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Guidelines for Analysis of Chemical Substances Contained in Products as Measures for EU RoHS Directive and Packaging Material Directive




Dec. 12 2017 TOTO LTD.,

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1. Purpose of Establishment of These Guidelines

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(1) For the purpose of compliance with the RoHS Directive and the Packaging Materials Directive, which are regulations set by the European Union (EU), the TOTO Group shall request suppliers to submit the certificate of conformity, the material composition chart, and analysis data of homogenous material units as evidence of compliance with laws and regulations.

(2) These Guidelines shall stipulate the requirements for analysis data to be submitted by suppliers to ensure compliance with these laws and regulations.
Suppliers shall be asked to submit analysis data using the analysis method according to the international standards.

2. Analysis Methods and Basic Analysis Flow

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2.1 Analysis Methods

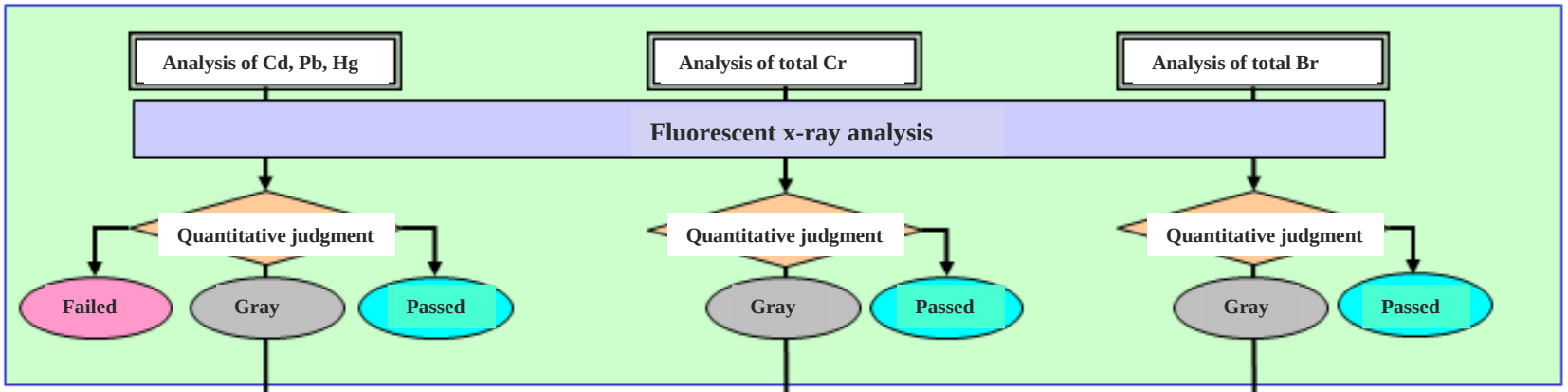
The following table shows the analysis methods for substances regulated in IEC 62321 by material. The analysis methods are roughly divided into screening analysis and precision analysis. For the positioning of screening analysis and precision analysis, refer to 2.2 Basic Analysis Flow.

