)

Structural unit	Definition of basic structural unit or material
EIP-A	Forms homogenous substances in electronic information products
EIP-B	Plating material on parts in electronic information products
EIP-C	Small parts of electronic information products, such as small or non-homogeneous parts that cannot be further dismantled, and are less than 4 mm^3 in volume (equivalent to 0805 flat items)

A2.2 Principles for dismantling

A2.2.1 Electronic information products must be dismantled strictly in accordance with the purposes for dismantling in Table A1, and after they have been dismantled, all structural elements must be assigned to category A, B or C.

A2.2.2 It is economically practical to take into account both the practicalities of measuring equipment and the needs of the electronics industry. Before dismantling an item, make an assessment of the likelihood of the presence of

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toxic substances by referring to A1.3. If the risk of toxic substances is very low (meaning, insignificant amounts added, and the possibility of contamination from toxic substances during the production process is also relatively low) the part need not be dismantled.

A2.2.3 When a sample is being prepared, first separate any special materials or special members from other parts (EIP – A, B, C).

A2.2.4 Samples with a volume of $\leq$1.2 mm² need not be further dismantled: these may form whole samples (in other words EIP-C type structural units; example: 0805 sealed units 2.0 mm x 1.2 mm x 0.5 mm need not be dismantled).

A2.2.5 Physical joints must be dismantled as far as possible down to the member before the joint or to a unit not exceeding 1.2 mm³ in volume.

A2.2.6 Chemical joints may, if plated joints (EIP-B) be prepared as lateral sections, using XRF or SEM/EDS to perform qualitative or semi-quantitative determinations in order to determine whether any significant amounts of regulated substances were added when the plated layer was formed. Samples of the main body of the objec! (substrate material) are to be prepared by removing the plated layer by mechanical or chemical means.

A2.2.7 If a chemical joint is a connection in which a surface material of one type is joined to a terminal of another type of material, or a terminal of two types of material, separate and remove any non-chemical joint in order to prepare the sample.

A2.2.8 If these technical means are not sufficient to dismantle an electronic information product and to prepare a sample, consideration may be given to substituting measurements of a sample of structural materials with measurements from the same batch of materials.

A3 Typical examples of dismantling

A3.1 When dismantling a circuit board, select the large solder points as far as possible and remove the solder material from within them; prevent the plated layers of wiring and pad from coming loose from the main unit. At the same time, take care to select the rubber parts that are used for joints or fixtures on the underside and remove all the electronic units and parts.
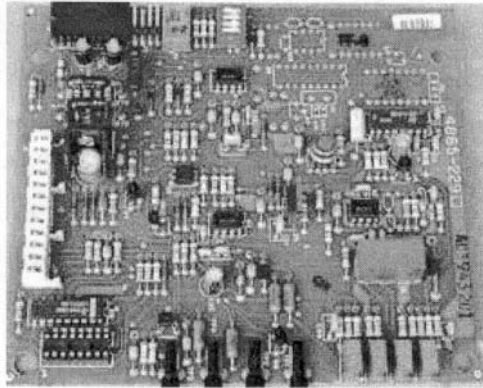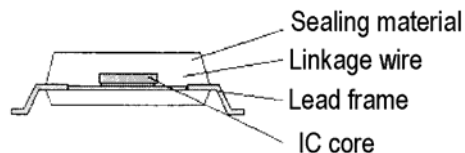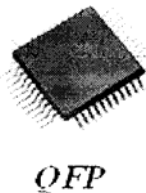
Example of electronic circuit board

A3.2 Example of dismantling electronic integrated circuits with pins

There are many types and shapes of integrated circuits with pins. These include DIP, SOP and QFP, with QFP being the most typical. This example is of the dismantling of a QFP type integrated circuit.

The principal risk with QFP type integrated circuits is that the lead and plastic seal over the leads may contain these regulated substances. The main body of the integrated circuit may also contain high temperature lead solder, which is a form of special material. Integrated circuits of >4 mm^3 should be dismantled into two parts: the pins and the rest of the body. QFP type integrated circuits of <1.2 mm^3 need not be dismantled. They should be treated as EIP-C as provided for in A2.2.

