RoHS Analysis Method Guidelines

Version 3.0

March 2019
SHARP Corporation

Table of Contents

Revision History **3**

Introduction **4**

1. Purpose 4
2. Scope 5
3. Reference standards 5
4. Glossary 6
5. Requirements for analysis data 9
6. Cases when analysis data is not required 9
7. Guideline structure 10

[1] Selection of RoHS analysis method **12**

1. What is RoHS analysis? 12
2. Types of RoHS analysis 12
3. Preparation for RoHS analysis 12
4. RoHS analysis 12
5. Sharp Original Standards 13

[2] Screening analysis **14**

1. What is screening analysis? 14
2. Screening analysis techniques 14
3. Screening analysis procedures 16

[3] Precision Analysis **24**

1. What is precision analysis? 24
2. Precision analysis methods 24
3. Precision analysis procedures 24
4. Performing precision analysis 25

[4] Handling the Analysis Data **38**

Annex I **39**

Annex II **40**

Annex III **42**

Annex IV **43**

Revision History

Version	Issued on	Remarks
1.1	July 2006	Guidelines based on analysis of each homogeneous material issued based on the following concepts. • RoHS Enforcement Guidance Document Version 1 issued in UK • Frequently Asked Questions on RoHS & WEEE issued by European Commission
1.2	November 2006	Reviewed conditions of extracting hexavalent chromium eluted from chromate film, and set provisional allowable value for control standards.
2.0	March 2009	Matched with IEC 62321 Ed. 1.0: 2008.
2.1	November 2010	Deleted FT-IR from screening and qualitative analysis for PBB and PBDE.
2.2	April 2012	Added secondary screening to PBB and PBDE analysis, and unified notifications.
3.0	March 2019	Added screening analysis method and criteria for determining inclusion of DEHP, DBP, BBP, and DIBP. Changed layout and configuration. Changed the title of this guidelines in accordance with the revised. • Before : SHARP corporation's RoHS Analysis Method Guidelines • After : RoHS Analysis Method Guidelines
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Introduction

1. Purpose

Sharp, together with the cooperation of our suppliers, is pursuing management of substances contained in products to reduce the environmentally impact of our products and to conform to globalized chemical substance restrictions. We have established the "Green Procurement Guidelines" to comply with legal restrictions, especially the EU RoHS Directive, which prohibits and restricts the inclusion of substances in products. From October 2003, Sharp has requested the provision of the chemical substances contained in product with the specifications of newly adopted products in these guidelines. Specifically, the provision of both the "Report on Chemical Substances Contained in the Product" and "Analysis Data" is requested.

[Submit and store evidences]

Green Procurement Guidelines Ver. 7.0
6.1 "Environmental Management Evaluation" (1) Chemical Substance Management Item No. 4

Evidence to show non-inclusion of RoHS restricted substances, such as analysis data, is submitted. And, originals of its evidence is stored and managed. .

As evidence to show non-inclusion of RoHS Directive restricted substances, "Analysis Data" should be attached. It is necessary to include measurement site, actual measurement value and name of measurement institute in the analysis data. Objective documents which can be confirmed conformity with standards of Sharp are accepted as evidence.

- Analysis report issued by third-party measurement body is preferred.
- In case of metal materials, compliance with JIS or other public standards that meet RoHS requirements is regarded as evidence.
- In case of parts or materials that consist of more than one homogeneous material, the document which is summarized the "Analysis Data" of each homogeneous material is acceptable. However, an original analysis data should be managed and submitted to Sharp as required.
- If parts or materials are clearly made with the same constituent material, "Analysis Data" for a typical part or material can be used for each maker, even if the part codes are different.
- "Analysis Data" less than one year old since the testing date should be submitted.
- Latest "Analysis Data" which is renewed within one year should be stored.

The first version of the "*Sharp's RoHS Analysis Method Guideline*" was issued in July 2006. These Guidelines are the index for preparing the "Analysis data", and outline the analysis methods for each homogeneous material of the six RoHS restricted substances lead (Pb), mercury (Hg), cadmium (Cd), hexavalent chromium (CrVI), polybrominated biphenyls (PBB), and polybrominated diphenyl ethers (PBDE).

The EU RoHS Directive was revised in June 2015. From July 22, 2019, the use of four types of phthalate esters "bis (2-ethylhexyl phthalate (DEHP), butyl benzyl phthalate (BBP), dibutyl phthalate (DBP), and diisobutyl phthalate (DIBP)" will be restricted. Based on these revisions, the “*RoHS Analysis Method Guidelines Version 3.0*” has been issued as an index for analysis methods, including these newly restricted phthalate esters.

These Guidelines conform to IEC 62321 "International standard of determination of certain substances in electrotechnical products", and include some "Sharp original standards". Refer to section [1] 5. Sharp original standards and the sections indicated with Sharp original standards for details on these original standards.

Please use these Guidelines during analysis at your company, and when commissioning analysis to a third-party agency.

2. **Scope**

(1) **Target items**

All of the following items procured or purchased by Sharp are subject to these guidelines.

1)	Direct materials:	Parts and raw materials to be assembled into Sharp products.
2)	Finished goods:	Finished goods Sharp purchases and delivers to customers.
3)	Semi-finished goods:	Semi-finished goods to be assembled in the Sharp products.
4)	Packaging materials:	Packaging materials to pack the Sharp products.
5)	Indirect materials:	Auxiliary materials such as chemical, gas, etc., used in the manufacturing process.
6)	Others:	Equipment and other consumable materials, etc.

Regarding item 4), packaging material that will be clearly disposed of at a Sharp base, etc., and for which there is no risk of restricted substance transferring or contaminating parts or materials shall be excluded from the target.

(2) **Target chemical substances**

The ten substances set forth by the European Commission (EU) 2015/863[1] (hereinafter, RoHS restricted substances), listed below are the chemical substances restricted by these guidelines. Each chemical substance will be indicated with the respective abbreviation in the following explanations.

Table 1 RoHS restricted substances

Chemical substance name	Barb.
Lead	Pb
Mercury	Hg
Cadmium	Cd
Hexavalent chromium	CrVI
Polybrominated biphenyls	PBB
Polybrominated diphenyl ethers	PBDE
Bis(2-ethylhexyl) phthalate	DEHP
Bis(butylbenzyl) phthalate	BBP
Dibutyl phthalate	DBP
Diisobutyl phthalate	DIBP

3. **Reference standards**

The reference standards for these guidelines are IEC 62321[2], the official analysis method for RoHS restricted substances.

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[1] Directive to replace Annex II setting forth the RoHS Directive (2011/65/EU) restricted substances.

[2] IEC62321 is an International Standard which specifies the procedures for determining the content of restricted substances in electrotechnical products. (Determination of certain substances in electrotechnical products)

4. **Glossary**

A glossary of terms used in these guidelines is given below.

Term and abbreviation	Explanation
(EU)2015/863	Directive revising the restricted substances listed in EU RoHS Directive (2011/65/EU) Annex II DEHP, BBP, DBP, and DIBP added to six restricted substances Pb, Hg, Cd, CrVI, PBB, and PBDE listed in RoHS Directive (2011/65/EU)
2011/65/EU	EU RoHS Directive Directive of the European Parliament and of the Council of 8 June 2011 on the restriction of the use of certain hazardous substances in electrical and electronic equipment
AAS	Atomic Absorption Spectroscopy An analysis method in which a sample solution is sprayed onto an acetylene combustion flame to identify the type and amount of elements from the absorbance at a specific wavelength absorbed by atomized elements in the sample solution.
AES	Atomic Emission Spectroscopy An analysis method in which a sample solution is sprayed onto a high temperature gas or plasma to identify the type and amount of elements from the intensity of light at a specific wavelength emitted by the atomized elements in the sample solution.
Absorption photometric analysis	Method of analyzing concentration of restricted substance from concentration of coloration substance in solution and absorbency
Analysis data	Documents used as evidence to show non-inclusion of RoHS restricted substances for the Report on Chemical Substances Contained in the Product.
BFR	Brominated Flame Retardants Substance containing molecular-form bromine to suppress combustion of resins (polymers).
Bulk	All solid samples that are not thin films
CV-AAS	Cold Vapor-Atomic Absorption Spectroscopy Flameless atomic absorption spectrometry used for analysis of mercury. This analysis method uses ASS to identify mercuric ion in the sample solution using the metallic mercury vapors reduced with tin chloride.
CV-ICP-OES	Cold Vapor-Inductively Coupled Plasma Optical Emission Spectroscopy Flameless atomic emission spectroscopy used for analysis of mercury. This analysis method uses ICP-OES to identify mercuric ion in the sample solution using the metallic mercury vapors reduced with tin chloride.
Chromate	Process that immerses galvanized steel material, etc., in a hexavalent chromium solution to passivate the metal surface and improve corrosion inhibiting performance.
Diphenylcarbazide	Reagent that forms a reddish-purple complex that absorbs hexavalent chromium and light of 540nm
FLAAS	Flameless Atomic Absorption Spectroscopy Analysis method that uses AAS to identify the type and amount of elements in sample solution placed in a graphite cell and atomized with electric heating.

