Analysis of Migratory Elements in Toy Samples using the Thermo Scientific iCAP 7200 ICP-OES

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Key Words
EN 71-3, Toy safety, ASTM F963-11, ISO 8124:2010

Goal

This application note describes the analysis of migratory elements in toy samples, according to the present regulations, using the Thermo Scientific™ iCAP™ 7200 ICP-OES. The pre-loaded template provides a simple and effective tool for routine analysis of toy samples for consumer safety verification.

Introduction

The analysis of toy samples for toxic trace elements has been carried out for many years, however, a number of recent cases have attracted global media coverage due to toys being contaminated with heavy metals. This has lead to an increased number of toy manufacturers carrying out in-house testing to ensure compliance with current regulations. This can prove to be more cost-effective than outsourcing such analyses. Regulations from around the world are largely based on a test method which monitors the levels of trace elements which can migrate from a toy material into an acidic solution. This is designed to simulate the release of toxic elements when toy components are ingested by a child.

The three most common test methods used are EN 71-3:1994/AC:2002 and ISO 8124:2010 “Safety of toys – Part 3: Migration of certain elements” and ASTM F963-11 “Standard Consumer Safety Specification for Toy Safety”. The maximum permissible concentration of the migrated elements is the same for these standards as shown in Table 1. These values are based on the bioavailability of the elements and the average quantities of toy components that are inadvertently consumed by a child on a daily basis (estimated at 8 mg per day).

Table 1. Limits of element migration from toy materials according to EN 71-3:1994/AC:2002, ASTM F963-11 and ISO 8124:2010. All values are mg/kg in solution

	As	Ba	Cd	Cr	Hg	Pb	Sb	Se
Any toy material with exception of modelling clay	25	1000	75	60	60	90	60	500
Modelling clay	25	250	50	25	25	90	60	500

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Instrumentation

The Thermo Scientific iCAP 7200 ICP-OES was used for the analysis. This is a dual view compact ICP-OES instrument that achieves powerful analyte detection and provides a highly cost effective solution for routine analysis of liquids in laboratories with standard sample throughput requirements. The instrument software, Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™, incorporates analysis-ready templates (see Figure 1) to simplify method development and enable simple 'out-of-the-box' analysis with little or no requirement for method development.

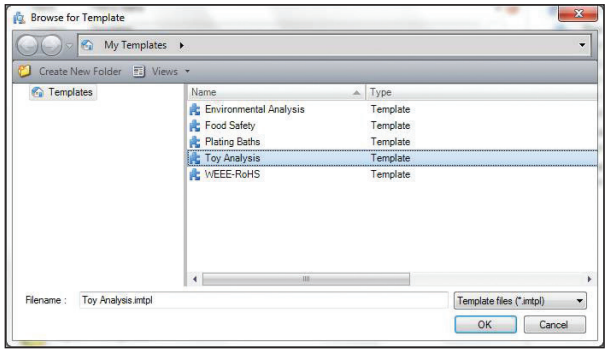

Figure 1. Toy analysis template selection

Method

Three toy materials were analyzed from a small toy car and a baby rattle. These were sampled as follows:

- Orange unpainted rubber body of a toy car (sample 1)
- Unpainted blue wheels of a toy car (sample 2)
- Sections of yellow plastic from a baby rattle (sample 3)

All samples were prepared in accordance with EN71-3, ASTM F963-11 and ISO 8124:2010 (as follows). A portion of the sample (a minimum of 100 mg) was immersed in 0.07 mol/L hydrochloric acid (50 times the mass of the sample) and agitated in the dark at 37 °C for one hour, followed by one more hour of immersion without agitation. All solid material was removed from the sample by filtration using a membrane filter. The remaining filtrate was analyzed immediately. A spike of each sample was also prepared to check recovery of the target elements in the matrix, this was added to the relevant sample prior to agitation.

Standard Preparation

Standards were prepared by diluting 1000 mg/L aqueous single element standards to the concentrations listed in Table 2. The concentrations of the standards are reflected in the pre-loaded Qtegra ISDS template. All standards were prepared to contain 0.07 mol/L hydrochloric acid.

Table 2. Concentrations of the standards prepared as recommended by the method template. All values are in mg/kg

Element	Blank	Standard 1	Standard 2	Standard 3
As	0	10	20	30
Ba	0	100	500	1000
Cd	0	25	50	75
Cr	0	10	30	60
Hg	0	10	30	60
Pb	0	25	30	90
Sb	0	5	10	25
Se	0	50	100	500

Method Development

The Toy Analysis template was opened in Qtegra ISDS. The standard sample introduction kit was chosen for the analysis as recommended by the method and the instrument was calibrated and samples analyzed in a single run (Analysis A). Re-analysis of the identical samples was also performed the following day to simulate a typical sample analysis regime (Analysis B). Parameters used by the template are shown below in Table 3.