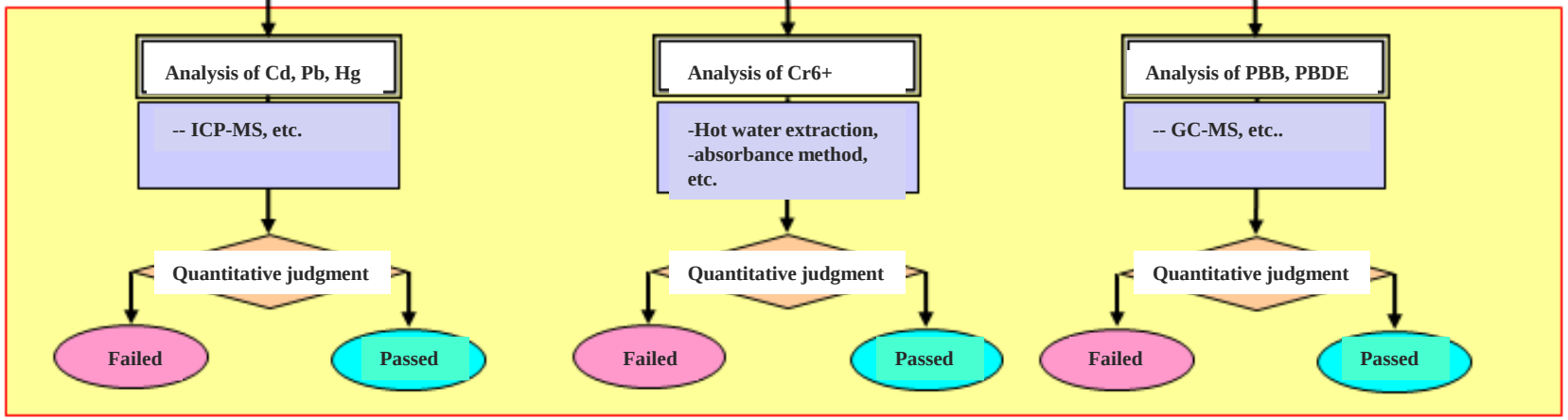
	Screening Analysis	Precision Analysis		
		Resin	Metal	Electronic Parts
Cd (Cadmium)	*Chapter 6 of IEC 62321 (Annex D) - XRF (Fluorescence X-ray analysis) *ED-XRF, WD-XRF	*Chapter 8 of IEC 62321 (Annex F)	*Chapter 9 of IEC 62321 (Annex G)	*Chapter 10 of IEC 62321 (Annex H)
Pb (Lead)		- ICP-OES (Inductively coupled plasma emission spectrometry) - ICP-MS (Inductively coupled plasma mass spectrometry) - AAS (Electron absorption spectrometry)		
Hg (Mercury)		**Chapter 7 of IEC 62321 (Annex E) - ICP-OES (Inductively coupled plasma emission spectrometry) - ICP-MS (Inductively coupled plasma mass spectrometry) - CV-AAS (Cold vapor electron absorption spectrometry) - CV-AFS (Cold vapor electron fluorescence analysis)		
Cr6+ (Hexavalent chromium)	*Total Cr based analysis	*Annex C - Colorimetric method	*Annex B - Hot water extraction method	*Annex C - Colorimetric method
PBB (Polybrominated biphenyl)	*Total Br based analysis	*Annex A - GC-MS (Gas chromatography mass spectrometry)	<<Not applicable>>	*Annex A - GC-MS (Gas chromatography mass spectrometry)
PBDE (Polybrominated diphenyl ether)				

2.2 Basic Analysis Flow

Perform a screening analysis. If it is difficult to make a pass or fail judgment (if the judgment result is considered gray), perform verification by conducting a precision analysis. Furthermore, it is fine to omit a screening analysis and perform a precision analysis only.

Screening Analysis





Precision Analysis

3. Details of Analysis Methods

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3.1 Analysis Methods for Cd, Pb

(1) Screening Analysis

Analyzer	Fluorescent x-ray analyzer (ED-XRF, WD-XRF) *The analyzer used for measurement shall satisfy the following standard of detection sensitivity. [Lower limit of detection] – Cd: Less than 35 ppm, Pb, Hg, Cr: Less than 350 ppm, Br: Less than 150 ppm Note: The lower limit of detection shall be a value obtained by actually measuring a substance whose main component is similar to the measurement sample.
Pretreatment method	1) Either non-destructive or destructive measurement can be performed. 2) In the case of a destructive measurement, perform processing by cutting, grinding, or pressing as needed to obtain a sample having a shape necessary for measurement (smooth surface, thickness).
Measurement method	1) Use the FP method or the calibration curve method to perform a quantitative analysis. 2) Since the fluorescent x-ray method is less accurate than a precision analysis, consider 3σ as a measurement value and judge the suitability according to Reference Material 1. If it is difficult to make a pass or fail judgment from a screening analysis (if the judgment result is gray), perform verification using a precision analysis.

(2) Precision Analysis

Analyzer	ICP-OES (Inductively coupled plasma emission spectrometry), ICP-MS (Inductively coupled plasma mass spectrometry), AAS (Atomic absorption spectrometry)
Pretreatment method	1) Collect samples in a size and a shape suitable for analysis by cutting them. 2) Using a method such as a wet digestion method with acid, analyze a sample that is turned into a solution.
Measurement method	1) Use a standard solution containing the target substance to create a calibration curve. 2) Measure the sample solution and calculate the amount of each target substance contained in the solution based on the calibration curve. 3) Calculate the concentration in the sample based on the amount contained in the solution.