Term and abbreviation	Explanation
GC-MS	Gas Chromatography Mass Spectrometry An analysis method that uses "mass spectrometry (MS)" to identify the type and amount of substances separated with "gas chromatography (GC)". GC: Method and apparatus that separates substances using the difference in time that is required for gasified substances to pass through capillary tubes coated with absorbent. MS: Analysis method and apparatus that decomposes and quantifies substances separated with GC into atomic and molecular level ion, and identifies the type and amount of substance.
Green Procurement Guidelines	Guidelines setting forth the evaluation criteria for purchasing material by Sharp
HPLC-UV	High Performance Liquid Chromatograph – UV Spectroscopy An analysis method that uses "Ultraviolet-Visible spectroscopy" to analyze the type and amount of substance separated with the "liquid chromatograph (LC)". LC: Method and apparatus that separates substances using the difference in time that is required for substances in solution to pass through column coated with absorbent. UV: An analysis method and apparatus used to identify the type and amount of substance from the absorbance of a specific wavelength absorbed by the substance separated with LC.
Homogeneous material	Homogeneous Material Material that is separated to a level that cannot be further separated with mechanical action.
Hydride generation	Method of atomizing mercuric ion in sample solution by adding reducing agent (tin chloride).
IAMS	Ion attached Mass Spectrometry An analysis method to identify the type and amount of substance with a mass spectrometer after attaching lithium ions to substance volatized and separated by heating resin sample.
ICP	Inductively Coupled Plasma Plasma generated by applying high-frequency current mainly onto argon gas, and used source of energy for exciting and emitting elements in analysis sample.
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry An analysis method that sprays and ionizes sample solution in ICP, and uses mass spectrometer to identify type and amount of elements.
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy An analysis method that sprays sample solution onto argon plasma generated with ICP to excite and emit light of elements in sample solution, and identify type and amount of elements from absorbance of waveform unique to element.
IEC 62321	International standards for measuring the content of restricted substances in electrotechnical devices and products Determination of certain substances in electrotechnical products Consistent standards specifying the details from sample preparation to analysis procedures used to confirm conformity of product to RoHS Directives
JIS	Japan Industry Standard

Term and abbreviation	Explanation
MS	Mass Spectrometry An analysis method that decomposes substance into atomic and molecular level ion, and identifies the type and amount of substance from the ion weight and distribution.
Microwave heating decomposition	Method of decomposing sample by placing acid and sample (volatile element, mercury, etc.) in sealed Teflon container, and directly heating with microwaves. Sample can be decomposed in a short time as inside of sealed container reaches high temperature and high humidity.
Open acid decomposition	Method of decomposing sample by heating container containing sample (polymer, metal) and acid.
PWB	Printed Wiring Board (also called: Print Circuit Board) Circuit board on which electronics are mounted, or a board on which parts are not yet mounted.
Plasticizer	Substance added to resins (polymers) to increase plasticity of resins.
Precision analysis	Quantitative analysis, including pre-treatment of measurement sample, using chemical methods
Pyro-GC-MS	Pyrolysis Gas Chromatography Mass Spectrometry An analysis method that separates substances volatized and separated by heating the resin sample with the GC, and then identifies the type and amount of substance with MS.
Report on Chemical Substances Contained in the Product	Sharp-designated document for reporting on inclusion of environmentally hazardous substances, etc., in procured goods. (Formerly: Report on Environmental Impact Substances Contained in the Product)
Screening	Action to categorize whether the sample conforms, does not conform, or falls in gray zone of legal restrictions If it cannot be determined that a sample is confirming or non-confirming, and is determined as in the gray zone, a secondary screening is performed to determine whether the sample is conforming, non-conforming, or gray zone.
Soxhlet extraction	Method of extracting substances from solid sample Extraction method that condenses substance in solvent by repeating the process of "heating vaporization, cooling condensation, and drip extraction" of the solvent into which the substance is being extracted from the solid specimen.
Supplier chain	Companies present in the process from raw materials to product. Process: Raw material manufacturer => primary processing manufacturer => secondary processing manufacturer => parts assembly manufacturer => module assembly manufacturer => finished goods assembly manufacturer => sales company
Thin film	Film with a thickness of less than 0.1µm
UV	Ultraviolet Visible Spectroscopy An analysis method to identify the type and amount of substance from the absorbance of a specific wavelength absorbed by the substance.
XRF	X-Ray Fluorescent analysis An analysis method to identify the type and concentration of element in a specimen from the intensity of the fluorescent X-ray unique to the element emitted when an X-ray is irradiated on the element.

5. Requirements for analysis data

In these Guidelines, the following requirements are set forth as "analysis data" prerequisites.

Make sure that the analysis data satisfies the following requirements.

- Actual measurement data of RoHS restricted substance based on IEC 62321*
- Analysis report is issued by third-party analysis agency, and name of measuring agency is clearly indicated
- The date of submission to Sharp is less than one year from the analysis data measurement date

*) These Guidelines conform to IEC 62321 "International standard of determination of certain substances in electrotechnical products", and include some "Sharp original standards". Refer to section [1] 5. Sharp original standards and the sections indicated with Sharp original standards for details on these original standards.

6. Cases when analysis data is not required

(1) When material acceptable as analysis data substitute is available

Submission of analysis data is not required when material other than the analysis data, is available to confirm the following RoHS compliance (non-inclusion of substances restricted by RoHS Directive).

- Composition table issued by material manufacturer
- For metal materials, documents indicating public standards such as JIS showing compliance to RoHS

(2) Members with low possibility of inclusion

Submission of analysis data is not required for the following members.

- Packaging material that will be clearly disposed of at a Sharp base, etc., and for which there is no risk of restricted substance transferring or contaminating parts or materials
- Members that, based on composition of part, clearly do not contain RoHS restricted substances[3]

Table 2 Members that clearly do not contain RoHS restricted substances

Member	RoHS restricted substances not requiring analysis
Glass for metal seal	PBB, PBDE, DEHP, DBP, BBP, DIBP
Chromate treated (Steel sheets, screws, nuts, washers, etc.) Electroless nickel plating	
Metal, ceramic parts	

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[3] It is clear that the components shown in Table 2 do not contain PBB, PBDE, DEHP, DBP, BBP, or DIBP. So in principle, analysis for these substances is not required. However, we may ask you to submit analysis data based on request from our customers. Incidentally, analysis data for these members is required because of they have possibility of containing lead, mercury, cadmium, or hexavalent chromium.

7. Guideline structure

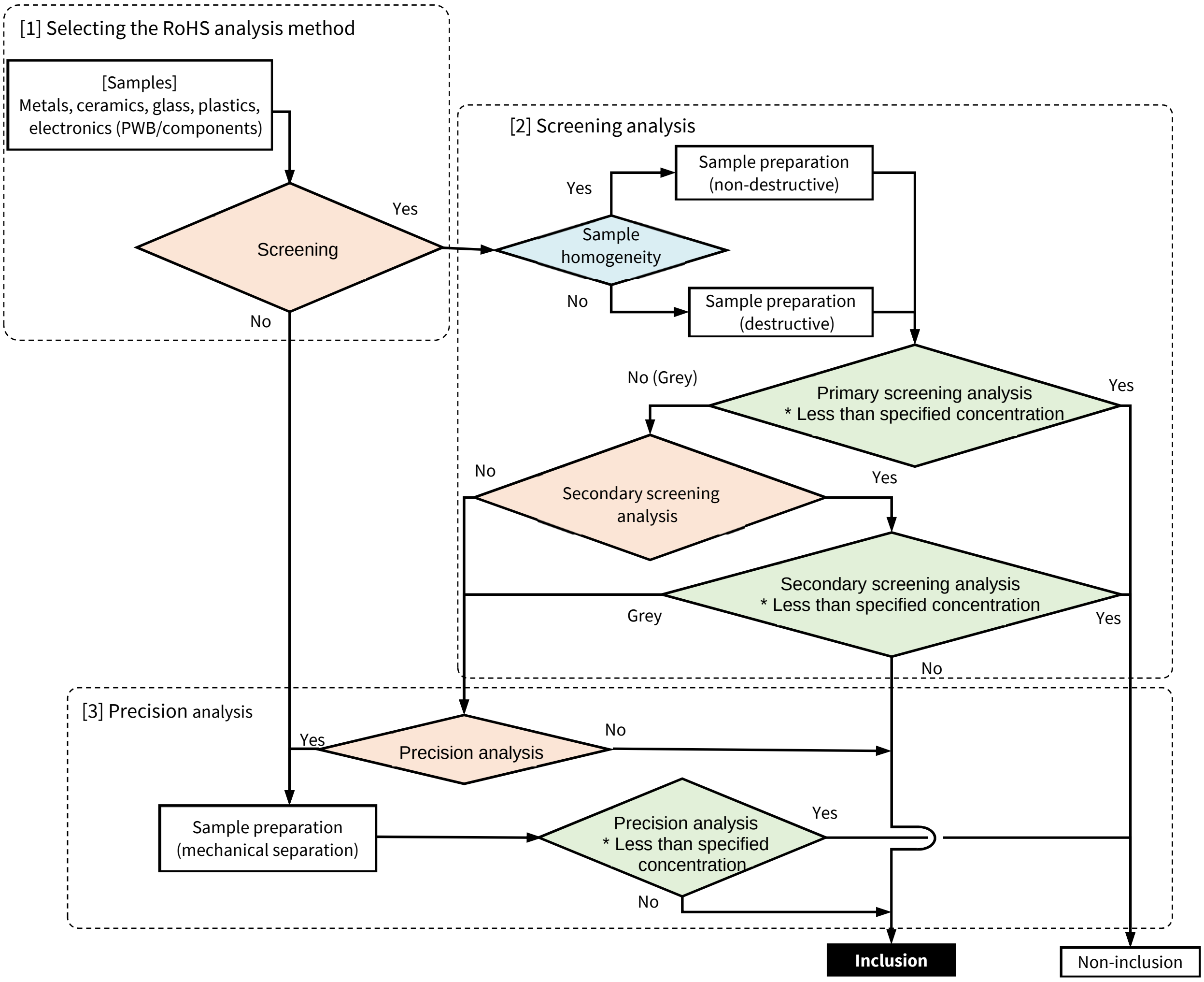
These Guidelines are structured as shown below. The results of either the screening analysis or quantitative analysis results are used to determine whether the product contains RoHS restricted substances.

Table 3 Guideline structure

Chapter	Item	Page
[1]	Selection of RoHS analysis method	13
[2]	Screening analysis (sample preparation, analysis, determination)	14
[3]	Precision analysis (sample preparation, analysis, determination)	24

(1) Conducting the analysis

The analyses outlined in these Guidelines are based on IEC 62321. As shown in Figure 1 "Flow chart of testing methods" shows, the analysis is performed by “[1] Selecting the RoHS analysis method”, and judging the acceptability of “[2] Screening analysis”, and “[3] Precision analysis”.



* Specified concentration: Cd 100ppm, Pb (Resin) 300ppm, Others 1,000ppm, (Heavy metal contained in packaging material 100ppm)

Figure 1 Flow chart of testing methods

(2) Classification by part material

The analysis method differs according to the part material, so the analysis method suitable for each method is explained in these Guidelines. As Table 4 shows, the screening analysis method differs according to the material category. Refer to section [2] Screening analysis for details.

Table 4 Classification of material for screening analysis

Category	Example
Metals, ceramics, glass	Chassis, frame, etc.
Plastics	Cabinet, cable coating (covering) etc.
Electronics, etc.	PWB, IC, LSI, etc.

