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Application News

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Inductively Coupled Plasma Atomic Emission Spectrometry

Analysis of Heavy Metals in Toys and Accessories by ICPE-9800 Series

Introduction

In recent years, safety issues related to inexpensive metal toys and accessories have become a social problem. In particular, the presence of heavy metals such as lead (Pb) and cadmium (Cd) in toys poses a health risk to infants if such contaminated toys are licked, placed in the mouth, or even swallowed. Therefore, toys and accessories are now required to be evaluated for safety with respect to their content using an appropriate test method. These test methods are defined according to various international standards, including ISO 8124[1)] (Safety of toys) and the European standard EN71-Part3[2)] (Safety of toys – Migration of certain elements). Both are dissolution tests based on the assumption that such toys and accessories will be ingested by infants, resulting in the absorption of toxic elements into the body. Such an analysis of toys and accessories requires that, in addition to being able to measure harmful trace elements with high sensitivity, measurement can be conducted for multiple samples quickly and at low cost.
Here, using the Shimadzu ICPE-9800 series multi-type ICP atomic emission spectrometer, we conducted measurement of commercially available toys and metallic accessories as samples. The ICPE-9800 series, with its mini-torch plasma and spectrometer capable of simultaneous analysis of all elements at all wavelengths, can conduct high-throughput, low-cost analysis with high sensitivity and high accuracy.

Samples

Commercially available toys (building block, vehicle, etc.)
Commercially available metallic accessories (section of key ring, etc.)
(including second-hand items such as those found at a flea market)

Sample Preparation

Processing was conducted according to the method specified in EN71-Part3.
(1) Paint coated on toy
Very fine paint scrapings are removed from the surface of the toy, and transferred to vials. Then, 5 mL of 0.07 mol/L hydrochloric acid is added for each 0.1 g of the scraped fragments, and after shaking for 1 hour in the dark, the vials are left standing at 37 °C for 1 hour. The eluates are then filtered.
(2) Accessory
A weighed sample is placed in a beaker 40 mm in diameter, and after immersing the sample in 0.07 mol/L hydrochloric acid heated at 37 °C, it is left standing at 37 °C for 2 hours, shielded from the light. The eluate is then filtered.
Then, 0.07 mol/L hydrochloric acid is added to the respective filtrates of (1) and (2) to adjust their volumes to 20 mL, and these are used as sample solutions.

Instrument and Analytical Conditions

The Shimadzu ICPE-9800 series multi-type ICP atomic emission spectrometer was used to conduct the measurements. Table 1 shows the measurement conditions. The ICPE-9800 series, with its echelle spectrometer and CCD detector, can conduct simultaneous measurement of all elements at all wavelengths, permitting high-throughput measurement even when there are many target elements and samples. Also, the plasma gas flowrate-suppressing mini-torch, the Eco mode which limits gas usage and power consumption during wait time, and the use of a vacuum spectrometer, which does not require purge gas, all greatly reduce running costs compared to conventional ICP instruments.

Table 1 Analytical Conditions

Instrument	: ICPE-9800 series
Radio Frequency Power	: 1.2 kW
Plasma Gas Flowrate	: 10 L/min
Auxiliary Gas Flowrate	: 0.6 L/min
Carrier Gas Flowrate	: 0.7 L/min
Sample Introduction	: Nebulizer 10
Misting Chamber	: Cyclone chamber
Plasma Torch	: Mini Torch
Observation	: Axial (AX)
Measurement Time	: 2.5 min/sample (Including rinse time)

Analysis

Quantitative analysis was conducted using the calibration curve method.

[References]
1) ISO 8124-1: 2009 (Safety of toys – Part 1)
2) BS EN71-3: 2013, Safety of toys – Migration of certain elements

Analytical Results

Table 2 shows the analytical results. Fig. 1 shows the spectral profiles.
The detection limit shows clearly the sufficient detection sensitivity to measure less than 1/10 reference value.

Conclusion

Use of the ICPE-9800 series for analysis of toys and accessories clearly demonstrated that high-sensitivity measurement can be conducted quickly and at low cost.