3.2 Analysis Method for Hg

(1) Screening Analysis

Analyzer	Same as 3.1. (1)
Pretreatment method	
Measurement method	

(2) Precision Analysis

Analyzer	ICP-OES (Inductively coupled plasma emission spectrometry), ICP-MS (Inductively coupled plasma mass spectrometry), CV-AAS (Cold vapor atomic absorption spectrometry), CV-AFS (Cold vapor atomic fluorescence analysis)
Pretreatment method	Same as 3.1. (2)
Measurement method	

3.3 Analysis Method for Hexavalent Chromium

(1) Screening Analysis (Analysis of total Cr)

Analyzer	Same as 3.1. (1)
Pretreatment method	
Measurement method	

(2) Precision Analysis (Analysis of hexavalent chromium)

Material	Resin, electronical parts	Metal
Analyzer	Hot water extraction – Absorbance method	
Pretreatment method	1) Make the sample as fine powder passing through a 250 μm sieve by cutting it. 2) Decompose this in an alkaline extract solution at 90°C to 95°C for three hours. 3) Filter the elution residue and use the constant volume of extracted liquid as the sample.	1) Collect the sample in a size and a shape suitable for analysis by cutting it. 2) Boil water in a beaker, make the sample sink in the water, and wait for 10 minutes. 3) Remove the sample and cool the water to room temperature. 4) Add water in the amount exceeding the amount of volatilized water and dilute the sample to obtain a constant volume. *As a blank sample, prepare water that was generated in the same manner but does not include the sample.
Measurement method	1) Add diphenylcarbazide reagent to the sample water. 2) Measure the absorbance at 540 nm and determine the concentration in the liquid based on the calibration curve. 3) Calculate the concentration in the sample based on the amount contained in the solution.	1) Add diphenylcarbazide reagent to the sample water. 2) Compare the color of the sample extract solution with that of the blank sample. If the sample extract solution is colored, it is judged that it contains hexavalent chromium (positive). *For color comparison, measure the absorbance at 540 nm or perform visual inspection.
Judgment method		Since elution from the surface is dominant in the case of metal, the elution amount per unit area of 0.1 μg/cm² shall be defined as the regulation value.

3.4 Analysis Method for PBB/PBDE

(1) Screening Analysis (Analysis of Total Br)

Analyzer	Same as 3.1. (1)
Pretreatment method	
Measurement method	
(Note) Judgment method	Since the fluorescent X-ray analysis quantifies the amount of Br in a compound, a Br concentration of 300 ppm shall be defined as the regulation value.

(2) Precision Analysis (Analysis of PBB/PBDE)

Analyzer	GC-MS (Gas chromatography / mass spectrometry)
Pretreatment method	1) Collect samples in a size and a shape suitable for analysis by cutting them, etc. 2) Using an organic solvent such as toluene, extract the liquid by the Soxhlet extraction method or the high-speed solvent extraction method.
Measurement method	1) Introduce the sample extract solution into GC-MS. 2) Perform qualitative analysis by TIC (Total Ion Chromatogram) and SIM (Selective Ion Monitoring) and perform quantification by the calibration curve method.
Points to note	Attention is required because the following alternate names are used for PBDE. PBDE/PBBO/PBDPE/PBBE/PBDPO/PBDO

4. Description in Report

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The following information shall be entered in the analysis result report.

Information of the analysis organization	**- Analysis organization name (corporate seal required)** **-Name of a person in charge of analysis, name of a responsible person**
Information of the parts to be analyzed	**- Model number of the parts** **- Materials configuring the parts** **- Substances to be analyzed in each material**
Information of the analysis method	**- Analysis method** **- Analyzer name (manufacturer, model)** **-Lower limit of analysis (lower limit of detection, quantitative lower limit)** **- Quantitative value and measurement accuracy (σ or 3σ)** **- Quantification method (calibration curve method, FP method)**

Note: Expiration date of analysis data of homogeneous material unit

We may ask you to submit the latest analysis data as needed. In that case, perform periodic quality management of materials so that you can submit the analysis data obtained within one year.

Reference Material 1: Screening Judgment Standard by Fluorescent X-ray Analysis (Products Subject to RoHS Directive)