(3) Classification by RoHS restricted substance

As Table 5 shows, the precision analysis method differs according to the chemical substance to be analyzed. Refer to section [3] Precision analysis for details.

Table 5 Classification of RoHS restricted substances and analysis methods

Target substance	Homogeneous material	Preparation and adjustment of measurement sample	Analysis method
Pb, Cd	Plating	Only the surface treated section of the sample is taken out by selectively polish, dissolve, or etc., and weigh the material, dissolve it with a suitable acid, etc., and analyze the dissolved solution.	AAS Atomic Absorption Spectrometry AES Atomic Emission Spectroscopy
	Solder, resin, paint, ink, pigment, etc.	Weigh the sample, dissolve it with a suitable acid, etc., and analyze the dissolved solution.	
Hg	All homogeneous materials	Weigh the sample, dissolve it with a suitable acid, etc., and analyze the dissolved solution.	CV-AAS Cold Vapor-Atomic Absorption Spectroscopy) CV-ICP-OES Cold Vapor-Inductively Coupled Plasma Optical Emission Spectroscopy
CrVI	Chromate treated metal	Add diphenylcarbazide to solution containing chromate obtained by boiling sample, of which chromate treated area has been measured, in boiling water (or warm water).	Diphenylcarbazide absorption spectrophotometric analysis
	Resin, paint, ink, pigment, electronics	Weigh the sample passed through 250 µm mesh screen after pulverization, extract CrVI with alkaline/warm water, neutralize the extraction, add diphenylcarbazide to the extraction and analyze it.	
PBB, PBDE, DEHP, DBP, BBP, DIBP	Resin	Pulverize to 500 µm or smaller, and analyze solution extracted with Soxhlet method.	GC-MS

[1] Selection of RoHS analysis method

1. What is RoHS analysis?

RoHS analysis is the procedure conducted in conformance to IEC 62321 to evaluate and determine whether a product contains RoHS restricted substances at concentrations exceeding the legal regulation threshold.

2. Types of RoHS analysis

RoHS analysis includes screening analysis that does not use chemical techniques for the preparation and analysis of the sample, and precision analysis that uses chemical techniques. If the results are determined as gray zone with the screening analysis, the inclusion is evaluated and determined with precision analysis.

3. Preparation for RoHS analysis

For both RoHS screening analysis and precision analysis, the sample is prepared with (1) Reduction (homogenizing) and (2) Preparation of sample.

(1) Disjoint (homogenizing)

The product, unit, or module comprising the product, and components are disjointed with unscrewing, cutting, pulverizing (crushing), grinding, or abrasive processes (polishing) into a "homogeneous material" that cannot be further disjointed into different materials with mechanical action. RoHS requires information on non-inclusion in a homogeneous material unit. Note that RoHS does not require separation or reduction with chemical techniques[4].

(2) Preparation of sample

The homogeneous material prepared in step (1) above is prepared into a shape suitable for analysis.

4. RoHS analysis

(1) Screening analysis

Screening analysis is an analysis method that is easy to perform. Evaluation and determination of the analysis results are categorized as "Definite inclusion", "Definite non-inclusion", and "Gray zone" in which a determination cannot be made. If the analysis results fall in the gray zone, evaluation and determination is completed with the precision analysis explained later.

(2) Precision analysis

Precision analysis is an analysis method more precise than the screening analysis, which typically uses chemical techniques. Expertise knowledge, techniques, and skills are required for all steps including preparation of the sample. Therefore, precision analysis is mainly used to determine "Inclusion" or "Non-inclusion" of a sample that is determined as "Gray zone" with the screening analysis.

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[4] Techniques using the difference of solubility into acid or solvent, etc.

5. Sharp Original Standards

Sharp original standards

Although screening analysis and precision analysis are both carried out in conformance with IEC 62321, the following seven items are set as Sharp original standards. Refer to each page for details.

Table 6 Sharp original standards

Item	Outline of Sharp original standards	Related page
(1) When plating is thinner than 0.1 µm and surface area is less than 10 mm^2	Screening analysis is difficult so perform precision analysis.	25, 30, 31
(2) When plating area is extra small and plating is ultra-thin	Direct analysis of the sample is difficult, so analysis of restricted substances in the solution in a plating bath is permitted[5]	25, 32
(3) Determination criteria for chromate analysis	The determination criteria for CrVI concentration in chromate coat are given below. • Boiling water extraction method: less than 0.10 [µg/cm^2] • Warm water extraction method: less than 0.05 [µg/cm^2]	25, 33
(4) Determination criteria for Pb in resin	The determination criteria for Pb in resin shall be 300ppm.	20
(5) Screening of PBB and PBDE in resin	Screening analysis of PBB and PBDE in resin using the following analysis methods is permitted. • Pyrolysis GC-MS: Pyro-GC-MS • Ion attached MS: IAMS • High Performance Liquid Chromatograph: HPLC	19, 23
(6) Gray zone for screening analysis of PBB and PBDE	The gray zone for screening analysis in PBB and PBDE in resin is as follows. 500ppm < analysis result ≤ 1,500ppm	20, 23
(7) Analysis by grinding micro size electronic devices into powder	If micro size electronic devices cannot be analyzed as a homogeneous material unit, an analysis by grinded powder form of such is permitted.	21

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[5] Hexavalent chromium used for chromium plating or chromate treatment is excluded.

[2] Screening analysis

1. What is screening analysis?

Screening analysis is an analysis method that is easy to perform and takes a short time to make a determination. Typically, this analysis method has a low precision, so the evaluation and determination of the analysis results are categorized as "Definite inclusion", "Definite non-inclusion", and "Gray zone" in which a determination cannot be made. If the analysis results fall in the gray zone, evaluation and determination is completed with the precision analysis explained later.

2. Screening analysis techniques

The screening analysis techniques are divided into the "elemental analysis" and "organic analysis". Elemental analysis is a method that analyzes the elements of the RoHS restricted chemical substance in homogeneous material. Organic analysis is a method that analyzes organic RoHS restricted chemical substances in the homogeneous material.

(1) Elemental analysis

1) Target substances

Elemental analysis analyzes the elements Pb, Cd, Hg, Chrome of CrVI, and bromine in PBB and PBDE in the RoHS restricted substance.

2) Analysis methods

The X-Ray Fluorescent (XRF) analysis method is used.

XRF is an analysis method that irradiates an X-ray on the elements in the sample, and obtains the concentration of elements in the sample based on the intensity of the X-ray (fluorescent X-ray) unique to the emitted elements.

The analysis results are obtained with N±3σ [unit: ppm][6]. A homogeneous material sample should be measured[7], and the element detected is determined with "N > 3σ". When "N < 3σ", the measurement time must be extended[8], and the specimen size must be increased in order to increase 3σ.

As shown in Table 7 "Outline of XRF analysis" shows, the calibration curve method, fundamental parameter (FP) method, or thin film FP method of XRF is used according to the homogeneous material.

Table 7 Outline of XRF analysis

Analysis method	Outline	Homogeneous material to be analyzed
Calibration curve method	Method of obtaining the element concentration of a sample by comparing the fluorescent X-ray intensity of a standard specimen of which the concentration is already known with the X-ray intensity of the sample.	Plastics Ceramics, glass
FP method	Method of theoretically inferring the type and concentration of elements from the fluorescent X-ray intensity	Metal
Thin film FP method	Method of identifying film thickness from intensity of fluorescent X-ray of elements comprising thin film[9], and theoretically inferring the element concentration in the thin film	Thin film (plating)

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[6] XRF is a measurement method that counts the fluorescent X-ray photons, so there may be statistical fluctuation (variation) σ. Therefore, the variation σ in the analysis results must be considered.

[7] Although processing, such as cutting to a size suitable for analysis is required, a dissolving treatment, etc., is not required.

[8] The measurement time must be quadrupled to halve 3σ.

[9] The thin film in these guidelines refers to a film thickness is less than 0.1 mm.

3) Determination

With elemental analysis screening, a determination is made with three standards "Definite non-inclusion", "Definite inclusion", and "Gray zone" as shown in Table 8. Refer to section 3 "Screening analysis procedures" for details.

Table 8 Determination of elemental analysis

RoHS restricted substance	Determination of elements analyzed with screening analysis
Pb, Cd, Hg	Determine with the three standards "Definite non-inclusion", "Definite inclusion", and "Gray zone" from the element concentration.
CrVI	Determine the CrVI "Definite non-inclusion" of CrVI from the concentration of chromium element. All states other than "Definite non-inclusion" are determined as "Gray zone".
PBB, PBDE*	Determine the "Definite non-inclusion" of PBB and PBDE from the concentration of bromine element. All states other than "Definite non-inclusion" are determined as "Gray zone".

* Note 1) Due to their composition, metals, ceramics, and glass do not contain PBB or PBDE.
2) Even if these materials are determined as gray zone, there is no need to perform a precision analysis.

(2) Organic analysis

If the RoHS restricted substance is an organic substance, then metals, ceramics, and glass are excluded from the organic analysis as they do not contain an organic substance[10]. The sample to be analyzed must be treated before the organic analysis.

1) Target substance

Table 9 shows the substances subject to organic analysis. When added to resin, these organic substances add a specific function to the resin.

Table 9 Functions of RoHS restricted organic substances

Target substance	Function on resin	Generic name
PBB, PBDE	Flame retardancy	Flame retardant
DEHP, BBP, DBP, DIBP	softening (plasticizing)	Plasticizer

2) Analysis method

The Pyro-GC-MS, HPLC-UV, or IAMS analysis method is used.

3) Determination

The categories of organic analysis screening are shown in Table 10.

Table 10 Determination of organic screening analysis

Determination	Concentration
Definite inclusion	1,500ppm or more
Gray zone	500 to 1,500ppm
Definite non-inclusion	Less than 500ppm

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[10] Metals, ceramics, and glass, etc., are processed with heat reaching several hundred degrees, so all organic substances are decomposed and are not present.

3. **Screening analysis procedures**

This chapter explains the details of each of the processes 1) Reduction and homogenizing of material, 2) Preparation of sample, 3) Selection of analysis method, and 4) Determination for each of the part types (1) Metals, ceramics, glass, (2) Plastics, and (3) Electronics.

If it is unclear whether the material of the part be screened and analyzed is (1), (2), or (3), refer to the section (2) Plastics.