Table 2 Analytical Results for Toys and Metallic Accessories (Unit: mg/kg)

Element	Toy Standard		Detection Limit	Sample Name						
	EN	ISO		Toy 1	Toy 2	Toy 3	Toy 4	Toy 5	Accessory 1	Accessory 2
Al	70000		0.2	493	209	1080	188	1860	41	14
Sb	560	60	1	<	2	<	<	<	<	<
As	47	25	1	<	<	4	<	<	<	<
Ba	56000	1000	0.003	404	353	12	11	21	0.7	0.1
B	15000		0.03	0.6	1.0	0.6	27.7	33.3	0.6	0.7
Cd	17	75	0.02	<	0.3	0.8	<	1.5	0.1	0.1
Cr	460 *	60	0.05	22	34	1.0	103	174	0.1	<
Co	130		0.04	<	<	<	<	<	0.2	2.2
Cu	7700		0.1	3	9	4	72	35	93	377
Pb	160	90	0.5	161	290	1	477	24	<	289
Mn	15000		0.006	7.4	14.1	1.0	2.4	10.4	0.1	0.2
Hg	94	60	0.3	<	<	<	<	<	<	<
Ni	930		0.06	1.8	1.4	2.7	20	9	179	361
Se	460	500	1	<	<	<	<	<	<	<
Sr	56000		0.002	20	9	29	2.3	1.2	0.19	0.13
Sn	180000		0.2	<	<	<	13.8	7.4	<	17.9
Zn	46000		0.04	1140	576	16900	407	18600	343	649

Concentrations in table = Measurement solution concentration × Dilution factor (20 mL/sample weight (g))
EN: EN71-Part3 Category Ⅱ
ISO: ISO8124-3: 2010
*: Cr^{3+} (trivalent chromium) standard value
<: Below the detection limit
Detection limit: 3 times the concentration of the standard deviation obtained from 10 repeated measurements of the calibration curve blank × Dilution factor (200)

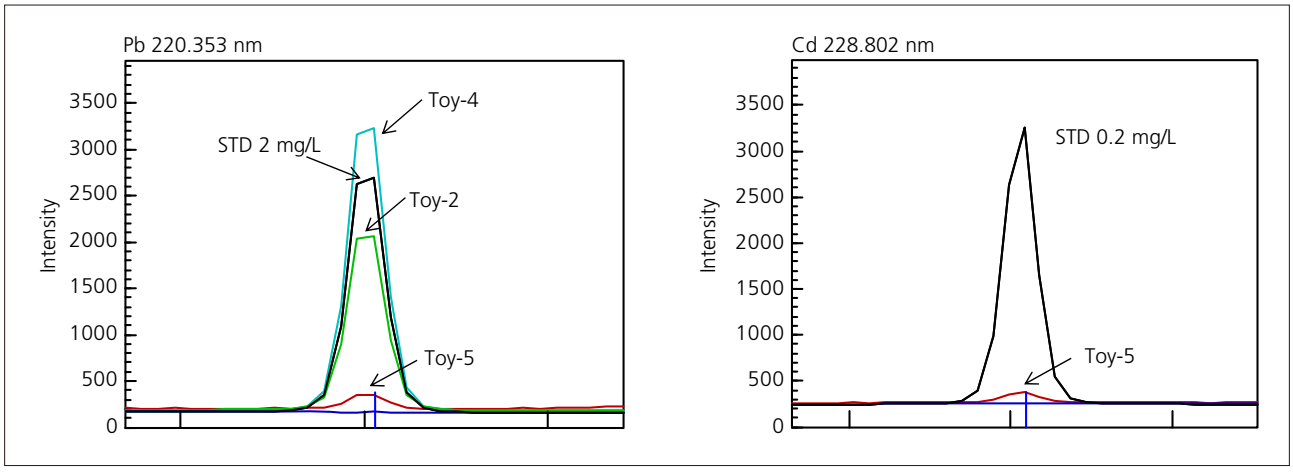

Fig. 1 Spectral Profiles of Toys

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