Table 3. Method parameters

Parameter	Setting
Pump tubing	Sample Tygon® orange/white Drain Tygon® white/white
Pump speed	45 rpm
Nebulizer	Glass concentric
Nebulizer gas flow	0.18 MPa
Spray chamber	Glass cyclonic
Center tube	2 mm quartz
RF Power	1150 W
Coolant gas flow	12 L/min
Auxiliary gas flow	0.5 L/min
Exposure time	Axial 5 sec Radial 5 sec

Results

The results of the analysis are shown in Table 4. This table also includes the plasma view used for each wavelength. A method detection limit (MDL) study was carried out by analyzing a 10 replicate, acid matched blank. The standard deviation of the 10 replicates was multiplied by three to determine the method detection limit. In all the samples analyzed all toxic elements were below the detection limit (except Ba). A search through the literature was carried out and this was found to be common. It is indicative of the high level of GMP (Good Manufacturing Practice) adhered to by the toy industry. The spike recoveries were all within acceptable limits (<10 % of the prepared values). Re-analysis of the same samples on the following day (Analysis B) demonstrated good reproducibility, again with all samples below the detection limit and comparable results for the spike recoveries (this is shown in Table 5).

Table 4. Results of the sample analysis (Analysis A), method detection limits and spike recoveries. All values are in mg/kg in solution, except recoveries which are in %

Element and Wavelength nm	Plasma View	MDL	Spike value	Sample 1	Sample 1 spike found	Sample 1 spike recovery	Sample 2	Sample 2 spike found	Sample 2 spike recovery	Sample 3	Sample 3 spike found	Sample 3 spike recovery
As 193.759	Axial	0.024	1.5	<DL	1.57	104.7	<DL	1.65	110.0	<DL	1.61	107.3
Ba 233.527	Radial	0.009	25.0	0.015	23.09	92.4	<DL	23.22	92.9	<DL	26.91	107.6
Cd 214.438	Axial	0.001	2.5	<DL	2.67	106.8	<DL	2.54	101.6	<DL	2.63	105.2
Cr 267.716	Axial	0.022	1.5	<DL	1.56	104.0	<DL	1.60	106.6	<DL	1.48	98.7
Hg 194.227	Axial	0.228	1.0	<DL	1.01	101.0	<DL	1.09	109.0	<DL	0.99	99.2
Pb 220.353	Axial	0.019	2.5	<DL	2.48	99.2	<DL	2.66	106.4	<DL	2.65	106.0
Sb 206.833	Axial	0.029	0.5	<DL	0.50	99.2	<DL	0.53	106.0	<DL	0.51	102.2
Se 196.090	Radial	0.024	2.5	<DL	2.56	102.4	<DL	2.57	102.8	<DL	0.53	106.0

Table 5. Results of the sample spikes from the two different analyses (A and B). All values are in mg/kg in solution

Element and Wavelength nm	Sample 1 spikes		Sample 2 spikes		Sample 3 spikes	
	A	B	A	B	A	B
As 193.759	1.57	1.52	1.65	1.53	1.61	1.59
Ba 233.527	23.09	23.20	23.22	24.34	26.91	25.02
Cd 214.438	2.67	2.59	2.54	2.46	2.63	2.53
Cr 267.716	1.56	1.54	1.60	1.47	1.48	1.52
Hg 194.227	1.01	1.03	1.09	0.99	0.99	1.02
Pb 220.353	2.48	2.45	2.66	2.59	2.65	2.49
Sb 206.833	0.49	0.51	0.53	0.51	0.51	0.51
Se 196.090	2.56	2.49	2.57	2.51	0.53	0.51

Conclusions

The analysis of migratory elements from toys in hydrochloric acid using the Thermo Scientific iCAP 7200 ICP-OES was made simple by the use of method template integrated within the intuitive design of the Qtegra ISDS. The use of a pre-loaded template with the iCAP 7200 ICP-OES and its optimised sample introduction system provides a total solution for regulatory compliant toy sample analysis. These tools enable both novice and experienced analysts to achieve excellent results with minimal requirements for method development, providing a highly cost effective solution for laboratories performing these analyses.

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