(1) **Metals, Ceramics, and Glass**

Metals, ceramics, and glass are mainly used for chassis and frames, etc.

Example: Coated and plated component, chromate treated steel material, soldered circuit board, electronics component, etc.

1) Reduction and homogenizing of material

Reduce the material to a level suitable for analysis as indicated in Table 11.

Table 11 Reduction level during homogenizing of metal, ceramic, or glass material

Material	Level of reduction
A) Plain material that is not coated or plated	• Homogeneous material processed into specific material • Wires do not need to be separated or reduced any further
B) Coated or plated material	• Reduce to plating or film that can be mechanically separated • If the material has multiple layers of plating, reduce into each layer • Mechanically separate plating with crushing, etc., and reduce base material and plating layer into homogeneous materials
C) Chromate treated material	• Separate material into "chromate layer", "galvanized layer", and "steel material".
D) Soldered item	• Separate "solder", "circuit formed on circuit board", and "terminal electrodes of electronics" into homogeneous materials • If terminal electrodes of electronics, etc., are plated, separate "plating" and "terminal electrodes" into homogeneous materials
E) Others	• Reduce printing ink on part and component surfaces to homogeneous materials

2) Preparation of sample

Pretreat the homogeneous materials by cutting, etc., into sizes suitable for screening analysis as shown in Table 12.

Table 12 Thickness and area of sample suitable for analysis

Type of analysis	Analysis method	Processing and pre-treatment of sample		
		Material	Thickness	Area
Elemental analysis	XRF	Metal	1 mm or more thick	Larger than X-ray irradiation area
		Nonferrous	5 mm or more thick	

3) Screening analysis

(a) Analysis method

Table 13 shows the methods for screening analysis of metals, ceramics, and glass. Select the method according to the "thin film" or "bulk" material class and the type of chemical substance to be measured.

Table 13 Screen analysis methods for metals, ceramics, and glass

Material class		Thin film	Bulk
Defined in these Guidelines		Film on homogeneous material less than 0.1 mm thick	Solid that is not thin film
Chemical substance to be measured	Pb, Hg, Cd, CrVI	XRF thin film FP method	XRF calibration curve method or FP method
	PBB, PBDE, DEHP, BBP, DBP, DIBP	Excluded from analysis target (These substances will not be included)	

4) Determination

(a) Pb, Hg, Cd, and CrVI

Table 14 shows the "Inclusion, Non-inclusion, and Gray zone" determination criteria for the screening analysis of Pb, Hg, Cd, and CrVI in metals, ceramics, and glass. A precision analysis is performed when the determination is "Gray zone".

The 3σ value in the XRF analysis results N±3σ [unit: ppm] is fit in the determination criteria for each RoHS restricted substance, and "Inclusion, Non-inclusion, or Gray zone" is determined from the N value.

Table 14 Determination criteria for Pb, Hg, Cd, and CrVI in metals, ceramics, and glass

Substance	Determination criteria (unit: ppm)		
	Non-inclusion	Gray zone	Inclusion
Pb, Hg	N ≤ 700 - 3σ	700 - 3σ < N < 1300 + 3σ	1300 + 3σ ≤ N
Cd	N ≤ 70 - 3σ	70 - 3σ < N < 130 + 3σ	130 + 3σ ≤ N
CrVI	N ≤ 700 - 3σ	700 - 3σ < N	Determination not possible

[Example of determination]

Assume that the XRF analysis results of Pb in a certain ceramic material is N = 600 (ppm), 3σ = 20 (ppm). If 3σ is substituted in the non-inclusion determination criteria, then 700-3σ = 700-20 = 680 (ppm). The analysis results are N = 600 (ppm) < 680 (ppm), so the determination is "Non-inclusion".

When the Cr analysis results obtained with XRF are higher than the standard value, it cannot be determined whether the element is "CrVI", "trivalent chromium (CrIII)" or "metal chrome" as shown in Table 15. In this case, the determination is gray zone, and the precision analysis described later is used to make a determination.

Table 15 Cr analysis results and determination

Chrome and chromium compound	Details and determination of chromium and chromium compound
CrVI	CrVI is the generic name for a compound for which the chromium oxidation number[11] is +6. If the XRF analysis results show that the chromium element concentration is less than the standard value, it can be determined that the CrVI concentration is also less than the standard value.
CrIII	CrIII is a substitute substance for CrVI, and is the generic name for a compound for which the chromium oxidation number is +3. XRF cannot distinguish CrIII from CrVI.
Metal chrome	Hard chrome plating used for decorations, etc., is metal chrome. XRF cannot distinguish metal chrome from CrVI.

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[11] The oxidation number is obtained by classifying the valence as positive or negative.

(b) PBB, PBDE, DEHP, BBP, DBP, DIBP

Excluded from analysis targets[12].

If the screening analysis results indicate gray zone, perform section [3] Precision analysis.

(2) Plastics

Plastics are used in cabinets and cable sheaths, etc.

(Example: Sheathing material for cabinets, chassis, power cables, etc., labels, stickers, etc.)

1) Reduction and homogenizing of material

Table 16 shows the reduction level for each material.

Table 16 Reduction level during homogenizing of plastics

Material	Reduction level
a) Plain material that is not coated or plated	• Sheathing material for cabinets, chassis, power cables, etc., shall be homogeneous material without further reduction. • Power cords, etc., may be comprised of several homogeneous materials such as "conductor sheathing material" and "sheathing material for bundling each conductor". In this case, each material shall be a homogeneous material. • If the plug attached to the power cable is comprised of a different material, that plug shall be a homogeneous material.
b) Material that is coated or plated	• For labels and stickers, etc., the "main body", "adhesive", and "paint such as printing ink, etc." shall each be a homogeneous material. • If the material surface is decorated[13], such as with plating, the "decoration such as plating film," and "main body" shall each be a homogeneous material. • If the decoration has multiple layers, each layer shall be a homogeneous material. • Ink printed on the surface of parts that include a power cord, etc., shall also be a homogeneous material.

2) Preparation of sample

As Table 17 shows, the material shall be reduced to a state suitable for analysis.

Table 17 Thickness and area of plastic sample suitable for analysis

Type of analysis	Analysis method	Processing and pre-treatment of sample		
		Material	Thickness	Area
Elemental analysis	XRF	Plastic	5 mm or more thick	Larger than X-ray irradiation area
Organic analysis	Pyro-GCMS, IAMS	Crush to diameter of 500 µm or smaller		
	HPLC-UV	Crush to a diameter of 500 µm or smaller, and then extract with solvent		

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[12] If bromine (Br) is detected from ceramics or glass using XRF, check the supply chain upstream to confirm whether the component materials contain inorganic brominated compound. With this confirmation, it can be determined that the Br in the analysis results is not derived from PBB or PBDE, and that the material does not contain PBB, PBDE.

[13] Metallic decorations created with electroless plating and vapor deposition, etc., are included.

3) Screening analysis

(a) Analysis method

The screening analysis methods for plastics are shown below. If a gray zone determination is made for PBB and PBDE in plastics with the primary screening analysis, a secondary screening analysis is recommended.

i) Primary screening (XRF)

Use the methods shown in Table 18 for the screening analysis of plastics. Select the method according to the "thin film" or "bulk" material class and the type of chemical substance to be measured.

Table 18 Screening analysis methods for plastics

Material class		Thin film	Bulk
Definition in these Guidelines		Homogeneous material film less than 0.1mm thick	Solid that is not thin film
Chemical substance to be measured	Pb, Hg, Cd, CrVI	XRF thin film FP method	XRF calibration curve method, or FP method
	PBB, PBDE	Perform secondary screening analysis when determination is gray zone	
	DEHP, BBP, DBP, DIBP	Not applicable (cannot be analyzed with XRF)	

If a gray zone determination is made for PBB and PBDE in plastics with the primary screening analysis, a secondary screening analysis is recommended.

ii) Secondary screening (Pyro-GC-MS, IAMS or HPLC-UV)

Sharp original standards

Secondary screening is performed in the following cases.

- Sample for which PBB and PBDE inclusion determined to be gray zone with primary screening (XRF)
- Sample for which primary screening of DEHP, BBP, DBP, or DIBP was not performed

Perform secondary screening with Pyro-GC-MS, IAMS, or HPLC-UV shown in Table 19, and determine inclusion, non-inclusion, or gray zone. Perform a precision analysis if a gray zone determination is made.

Table 19 Chemical substances and analysis method for secondary screening of plastics

Chemical substance	Analysis method
PBB, PBDE, DEHP, BBP, DBP, DIBP	Pyro-GC-MS, IAMS or HPLC-UV

4) Determination

The following table shows the "Inclusion, Non-inclusion, and Gray zone" determination criteria for the screening analysis of plastics. A precision analysis is performed when the determination is "Gray zone".

(a) Pb, Hg, Cd, CrVI

Table 20 shows the "Inclusion, Non-inclusion, and Gray zone" determination criteria for the screening analysis of Pb, Hg, Cd, and CrVI in plastics. A precision analysis is performed when the determination is "Gray zone".

Table 20 Determination criteria for Pb, Hg, Cd, and CrVI in plastics

Substance	Determination criteria (unit: ppm)		
	Non-inclusion	Gray zone	Inclusion
Pb	N ≤ 210 - 3σ	210 - 3σ < N < 390 + 3σ	390 + 3σ ≤ N
Hg	N ≤ 700 - 3σ	700 - 3σ < N < 1300 + 3σ	1300 + 3σ ≤ N
Cd	N ≤ 70 - 3σ	70 - 3σ < N < 130 + 3σ	130 + 3σ ≤ N
CrVI	N ≤ 700 - 3σ	700 - 3σ < N	Determination not possible

Sharp original standards

In the "Report on Chemical Substances Contained in the Product" shown in Table 21, Sharp has set the inclusion of Pb in plastics at a level of 300ppm[14], which is stricter than the RoHS level. Make a determination following this standard.

Table 21 Sharp Corporation Report on Chemical Substances Contained in the Product (excerpt)

No.	Substances	Check point (Criteria)	Result
2	Lead and its compound	Content in plastics is 300ppm or less. Content in others is 1,000ppm or less. (omitted bellow)	< Applicable/ Not Applicable >

The determination criteria for Hg, Cd, and CrVI in plastics conform to (1) Metals, Ceramics, and Glass.