A3.3 Example of dismantling array type integrated circuits

More specifically, array type integrated circuits include the ball grid array and column grid array and so forth, and they may consist of one or multiple type of array. Some examples of ball grid arrays are PBGA, FCBGA, CSP and WLCSP etc. The principal risks with BGA and CSP sealed type integrated circuits is the lead in the solder globules, and other regulated substances that may be present in the plastic sealing material. The main body of these integrated circuits often contains special types of materials (exempt materials) such as high temperature solder containing lead.

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Examples of dismantling PBGA and FCBGA type integrated circuits are shown.
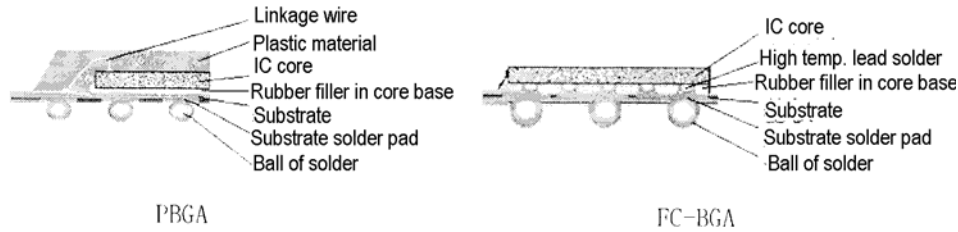
Criterion:

Possible to separate the solder and main body (Note: The main body may contain special exempt materials.)

A3.4 Example of dismantling printed circuit boards

Printed circuit boards may be divided according to the material forming the substrate into inorganic substrates and organic substrates. They generally consist of a silk screen printing ink, a solder block film, a solder pad, surface layer copper wires, inner layer copper wires, porous plated copper and the substrate. The important points to consider with this type of circuit board are the effects of the solder on the coated layer and printed parts, and the additives and flame-retardants that may occur in the organic materials.

Method of disassembly: Cut away the solder and sample the printing ink and the organic material.

Test the solder pad as for category EIP-B coated layer.

Cut a sample from a position on the organic substrate that does not contain any parts, perforations or copper.

A3.5 Example of dismantling rectangular tabular part without pins

There are many different types and shapes of rectangular tabular parts without pins. This example is of the dismantling a particular type of resistor.
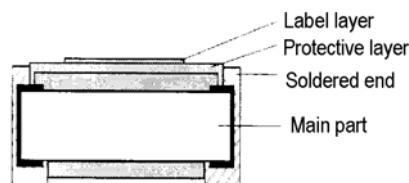
A tabular shaped resistor consists of a label layer, a protective layer, solder ends and the main part.

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Criteria for dismantling:

> If volume is $\leq$1.2 mm^3, use the whole unit as the sample
> If volume is >1.2 mm^3: if the solder forms a plated layer, prepare a sample in the normal manner for plated layers; if there is a physical join, dismantle the terminals and sample
> Sample material of the main part directly.

A3.6 Example of dismantling separate plug-in units

There are many types of such units, including resistors, capacitors, sensors and diodes and triodes and so forth.

Criteria for dismantling:

> Sample with leads sheared:
> > If the volume is $\leq$ 1.2 mm^3, use the whole unit as the sample
> > If the volume is >1.2 mm^3, refer to the general principles on dismantling to sample

A3.7 Example of dismantling plug-in electrolytic capacitors

The structures of plug-in electrolytic capacitors are relatively complex; they consist principally of a casing, rubber, electrolyte solution, electrolyte paper separators, aluminium foil, aluminium casing and pins.

If the volume of the main part of the capacitor is $\leq$1.2 mm^3, dismantle into the pins and main part.

If the volume of the main part of the capacitor is >1.2 mm^3, dismantle into the pins, outer casing, separator and positive and negative poles.

A3.8 Example of dismantling wires and cables

There are very many types of wires and cables: they include electrical wiring, electrical cabling, optical fibres and optical cable.

These types of materials have relatively simple structures, generally consisting of an outer protective layer, an inner protective layer and an inorganic core. They should be dismantled according to their structures.

A3.9 Metal plated layer samples

Prepare samples according to the principles listed in A2.2, or do not sample, but use XRF or colorimetry or other qualitative methods to make a determination.

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