Innovation for Customers

Method for Analyzing Prohibited Substances in Purchased Products

September 1, 2018 Ver.3

Nitto Group

1.Definitions of Terms

Term	Description
Threshold value	Acceptable condensation of target substance
Substance analyzed	Substance described as “analysis data required” in prohibited substances in products purchased by Nitto Group. To be concrete, they are the following substances stipulated by the ELV and RoHS Directive: Cadmium, lead, mercury, hexavalent chromium, and specified bromine-based flame retardants (PBB and PBDE), specific phthalate esters (DEHP, BBP, DBP, DIBP) and asbestos.
Analyzed data	Results of analysis performed according to the methods described in this instruction manual
Precision analysis	Analysis that identifies / quantifies the content of the target substances
Screening analysis	Analysis that identifies the element content of the target substances (Simplified analysis)
XRF	Abbreviation for X-ray fluorescence
ICP	Generic term for ICP-AES, -OES (inductively-coupled plasma atomic (optical) emission spectrophotometer) and ICP-MS (inductively-coupled plasma mass spectroscope)
AAS	Abbreviation for atomic absorption spectrometer
GC／MS	Abbreviation for gas chromatograph-mass spectrometry
PY／TD－GC／MS(PY - GC／MS)	Thermal extraction GC／MS(Pyrolysis-GC／MS)
IC	Abbreviation for ion chromatograph analyzer
Packing materials	(1) Packing materials that are used for raw materials to be delivered (2) Packing materials that are used for packing products from the Nittogroup companies

2. Purpose

The purpose of this manual is to clearly define the methods of analysis to identify ten substances* which are restricted for use by EU Directive such as ELV and RoHS and to control procurement activities under the legal restrictions of the industry concerning chemical substances in products and to clearly define the methods for analyzing asbestos.

*) cadmium, lead, mercury, hexavalent chromium, specific bromine-based flame retardants (PBB, PBDE), specific phthalate esters (DEHP, BBP, DBP, DIBP)

3. Target Substance for Analysis

The method of analysis shall apply to cadmium, lead, mercury, hexavalent chromium, specified bromine-based flame retardants (PBB and PBDE), and specific phthalate esters (DEHP [DOP]): Di(2-ethylhexyl)phthalate, BBP: Benzyl butyl phthalate, DBP: Dibutyl phthalate, DIBP: Diisobutyl phthalate concerning raw materials delivered to the Nitto Group (including packing materials used for raw materials to be delivered thereto) and packing materials for packing products from the Nitto Group and also apply to mineral raw material in which asbestos such as talc may be mixed.

4. Overview of Method of Analysis

You are requested to verify the materials for delivery to the Nitto group to confirm that such materials do not include the target substances exceeding the acceptable concentration levels.

Table 1 shows the method of analysis and acceptable concentration levels (threshold values). As for the method of analysis, be sure to comply with official IEC 62321, etc. and to follow the instructions of the requesting department.

Table 1

	Method of Analysis		Threshold Values (ppm)	
	Precision Analysis	Screening Analysis	Raw Material	Packing Material
Cadmium	ICP method AAS method	XRF	Less than 5	Less than 100 ppm of total weight of included heavy metal: Cadmium, lead, mercury, hexavalent chromium, compared with weight
Lead	ICP method AAS method	XRF	Less than 100	
Hexavalent chromium	Diphenylcarbazide optical density method	XRF ICP method	Less than 100	
Mercury	ICP method	XRF	Less than 100	
PBBs	GC/MS method	XRF IC method	Less than 100	-
PBDEs (incl. decabromodiphenylether)			Less than 100	-
Specific phthalate Esters	GC/MS method	PY-GC/MS method	Less than 1000	
Asbestos	Refer to (March 2018) "Asbestos Analysis Manual of Preliminary Survey Based on the Ordinance on Prevention of Health Impairment due to Asbestos [1.20 ver.] (March 2018) published by the Ministry of Health, Labour and Welfare.		Less than 1000	

*1: The result of measuring chromium by the XRF or ICP method shows total chromium content. The result of measuring PBB, PBDE by XRF or IC method shows total bromine content.

5. Methods of Analysis and Determination

5.1 Screening by X-ray fluorescence analysis method

The presence or absence of the five target elements (cadmium, lead, mercury, total chromium, total bromine) and the measured value to be less than the threshold value are determined.

* In the case that the determining numerical value of total chromium is 100 ppm or more, implement precision analysis for hexavalent chromium (diphenylcarbazide absorption photometric method) and re-determine the value.
* In the case that the determining numerical value of total bromine is 30 ppm or more, implement precision analysis for PBB and PBDE (GC / MS method) and re-determine the value.

5.2 Screening by ICP Method

The presence or absence of total chromium content and the measured value are determined.

* In the case that the determining numerical value of total chromium is 100 ppm or more, implement precision analysis for hexavalent chromium (diphenylcarbazide absorption photometric method) and re-determine the value.

5.3 Screening by Ion Chromatography Method

The presence or absence of the target bromine (total bromine) content and the measured value are determined.

* In the case that the determining numerical value of total bromine is 30 ppm or more, implement precision analysis for PBB and PBDE (GC / MS method) and re-determine the value.