(b) PBB, PBDE, DEHP, BBP, DBP, DIBP

Table 22 shows the "Inclusion, Non-inclusion, and Gray zone" determination criteria for the screening analysis of PBB, PBDE, DEHP, BBP, DBP, and DIBP in plastics.

Table 22 Determination criteria for PBB, PBDE, DEHP, BBP, DBP, and DIBP in plastics

Substance	Analysis method	Determination criteria (unit: ppm)		
		Non-inclusion	Gray zone	Inclusion
PBB, PBDE	Elemental analysis	N ≤ 300 - 3σ	300 - 3σ < N	Determination not possible
	Organic analysis	N ≤ 500	500 < N < 1,500	1,500≤ N
DEHP, BBP, DBP, DIBP	Organic analysis	N ≤ 500	500 < N < 1,500	1,500≤ N

PBB and PBDE contain 1 to 10 bromine (Br) per molecule. In these Guidelines, if Br is less than 300ppm in the primary screening (elemental analysis) using XRF, the result is determined as "Non-inclusion" with no PBB or PBDE having one bromine per bromine molecule. All other cases are determined as “Gray zone”.

Sharp original standards

If the PBB and PBDE elemental analysis is determined to be "Gray zone", secondary screening using organic analysis is recommended. The Sharp original standards shall be applied as the determination criteria for organic analysis of PBB and PBDE.

If the screening analysis of PBB, PBDE, DEHP, BBP, DBP, and DIBP in plastics is determined as "Gray zone", perform the [3] Precision analysis.

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[14] Based on the Settlement Agreement of the California (US) Proposition 65 warning label lawsuit.

(3) **Electronics**

Electronics refer to miniature electronics such as chip capacitors and LSI that are mounted on a PWB, etc.

1) Reduction and homogenizing of material

Miniature electronics have the following issues, and can be difficult to measure as a homogeneous material.

- It is difficult to collect[15] sufficient homogeneous material for analysis from miniature electronics on module or unit.
- Many electronics[16] do not have uniform chemical composition, so separation into homogeneous materials with mechanical methods is difficult.

Based on the above issues, it is best to obtain the pre-processing or pre-manufacturing members of the miniature electronics, and analyze them in homogeneous material units. In this case, the analysis methods for metals, ceramics, glass, or plastics explained earlier are applied.

Analysis performed by crushing miniature electronics is permitted if analysis of delivered members is difficult. However, the analysis results will be the average value of the configuration materials. In this case, the evaluation and determination of analysis results must be based on knowledge of the structure and materials of the configuration parts.

2) Preparation of sample

Reduce the sample to a state suitable for analysis as shown in Table 23.

Table 23 Processing and pre-treatment of electronics to state suitable for analysis

Type of analysis	Analysis method	Processing and pre-treatment of sample	
Elemental analysis	XRF	Delivered component before processing	Apply the methods for metals, ceramics, glass, or plastics
		Miniature electronics	Crush to powder of diameter of 250μm or less ★
Organic analysis	Pyro-GCMS, IAMS	Crush to diameter of 500μm or less	
	HPLC-UV	Crush to a diameter of 500μm or smaller, and then extract with solvent	

★ Sharp original standards

3) Screening analysis

(a) Analysis method

This section explains the methods for analyzing miniature electronics. Use the methods for metals, ceramics, glass, or plastics if homogeneous materials, such as delivered pre-processing components can be obtained.

i) **Primary screening (XRF)**

Table 24 shows the screening analysis methods for electronics. Analyzing powder with XRF performs elemental analysis of electronics. If a gray zone determination is made for PBB and PBDE in electronics with the primary screening analysis, a secondary screening analysis is recommended.

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[15] For example, a laminated ceramic chip capacitor with a volume of 0.01 mm^3 or less is configured of an external (terminal) electrode, conductor layer, and internal electrode. The quantity required for screening analysis is several 1000 to several 100,000 units, which is not practical.

[16] For ICs and LSIs, it is difficult to mechanically reduce elements such as the transistors and the metal in the wiring.

Table 24 Analysis methods for primary screening of electronics

Chemical substance	Analysis method
Pb, Hg, Cd, CrVI	XRF calibration curve method
PBB, PBDE,	Perform secondary screening analysis if determination is gray zone
DEHP, BBP, DBP, DIBP	Not applicable (cannot be analyzed with XRF)

ii) Secondary screening (Pyro-GC-MS, IAMS or HPLC-UV)

Sharp original standards

Secondary screening is performed in the following cases.

- Sample for which PBB and PBDE inclusion determined to be gray zone with primary screening (XRF)
- Sample for which primary screening of DEHP, BBP, DBP, or DIBP was not performed

Perform secondary screening with Pyro-GC-MS, IAMS, or HPLC-UV shown in Table 25, and determine "Inclusion, Non-inclusion, and Gray zone". Perform a precision analysis if a gray zone determination is made.

Table 25 Chemical substances and analysis methods for secondary screening of electronics

Chemical substance	Analysis method
PBB, PBDE, DEHP, BBP, DBP, DIBP	Pyro-GC-MS, AMS or HPLC-UV

4) Determination

Table 26 shows the "Inclusion, Non-inclusion, and Gray zone" determination criteria for the screening analysis of electronics. A precision analysis is performed when the determination is "Gray zone".

The safety coefficient for the electronics determination criteria is set at 50% based on IEC 62321.

(a) Pb, Hg, Cd, CrVI

Table 26 Determination criteria for Pb, Hg, Cd, and CrVI in electronics

Substance	Determination criteria (unit: ppm)		
	Non-inclusion	Gray zone	Inclusion
Pb, Hg	$N \leq 500 - 3\sigma$	$500 - 3\sigma < N < 1{,}500 + 3\sigma$	$1{,}500 + 3\sigma \leq N$
Cd	N ≤ detection lower limit	Detection lower limit < N < $150 + 3\sigma$	$150 + 3\sigma \leq N$
CrVI	$N \leq 500 - 3\sigma$	$500 - 3\sigma < N$	Determination not possible

If Cd is not detected in powdered electronics, the determination shall be Non-inclusion.

(b) PBB, PBDE, DEHP, BBP, DBP, and DIBP

Table 27 shows the "Inclusion, Non-inclusion, and Gray zone" determination criteria for the screening analysis of PBB, PBDE, DEHP, BBP, DBP, and DIBP in electronics.

Table 27 Determination criteria for PBB, PBDE, DEHP, BBP, DBP, and DIBP in electronics

Substance	Analysis method	Determination criteria (unit: ppm)		
		Non-inclusion	Gray zone	Inclusion
PBB, PBDE	Elemental analysis	N ≤ 250 - 3σ	250 - 3σ < N	Determination not possible
	Organic analysis	N ≤ 500	500 < N < 1,500	1,500≤ N
DEHP, BBP, DBP, DIBP	Organic analysis	N ≤ 500	500 < N < 1,500	1,500≤ N

Sharp original standards

If the PBB and PBDE elemental analysis is determined to be "Gray zone", secondary screening using organic analysis is recommended. The Sharp original standards shall be applied as the determination criteria for organic analysis of PBB and PBDE.

If the screening analysis of PBB, PBDE, DEHP, BBP, DBP, and DIBP in electronics is determined as "Gray zone", perform the [3] Precision analysis.

[3] Precision Analysis

1. What is precision analysis?

Precision analysis is an analysis used to evaluate and determine a sample on which a screening analysis was not performed, or that was determined as "Gray zone" in the screening analysis. Either "Definite inclusion" or "Definite non-inclusion" is evaluated with the precision analysis.

2. Precision analysis methods

In the same manner as screening analysis, the precision analysis methods are divided into "(1) Elemental analysis" and "(2) Organic analysis" as shown in Table 28.

Table 28 Analysis techniques for precision analysis, target substances, and details of analysis methods

Analysis method	Target substance (element)	Details of analysis methods used for precision analysis
(1) Elemental analysis	Pb, Cd, Hg, Cr	AAS, AES, ICP-OES, ICP-MS, etc.
	CrVI	Diphenylcarbazide absorption spectrophotometry Method of preparing the sample differs for chromate treated parts and all other parts
(2) Organic analysis	PBB, PBDE, DEHP, BBP, DBP, DIBP	Solvent extraction GCMS method, etc.

3. Precision analysis procedures

The weight of a sample reduced to homogeneous material, and the weight of the solvent or acid used to dissolve the sample are weighted, to constantly control the concentration of sample in the analysis solution. The sample is analyzed with an analysis method that corresponds to the target substance.

4. **Performing precision analysis**

The precision analysis is performed with the following process: 1) Collecting the sample, 2) Preparing for analysis (reduction and preparation of sample, weighing, selecting and weighing the acid, dissolution of sample, etc.), 3) Analyzing the substances, and 4) Determination. As shown in Table 29, the implementation method is divided into seven types according to the sample form and the analysis method. The sample preparation method varies according to the sample material, substance to be analyzed, and the sample form.

Table 29 Sample materials, target substances, forms, and analysis methods for precision analysis

Sample material	Target substance	Sample form	Analysis method	Reference page
Metal Ceramic Glass Plastic Electronics	Pb Cd Hg Cr	Bulk[17]	Elemental analysis of bulk acid solution	(1) Inorganic substance in bulk (Pb, Cd, Hg, Cr)
		General plating, etc.	Elemental analysis of plating acid solution	(2) Surface treated part such as general plating, etc.
		Film thickness less than 0.1 µm and surface area less than 10 mm^2	Elemental analysis of dissolving solution dissolved with bulk and acid	(3) When plating is thinner than 0.1 µm and surface area is less than 10 mm^2 ★
		Ultra-thin plating on extra small area	Elemental analysis of plating bath solution	(4) When plating area is extra small and plating is ultra-thin ★
Metal (Chromate treatment)	CrVI	Chromate treatment	Boiling water (or warm water) extraction - Diphenylcarbazide absorption spectrophotometry	(5) Analysis of CrVI in chromate of chromate treated metal ★
Plastic Electronics	PBB PBDE DEHP BBP DBP DIBP	Bulk	GC-MS	(6) Analysis of organic substances in plastic or electronics
	CrVI	Bulk	Alkaline extraction-Diphenylcarbazide absorption spectrophotometry	(7) Analysis of CrVI in plastic or electronics

★ Sharp original standards

(1) **Inorganic substance in bulk (Pb, Cd, Hg, Cr)**

This section explains the procedures for qualitative analysis of inorganic substances in the bulk materials (metals, ceramics, glass, plastics, or electronics) shown in Table 30.