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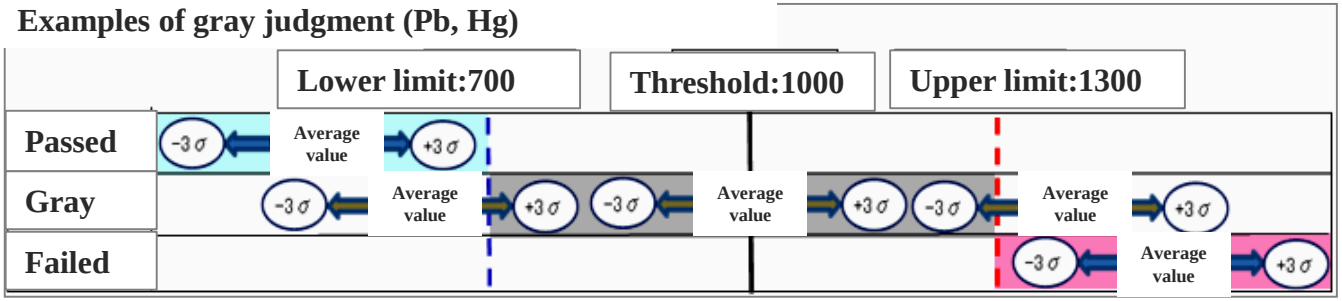
For the judgment of screening analysis using fluorescent x-rays, take measurement accuracy (3σ) into account. If the value obtained by adding the measurement variation (3σ) to the measured concentration value falls within the gray judgment area, it is difficult to make a precise pass or fail judgment, which requires a more precise analysis to make the judgment.

		Resin	Metal	Electronic component composition material
Target substance	Threshold (ppm)	Estimated errors: Threshold plus or minus 30%	Threshold plus or minus 30%	Threshold plus or minus 50%
Cd	100	Passed: Measurement value + 3σ < 70 Failed: Measurement value - 3σ ≥ 130 Gray: Others	Passed: Measurement value + 3σ < 70 Failed: Measurement value - 3σ ≥ 130 Gray: Others	Passed: Measurement value + 3σ < 50 Failed: Measurement value - 3σ ≥ 150 Gray: Others
Pb	1000	Passed: Measurement value + 3σ < 700 Failed: Measurement value - 3σ ≥ 1300 Gray: Others	Passed: Measurement value + 3σ < 700 Failed: Measurement value - 3σ ≥ 1300 Gray: Others	Passed: Measurement value + 3σ < 500 Failed: Measurement value - 3σ ≥ 1500 Gray: Others
Hg	1000	Passed: Measurement value + 3σ < 700 Failed: Measurement value - 3σ ≥ 1300 Gray: Others	Passed: Measurement value + 3σ < 700 Failed: Measurement value - 3σ ≥ 1300 Gray: Others	Passed: Measurement value + 3σ < 500 Failed: Measurement value - 3σ ≥ 1500 Gray: Others
Total Cr (Cr6+)	1000 (1000)	Passed: Measurement value + 3σ < 700 Gray: Others	Passed: Measurement value + 3σ < 700 Gray: Others	Passed: Measurement value + 3σ < 500 Gray: Others
Total Br (PBB, PBDE)	300 (1000)	Passed: Measurement value + 3σ < 300 Gray: Others	(Analysis not required)	Passed: Measurement value + 3σ < 250 Gray: Others

Examples of gray judgment (Pb, Hg)



Reference Material 2: Judgment Standard by Precision Analysis (Products Subject to RoHS Directive)

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RoHS Directive-regulated substance		Judgment standard
Cadmium	Cd	Less than 100 ppm
Lead	Pb	Less than 1000 ppm
Mercury	Hg	Less than 1000 ppm
Hexavalent chromium	Cr6+	Less than 1000 ppm
Polybrominated biphenyl	PBB	Less than 1000 ppm
Polybrominated diphenyl ether	PBDE	Less than 1000 ppm

(1)Screening Analysis

Analyzer	Fluorescent x-ray analyzer (ED-XRF, WD-XRF) *The analyzer used for the measurement shall satisfy the following standard of detection sensitivity. [Lower limit of detection] – Cd, Pb, Hg, Cr: Less than 25 ppm Note: The lower limit of detection shall be a value obtained by actually measuring a substance whose main component is similar to the measurement sample.
Pretreatment method Measurement method	Same as 3.1. (1)
(Note) Judgment method	- Criteria for passing: Pb, Cd, Hg, Total hexavalent Cr < 70 ppm - In the case of Total of Pb, Cd, Hg < 70 ppm AND Total of Pb, Cd, Hg, Cr < 130 ppm, perform precision analysis of hexavalent chromium.

(2)Precision Analysis

If it is difficult to make a precise pass or fail judgment because of the high lower limit of detection by screening analysis, perform verification using a precision analysis.

Pretreatment method Measurement method	Same as 3.1. (2), 3.2. (2), and 3.3. (2)
(Note) Judgment method	Criteria for passing: Pb, Cd, Hg, Total hexavalent Cr < 100 ppm