5.4 Screening by Thermal Extraction GC / MS Method

The presence or absence of the target specific phthalate esters (DEHP, BBP, DBP, DIBP) content and the measured value are determined.

* In the case that the determining numerical value is more than 500 ppm and less than 1500 ppm, implement precision analysis for specific phthalate ester (GC / MS method) and re-determine the value.

5.5 Precision Analysis for Cadmium and Lead

As for the quantitative limit, methods shall be permitted that guarantee the following values:

Cadmium: The “Certificate for Non-Use of Prohibited Chemical Substances in Delivered Products” has been submitted

Lead : (less than 20 ppm)

(1) Pretreatment

The following types of pretreatment methods can be used:

・Ashing method in the presence of sulfuric acid:

・Pressurized acid decomposition method in an airtight vessel, including microwavedecomposition methods, for example, EN 13346: 2000 and EPA3052 (1996);

・Acid decomposition method using nitric acid, hydrogen peroxide solution, or hydrochloric acid. For example, EPA3050B Rev. 2 (1996); or

・Wet decomposition method using sulfuric acid, nitric acid, or hydrogen peroxide solution. For example, BS EN1122 (2001).

・Method complying with IEC 62321-2: 2013 or IEC 62321-5: 2013

* If any deposits (insoluble substances) are generated, it is necessary to fully resolve such deposits into solution in some way (alkaline elution procedure, etc.).

(2) Measuring Method

For the measuring method, the following types of methods shall be permitted:

・Inductively-coupled plasma emission spectrophotometer (ICP-AES, ICP-OES). For example, EN ISO 11885 (1998)

・Atomic absorption spectrometer (AAS). For example, EN ISO 5961 (1995).

・Inductively-coupled plasma mass spectroscope (ICP-MS)

・Method complying with IEC 62321-5: 2013

Note：The elution analysis method as represented by EN71-3 (2014), ASTM F963-96a, ASTM D 5117, and ISO 8124-3 cannot be applied for pretreatment.

5.6 Precision Analysis for Hexavalent Chromium

Methods shall be permitted that guarantee that the determination limit is less than 20 ppm.

(1) Pretreatment

The following type of pretreatment method can be used:

Extraction method (JIS H 8625:1993, EPA 3060A)

(2) Measuring Method

For the measuring method, the following types of methods shall be permitted:

・Diphenylcarbazide absorption photometric method (JIS K 0102, JIS K0400-65-20:1998, EPA 7196A)

・Method complying with IEC 62321-7-1: 2015 and IEC 62321-7-2: 2017

5.7 Precision Analysis for Mercury

Methods shall be permitted that guarantee that the determination limit is less than 20 ppm.

(1) Pretreatment

The following types of pretreatment methods can be used:

- Pressurized acid decomposition method in a airtight vessel, includingmicrowave decomposition methods, for example, EPA3052 (1996);
- Heating vaporization - atomic absorption method;
- Wet decomposition method with sulfuric acid, or nitric acid by using a decomposition flask with reflux condenser (Kjeldahl method).
- Method complying with IEC 62321-4: 2013

* For each of the above-stated methods, avoid allowing sublimation of mercury. In addition, if any deposits are generated, it is necessary to resolve such deposits in the solution in some way.

(2) Measuring Method

Measurements can be made with similar methods as those for cadmium or lead. However, if use of low-concentration mercury is anticipated, analysis with chemical-reduction vaporization atomic absorption method, ICP-AES (ICP-OES) method, or ICP-MS method will be considered to be appropriate.

5.8 Precision Analysis for PBB and PBDE

Methods shall be permitted that guarantee that the determination limit is less than 20 ppm.

(1) Pretreatment Dissolution - Extraction

(2) Measuring Method

Gas chromatograph-Mass Spectroscope; Method complying with IEC 62321-6: 2015, etc.

5.9 Precision Analysis for DEHP, BBP, DBP, and DIBP

Methods shall be permitted that guarantee that the determination limit is less than 300 ppm.

(1) Pretreatment

After grinding - Soxhlet extraction or ultrasonic extraction

(2) Measuring Method

Gas chromatograph-Mass Spectroscope; Method complying with IEC 62321-8: 2017, etc.

6. Method for Judging Packing Materials

(1) Screening Analysis

・It is judged acceptable if the total levels of cadmium, lead, mercury, and whole chromium are less than 100 ppm.

・If the total levels of cadmium, lead, mercury, and whole chromium are 100 ppm or over, execute precision analysis of hexavalent chromium (diphenylcarbazide optical density method) and make the determination based on the total levels of cadmium, lead, mercury, and chromium.

* When energy dispersive XRF is used, the determination shall be made by totaling the measured value of +3σ of each substance.

(2) Precision Analysis

The determination shall be made based on the total levels of cadmium, lead, mercury, and hexavalent chromium.

7. Items Included in the Analysis Data and Report

The following items shall be included in the analysis report:

・Name of analysis targets (product and article name of materials to be delivered)
・Name of institution that performed the analysis (if measured by an outside analysis institute);
・Analysis method (pretreatment method, measuring instruments, measuring method name, etc.)
・Date of analysis
・Analysis results (in the case of N.D. (not detectable), describe the lower limit of determination)
・3 σ (only for energy dispersive XRF)
・Analysis flow chart

Analytical data from laboratories conforming to ISO / IEC 17025 is recommended.

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