Table 30 Substances to be analyzed in bulk materials, and analysis method

Sample material	Target substance	Sample form	Analysis method
• Metal, ceramic, glass • Plastic • Electronics	Pb, Cd, Hg, Cr	Bulk	Elemental analysis of acid solution

Note) The Cr analysis result obtained here is the same Cr element concentration as the screening analysis, and is not the CrVI concentration.

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[17] Bulk in these Guidelines refers to solids that are not a thin film.

Because metals, ceramics, and glass are heated to a temperature of several hundred degrees during the manufacturing process, they contain no organic substances. Therefore, analysis of organic substances in metals, ceramics, or glass is not required.

1) Collecting the sample

Collect a sample that is an average sample to represent the measurement section.

2) Preparing for analysis

(a) Reducing and preparing the sample

Reduce the sample into homogeneous material, and cut or break down into a size suitable for weighing or dissolving.

(b) Weighing the sample

Weigh the cut and reduced homogeneous material with a precision balance.

(c) Selecting and weighing the acid

Table 31 shows the recommended acids for each sample material. The acid used differs according to the elements present in the sample. Refer to IEC 62321 for details. Weigh the selected acid for dissolving the sample.

Table 31 Sample bulk materials and acids used for dissolution

Sample material	Acid
Steel, copper, aluminum, solder	Aqua regia, nitric acid, hydrochloric acid, hydrofluoric acid, hydrogen peroxide
Gold, platinum, palladium, ceramic	Aqua regia
Plastic	Sulfuric acid[18], hydrogen peroxide, nitric acid, hydrochloric acid
Glass	Nitric acid, hydrofluoric acid
Silver	Nitric acid
Others	Various acids

(d) Dissolving the sample

Dissolve the weighed sample in the acid recommended for each method, and use that dissolved solution for analysis. As Table 32 shows, the dissolution method varies according to the type of element to be analyzed.

Table 32 Substances to be measured in bulk material, and dissolution methods

Element	Dissolution method
Pb, Cd, Cr	The sample is dissolved with sealed microwave digestion or open acid decomposition. These Guidelines recommend the microwave digestion method.
Hg	Mercury has a high volatility compared to other metals. The element could be lost if it is heated and vaporized during sample preparation, so use the microwave digestion method.

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[18] Lead is not suitable for quantitative analysis as it will react with sulfuric acid and generate lead sulfate that does not dissolve easily in water.

3) Analyzing the substances

As Table 33 shows, the analysis method differs according to the element to be analyzed.

Table 33 Element to be analyzed in bulk material, and analysis method

Element	Analysis method
Pb, Cd, Cr	• Atomic Absorption Spectrometry (AAS) • Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) • Flameless Atomic Absorption Spectroscopy (FLAAS) • Inductively Coupled Plasma - Mass Spectrometry (ICP-MS)
Hg	• Cold Vapor-Atomic Absorption Spectroscopy (CV-AAS) • Cold Vapor-Inductively Coupled Plasma Optical Emission Spectroscopy (CV-ICP-OES) • Inductively Coupled Plasma - Mass Spectrometry (ICP-MS)

4) Determination

Determine inclusion or non-inclusion based on the inclusion standards shown in Table 34.

Table 34 Element to be analyzed in bulk material, and determination of analysis results

Element	Determination criteria
Pb, Hg	1,000ppm
Cd	100ppm

Note) The Cr analysis result obtained here is the same Cr element concentration as the screening analysis, and is not the CrVI concentration. The result is not used for the determination.

(2) Surface-treated parts such as general plating

This section shows the procedures for performing quantitative analysis of elements in surface treated parts, such as general plating.

Table 35 Substances to be analyzed in surface treated parts, and analysis method

Sample material	Target substance	Sample form	Analysis method
• Metal • Ceramic • Glass • Plastic • Electronics	Pb Cd Hg Cr	General plating, etc.	Elemental analysis of plating acid solution

Note) The Cr analysis result obtained here is the same Cr element concentration as the screening analysis, and is not the CrVI concentration.

1) Collecting the sample

Collect a sample that is an average sample to represent the measurement section.

2) Preparing for analysis

(a) Reducing and preparing the sample

Selectively crush and dissolve, etc., only the surface treated section of the sample, and extract as a homogeneous material.

(b) Weighing the sample

Weigh the homogeneous material removed above with a precision balance.

(c) Selecting and weighing the acid

Table 36 shows the recommended acids for each sample material. The acid used differs according to the elements present in the sample. Refer to IEC 62321 for details. Weigh the selected acid for dissolving the sample.

Table 36 Material of surface treated parts, and acids used for dissolution

Sample material	Acid
Steel, copper, aluminum, solder	Aqua regia, nitric acid, hydrochloric acid, hydrofluoric acid, hydrogen peroxide
Gold, platinum, palladium, ceramic	Aqua regia
Plastics	Sulfuric acid, hydrogen peroxide, nitric acid, hydrochloric acid
Glass	Nitric acid, hydrofluoric acid
Silver	Nitric acid
Others	Various acids

(d) Dissolving the sample

Dissolve the weighed sample in the acid recommended for each method, and use that dissolved solution for analysis. As Table 37 shows, the dissolution method varies according to the type of element to be analyzed.

Table 37 Substances to be measured in surface treated part, and dissolution methods

Element	Dissolution method
Pb, Cd, Cr	The sample is dissolved with sealed microwave digestion or open acid decomposition. These Guidelines recommend the microwave digestion method.
Hg	Mercury has a high volatility compared to other metals. The element could be lost if it is heated and vaporized during sample preparation, so use the microwave digestion method.

3) Analyzing the substances

As Table 38 shows, the analysis method differs according to the element to be analyzed.

Table 38 Element to be analyzed in surface treated part, and analysis methods

Element	Analysis method
Pb, Cd, Cr	• Atomic Absorption Spectrometry (AAS) • Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) • Flameless Atomic Absorption Spectroscopy (FLAAS) • Inductively Coupled Plasma - Mass Spectrometry (ICP-MS)
Hg	• Cold Vapor-Atomic Absorption Spectroscopy (CV-AAS) • Cold Vapor-Inductively Coupled Plasma Optical Emission Spectroscopy (CV-ICP-OES) • Inductively Coupled Plasma - Mass Spectrometry (ICP-MS)

4) Determination

Determine inclusion or non-inclusion based on the inclusion standards shown in Table 39.

Table 39 Element to be analyzed in surface treatment part, and determination criteria

Element	Determination criteria
Pb, Hg	1,000ppm
Cd	100ppm

Note) The Cr analysis result obtained here is the same Cr element concentration as the screening analysis, and is not the CrVI concentration. The result is not used for the determination.

(3) When plating is thinner than 0.1 µm and surface area is less than 10mm^2

Sharp original standards

This section explains the procedures for qualitative analysis of elements when the plating is thinner than 0.1µm and surface area is less than 10mm^2 as shown in Table 40.

Table 40 Substances to be analyzed in plating thinner than 0.1µm and surface area less than 10 mm^2, and analysis methods

Sample material	Target substance	Sample form	Analysis method
• Metal, ceramic, glass • Plastic • Electronics	Pb, Cd, Hg, Cr	Plating thinner than 0.1 µm, and surface area less than 10 mm^2	Elemental analysis of dissolving solution dissolved with bulk and acid

Note) The Cr analysis result obtained here is the same Cr element concentration as the screening analysis, and is not the CrVI concentration.

With the Sharp original standards, if the plating is thinner than 0.1 µm and the surface area is less than 19 mm^2, it is difficult to perform a screening analysis, so the following bulk analysis is permitted.

Sharp original standards

1) Collecting the sample

Collect a sample that is an average sample to represent the measurement section.

2) Preparing for analysis

(a) Reducing and preparing the sample

Reduce the sample into homogeneous material, and cut or break down into a size suitable for weighing or dissolving.

(b) Weighing the sample

Weigh the cut and reduced homogeneous material with a precision balance.

(c) Selecting and weighing the acid

Table 41 shows the recommended acids for each sample material. The acid used differs according to the elements present in the sample. Refer to IEC 62321 for details. Weigh the selected acid for dissolving the sample.

Table 41 Sample material of plating film with thickness of less than 0.1 µm and surface area of less than 10 mm^2, and acid used for dissolution

Sample material	Acid
Steel, copper, aluminum, solder	Aqua regia, nitric acid, hydrochloric acid, hydrofluoric acid, hydrogen peroxide
Gold, platinum, palladium, ceramic	Aqua regia
Plastics	Sulfuric acid, hydrogen peroxide, nitric acid, hydrochloric acid
Glass	Nitric acid, hydrofluoric acid
Silver	Nitric acid
Others	Various acids

(d) Dissolving the sample

Dissolve the weighed sample in the acid recommended for each method, and use that dissolved solution for analysis. As Table 42 shows, the dissolution method varies according to the type of element to be analyzed.

Table 42 Substance to be measured in plating film with thickness of less than 0.1 µm and surface area of less than 10 mm^2, and dissolution methods

Element	Dissolution method
Pb, Cd, Cr	The sample is dissolved with sealed microwave digestion or open acid decomposition. These Guidelines recommend the microwave digestion method.
Hg	Mercury has a high volatility compared to other metals. The element could be lost if it is heated and vaporized during sample preparation, so use the microwave digestion method.

3) Analyzing the substances

As Table 43 shows, the analysis method differs according to the element to be analyzed.

Table 43 Element to be analyzed in plating film with thickness of less than 0.1 µm and surface area of less than 10 mm^2, and analysis methods

Element	Analysis method
Pb, Cd, Cr	• Atomic Absorption Spectrometry (AAS) • Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) • Flameless Atomic Absorption Spectroscopy (FLAAS) • Inductively Coupled Plasma - Mass Spectrometry (ICP-MS)
Hg	• Cold Vapor-Atomic Absorption Spectroscopy (CV-AAS) • Cold Vapor-Inductively Coupled Plasma Optical Emission Spectroscopy (CV-ICP-OES) • Inductively Coupled Plasma - Mass Spectrometry (ICP-MS)

4) Determination

Determine inclusion or non-inclusion based on the inclusion standards shown in Table 44.

Table 44 Element to be analyzed in plating film with thickness of less than 0.1 µm and surface area of less than 10 mm^2, and determination criteria

Element	Determination criteria
Pb, Hg	1,000ppm
Cd	100ppm

Note) The Cr analysis result obtained here is the same Cr element concentration as the screening analysis, and is not the CrVI concentration. The result is not used for the determination.

These determination criteria are not set with IEC 62321. Sharp has set the above determination criteria based on the "EU Commission's RoHS Implementation Guidance Document"[19].

Sharp original standards

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[19] "RoHS Enforcement Guidance Document" (Version 1 issued May 2006) https://www.epa.ie/pubs/advice/waste/rohs/RoHS%20Enforcement%20Guidance%20Document%20-%20v%201%20May%2020061.pdf

(4) When plating area is extra small and plating is ultra-thin

Sharp original standards

This section explains the procedures for qualitative analysis of elements when the "plating is ultra-thin on an extra small area" as shown in Table 45, and analysis with "(2) Surface treated part such as general plating", or "(3) When plating is thinner than 0.1 µm and surface area is less than 10 mm^2" is difficult. In this case, the concentration of the target substance in the plating bath solution is analyzed.

Table 45 Substance to be analyzed when plating area is extra small and plating is ultra-thin, and analysis methods

Sample material	Target substance	Sample form	Analysis method
• Metal, ceramic, glass • Plastic • Electronics	Pb, Hg Cd, Cr	Ultra-thin plating on extra small area	Analysis of plating bath solution

Note) The Cr analysis result obtained here is the same Cr element concentration as the screening analysis, and is not the CrVI concentration.

1) Collecting the sample

Collect the plating bath solution.

2) Preparing for analysis

Use the plating bath solution as the sample.

3) Analyzing the substances

As Table 46 shows, the analysis method differs according to the element to be analyzed.

Table 46 Element to be analyzed when plating area is extremely small and plating is ultra-thin, and analysis method

Element	Analysis method
Pb, Cd, Cr	• Atomic Absorption Spectrometry (AAS) • Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) • Flameless Atomic Absorption Spectroscopy (FLAAS) • Inductively Coupled Plasma - Mass Spectrometry (ICP-MS)
Hg	• Cold Vapor-Atomic Absorption Spectroscopy (CV-AAS) • Cold Vapor-Inductively Coupled Plasma Optical Emission Spectroscopy (CV-ICP-OES) • Inductively Coupled Plasma - Mass Spectrometry (ICP-MS)

4) Determination

Sharp original standards

Sharp uses original standards based on Table 47 "Plating bath solution control concentration" to determine inclusion and non-inclusion. The plating bath solution control concentration is 1/100 of the RoHS allowable concentration.

Table 47 Elements to be analyzed when plating area is extremely small and plating is ultra-thin, and determination criteria

Target element	Determination criteria for plating bath solution control concentration
Cd	1ppm
Pb, Hg	10ppm

Note) The Cr analysis result obtained here is the same Cr element concentration as the screening analysis, and is not the CrVI concentration. The result is not used for the determination.

(5) Analysis of CrVI in chromate applied on chromate treated metal

This section explains the procedures for quantitative analysis of CrVI in chromate applied on chromate treated metal.

Table 48 Analysis of CrVI in chromate applied on chromate treated metal

Sample material	Target substance	Sample form	Analysis method
• Metal (Chromate treatment)	CrVI	Chromate treatment	Boiling water (or warm water) extraction - Diphenylcarbazide absorption spectrophotometry

1) Collecting the sample

Collect a sample so that the surface area[20] is 50 cm^2 (20 to 30 cm^2).

2) Preparing for analysis

(a) Extracting the CrVI

Sharp original standards

As shown in Table 49, immerse the sample in 100°C boiling water (or 80°C warm water), and extract for 10 minutes.

IEC 62321 specifies the boiling water extraction method. However, as there are risks of burns with boiling water, the Sharp original standards allow use of the warm water extraction method.

Note that with the warm water extraction method, the CrVI extraction efficiency will be 50%.

Table 49 Methods of extracting CrVI in chromate applied on chromate treated metal

Item	Boiling water extraction method (IEC 62321)	Warm water extraction method Sharp original standards
Extraction water temperature (°C)	100	80
Extraction time (minute)	10 ± 0.5	10 ± 0.5
Sample surface area (cm^2)	50 ± 5	25 ± 5
Extraction water volume (ml)	50	30
Extraction efficiency (%)	100	50

(b) Weighing the sample

Remove the sample after extraction. Add pure water to the extraction solution to make 50 ml (30 ml), and use the solution for the analysis.

3) Analyzing the substances

Selectively quantify only the CrVI from the sample bath solution using diphenylcarbazide absorption spectrophotometry as shown in Table 50.

The CrVI extraction amount [µg/cm^2] is calculated from the extracted CrVI amount and sample surface area.

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[20] The screw surface area can be calculated with the surface area calculation equation given in IEC62321, or with the surface area calculation equation using the schematic diagram and approximation equation given in the Annex of these Guidelines, etc.

Table 50 Method of analyzing CrVI in chromate applied on chromate treated metal

Target substance	Analysis method
CrVI	Boiling water (or warm water) extraction - Diphenylcarbazide absorption spectrophotometry

4) Determination

Determine inclusion or non-inclusion based on the inclusion standards shown in Table 51.

Table 51 Method of extracting CrVI in chromate applied on chromate treated metal, and determination criteria

Extraction method	Determination criteria
Boiling water extraction method IEC 62321	0.10 [μg/cm^2]
Warm water extraction method Sharp original standards	0.05 [μg/cm^2]

(6) Analysis of organic substances in plastic or electronics

This section explains the procedures for quantitative analysis of organic substances in plastics or electronics shown in Table 52.

Table 52 Substances to be analyzed in plastics and electronics, and analysis method

Sample material	Target substance	Sample form	Analysis method
• Plastic • Electronics	PBB, PBDE, DEHP, BBP, DBP, DIBP	Bulk	GC-MS

1) Collecting the sample

Collect a sample that is an average sample to represent the measurement section.

2) Preparing for analysis

(a) Reducing and preparing the sample

Reduce the sample into homogeneous material, and cut or break down into a size suitable for weighing or dissolving.
Crush the reduced homogeneous materials to spheres with a diameter of 500 µm or smaller.

(b) Weighing the sample

Weigh the crushed homogeneous material with a precision balance.

(c) Extraction

To extract the sample, perform "Selecting the solvent and extraction method", and then perform extraction.

i) Selecting the solvent and extraction method

Select the solvent and surrogate suitable for the extraction method shown in Table 53.
Refer to "Annex IV" for details on the extraction precautions, solvent, surrogate, and re-precipitation.

Table 53 Extraction method, and required solvents and reagents

Extraction method	Extraction solvent	Surrogate addition	Re-precipitation solvent
Soxhlet extraction	Poor solvent	Required	Not required
Ultrasonic extraction	Good solvent	Required	Poor solvent

ii) Performing extraction

Perform extraction with the Soxhlet extraction or Ultrasonic extraction method selected in step i). Refer to Annex IV for details on extraction precautions.

iii) Removal of polymer elements

Remove the polymer elements from the extraction solution using silica gel, etc., and use the remaining solution for analysis.

3) Analyzing the substances

As Table 54 shows, the extraction solution is analyzed with GCMS.

Table 54 Substance to be analyzed in plastics and electronics, and analysis method

Target substance	Analysis method
PBB, PBDE, DEHP, BBP, DBP, DIBP	GC-MS

4) Determination

Determine inclusion or non-inclusion based on the inclusion standards shown in Table 55.

Table 55 Substance to be analyzed in plastics and electronics, and determination criteria

Target substance	Determination criteria
PBB, PBDE, DEHP, BBP, DBP, DIBP	1,000ppm

(7) Analysis of CrVI in plastic or electronics

This section explains the procedures for quantitative analysis of CrVI in the plastics and electronics shown in Table 56.

Table 56 Analysis method of CrVI in plastic or electronics

Sample material	Target substance	Sample form	Analysis method
• Plastic • Electronics	CrVI	Bulk	Alkaline extraction - Diphenylcarbazide absorption spectrophotometry

1) Collecting the sample

Collect a sample that is an average sample to represent the measurement section.

2) Preparing the sample

Crush the collected homogeneous material, pass it through a sieve (250 µm), and use as the sample. After weighing, extract with alkaline and warm water.

(a) Weighing the sample

Weigh the sample sifted in step 2) above.

(b) Extraction

Extract the sample with alkaline and warm water.

The CrVI extraction efficiency differs greatly according to the resin. The acceptable extraction method for each resin material is shown in Table 57.

Table 57 Acceptable methods of extracting CrVI from materials in plastic and electronics

Resin material	PVC, ABS	EVAC, PE	Others
Acceptability	Acceptable	Not acceptable	Experimentally obtain extraction efficiency and compensate

(c) pH adjustment

After extracting, remove the sample, and use nitric acid to adjust the pH of the extraction solution to 7.5±0.5. Use the remaining solution as the sample.

3) Analysis method

Selectively quantify the CrVI in the sample solution using the diphenylcarbazide absorption spectrophotometry shown in Table 58.

Table 58 Method of analyzing CrVI in plastic and electronics

Target substance	Analysis method
CrVI	Alkaline extraction - Diphenylcarbazide absorption spectrophotometry

4) Determination

Convert the quantification results to the CrVI content [µg] in the homogeneous material / sample weight [g], and compare with the determination criteria shown in Table 59.

Table 59 Determination criteria for CrVI in plastic or electronics

Target substance	Determination criteria
CrVI	1,000ppm

[4] Handling the Analysis Data

Please attach the “Analysis data” obtained with these Guidelines and the “Report on Chemical Substances Contained in the Product” to the specifications.

Note that if the determination is "**Inclusion**", SHARP will not adopt the corresponding part as a general rule.

For more information on these Guidelines:

SHARP Corporation
Environmental Promotion Group,
Quality and Environmental Promotion Unit
E-mail: chem.epg@sharp.co.jp

Annex I

Screening criteria for PBB and PBDE

Figure 2 shows the structural formula of typical Mono BB and Mono BDE with the minimum number of bromine Br=1, and Deca BB and Deca BDE with the maximum number of bromine Br=10 in PBB and PBDE.

PBB

Mono Brominated Biphenyl
Mono BB (Br=1)

Deca Brominated Biphenyl
Deca BB (Br=10)

PBDE

Mono Brominated Diphenyl Ether
Mono BDE (Br=1)

Deca Brominated Diphenyl Ether
Deca BDE (Br=10))

Figure 2 Structural formula of Mono BB, Deca BB, Mono BDE, and Deca BDE

Table 60 shows the molecular weight and number of Br in Mono BB, Deca BB, Mono BDE, and Deca BDE, the amount of Br in one molecule, and the BR composition ratio. Mono BDE has the smallest number of Br and the smallest Br composition ratio of 32%.

Table 60 Molecular weight of Mono BB, Deca BB, Mono BDE, and Deca BB, and the Br composition ratio

Substance	Molecular weight	Number of Br	Amount of Br in one molecule	Br composition ratio
Mono BB	233.1	1	79.9	34%
Deca BB	943.2	10	799.0	85%
Mono BDE	249.1	1	79.9	32%
Deca BDE	959.2	10	799.0	83%

When the Mono BDE having the smallest Br composition ratio is 1,000ppm, the Br measured with XFR is 320ppm. Therefore, if Br is less than 300ppm when measured with XFR, it can be determined that the PBB and PBDE are less than the standard value.

Annex II

Concentration of CrVI in chromate coating obtained from concentration of CrVI in extraction water

1. Characteristics of chromate coating

Chromate coating has a complex multinuclear complex structure. It is known that the coating thickness and specific gravity change depending on the ambient humidity.

2. Characteristics of hot water extraction method

The hot water extraction method obtains the mass of CrVI extracted from the sample surface. Therefore, the measurement results are obtained as the concentration [μg/cm^2] of CrVI per unit area of the sample.

3. Unit conversion

Conformity to RoHS Directives is determined by the concentration of CrVI in the homogeneous material. The concentration [μg/cm^2] of CrVI per unit area obtained with the hot water extraction method must be converted to the concentration of CrVI in the homogeneous material. The chromate coating thickness and coating specific gravity are required for the conversion, but the calculation is difficult because the coating thickness and specific gravity of the chromate coating change depending on the ambient humidity.

4. IEC 62321 determination standards

1) Detection lower limit

The detection lower limit of CrVI in the extraction water[21] with diphenylcarbazide absorption spectrophotometry is 0.02 [mg/kg].

2) IEC 62321 Qualitative determination criteria

Table 61 shows the IEC 62321 qualitative determination criteria.

Table 61 Determination criteria in IEC 62321

CrVI concentration measured with diphenylcarbazide absorption spectrophotometry	Qualitative determination criteria
Less than 0.10 [μg/cm^2]	Non-inclusion
0.10 to 0.13 [μg/cm^2]	Gray zone The sample surface is measured three times, and determination is made with the average value. If the average value is in the gray zone, it is interpreted as Inclusion.
0.13 [μg/cm^2] or more	Inclusion

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[21] This refers to the concentration in the “extraction water”, and is not the concentration of the chromate coating.

5. Assumption calculation

If it is assumed that the chromate coating thickness is 0.25 [µm] and the specific gravity is [4.0g/cm^3], when the CrVI concentration per unit area is 0.10 [µg/cm^2], the CrVI concentration in the chromate coating will be 0.1 [wt%].

In the above assumption, it is calculated that the "approximate value of CrVI concentration in chromate coating as 0.02 [wt%]" from the "assumed coating thickness and specific gravity values" and the "CrVI detection lower limit concentration 0.02 [mg/kg] in extraction water", and this assumed value is obtained. The outline is indicated below.

i) Recommended conditions for analysis sample

Surface area 50 [cm^2] and extraction water 50 [mL] (= 0.05[kg])

ii) Detection lower limit concentration and CrVI amount

There is 0.001 [mg] of CrVI in the CrVI detection lower limit concentration 0.02 [mg/kg] in extraction water 50 [ml] (= 0.05 [kg]).

$$0.02 \text{ [mg/kg]} \times 0.05 \text{ [kg]} = 0.001 \text{ [mg]}$$

iii) Detection lower limit value of CrVI per unit area

Based on i) and ii) above, the detection lower limit value per CrVI unit area (=1 [cm^2]) is 0.02 [µg/cm^2].

$$0.001 \text{ [mg]} / 50 \text{ [cm}^2\text{]} = 0.02 \text{ [µg/cm}^2\text{]}$$

iv) Range of chromate coating properties

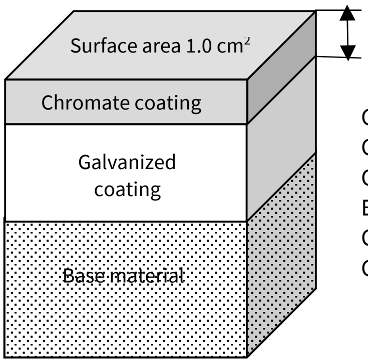
The coating thickness and specific gravity of the chromate coating are undefined, but it is said that the coating thickness is 0.2 to 0.5 [µm], and the specific gravity is 2.0 to 5.0 [g/cm^3].

From these values, we assume that the chromate coating thickness is 0.25 [µm], and the specific gravity is [4.0g/cm^3].

v) Assumption calculation

The following parameters are used for calculation.

- Assumed value: Coating thickness 0.25 [µm], specific gravity [4.0g/cm^3]
- Detection lower limit per unit area of CrVI obtained in iii): 0.02 [µg/cm^2]
  We can calculate that the concentration of CrVI in this chromate coating is 0.02 [wt%] (=200 [ppm]).



Assumed value: Coating thickness 0.25 µm, specific gravity 4.0 g/cm^3

Coating area…1.0 [cm^2]
Coating volume…1.0 [cm^2] x 0.25 [µm] = 0.25 x 10^{-4} [cm^3]
Coating mass…0.25 x 10^{-4} [cm^3] x 4.0 [g/cm^3] = 1.0 x 10^{-4} [g]
Extraction efficiency…100%
CrVI in extraction solution = 0.02 [mg/kg] = 0.02 [µg/g]
CrVI in coating = 0.02 g/ (1.0 x 10^{-4} g) =0.02 x 10^4 [g/g] = 0.02 wt%

Figure 3 Calculation of CrVI in chromate coating

vi) Concentration of CrVI per unit area in RoHS standards

In v) above, the CrVI concentration in the detection lower limit value 0.02 [µg/cm^2] chromate coating is 0.02wt%, so we can calculate that if the CrVI concentration is 0.01 [wt%], the concentration of CrVI in the chromate coating is 0.10 [µg/cm^2].

Annex III

Thread surface area approximation formula

The following formula is obtained when the cross-section of the threads is approximated with an equilateral triangle. The approximate value is larger than the actual surface area.

Total surface of threads ＝ [1] Screw head base surface area + [2] Thread surface area

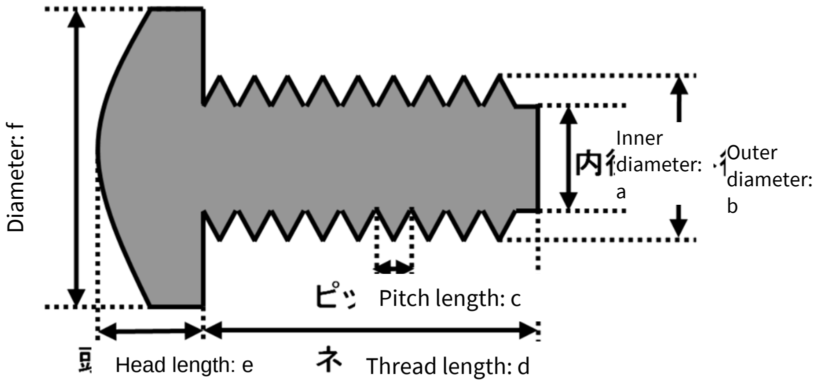


Figure 4 Schematic drawing of screw (thread cross-section approximated with equilateral triangle)

[1] Screw head base surface area $= \pi \times f \times e + 2 \times \pi \times \left(\frac{f}{2}\right)^2$

[2] Thread surface area$= \pi \times \left\{\left(\frac{b}{2}\right)^2 - \left(\frac{a}{2}\right)^2\right\} \times \frac{d}{c} \times 2 \times \frac{2}{\sqrt{3}}$

Annex IV

Reagents required for extraction and analysis of organic substances in plastic and electronics

(1) Blocking of UV rays during PBB and PBDE extraction

When PBB and PBDE are subject to UV rays, "debromination" occurs in which the bromine in the molecules is desorbed. To prevent this, the sample must be protected from UV rays during extraction. Either use a shielded extraction apparatus, or cover the extraction apparatus with aluminum foil, etc., to block the PBB and PBDE in the sample being extracted from UB rays.

(2) Poor solvent and good solvent

As Table 62 shows, solvents are divided into "good solvents" that dissolve the target substance and the polymer, which is the main material of plastic, and "poor solvent" that dissolves the target substance but does not dissolve the polymer.

Table 62 Good solvent and poor solvent

Solvent	Description	Typical example
Good solvent	Dissolves both target substance and polymer	Tetrahydrofuran (THF), toluene, acetone
Poor solvent	Dissolves target substance, but does not dissolve polymer	Hexane, acetonitrile, methanol

(3) Surrogates

The concentration of target substance in the extraction solution may be lower than the actual concentration because the target substance may remain in the polymer residue after the extraction process. In this case, "surrogate" a substance similar to the substance to be analyzed is weighed and added to the extraction solution. The change in target substance concentration is compensated based on the change in the surrogate concentration during the extraction process. A stable substance such as anthracene is used for the surrogate.

(4) Re-precipitation

Re-precipitation is a technique for separating high polymers from non-polymeric substances in a solution obtained by dissolving the high polymers and non-polymeric substance with "good solvent". If a solution containing high polymers and non-polymeric substance is dripped into the "poor solvent", the high polymers will precipitate, suspend, and settle. The non-polymeric substance will be dissolved in the poor solvent at this time. The high polymer can be separated from the non-polymeric substance by filtering the suspended and precipitated substance from the poor solvent.

Note that it may be possible to separate the high polymer by dripping poor solvent into the solution containing high polymers and non-polymeric substance. In IEC 62321, acetonitrile is dripped into PVB THF solution to which phthalate esters have been added to precipitate the PVC.