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APPLICATION BRIEF

SEA no.32 Analysis of Lead in Metal Accessories

2007.8

1. Introduction

In March 2006, an investigation by the Tokyo metropolitan government found harmful levels of lead in commercially available, low-cost metal accessories. While simple skin contact with lead is not harmful, it is known that developmental disorders of brain or nervous system may occur if an infant ingests a large amount of lead by licking or swallowing it.

The city investigated the amount of lead extracted from accessories in the stomach and found that 14 of 76 low-cost accessories purchased at supermarkets and other outlets exceeded the limits set by the American Consumer Product Safety Commission (CPSC). While Japan does not currently regulate the amount of lead in metal accessories, this incident has spurred the city to urge manufacturers and sellers to investigate thoroughly the lead content in accessories.

This application brief presents the results of analysis of commercially available accessories by SPS3100 ICP Optical Emission Spectrometer and the SEA5120A Micro Element Monitor.

2. Experiment

2.1 Consumer Product Safety Commission (CPSC) Screening Test for Total Pb Analysis
(This test determines the total lead concentration of the test substance)

Test method:

1. Place a test sample of 50 to 70 mg in a 100 ml beaker.
2. Add 8 ml of nitric acid.
3. Heat the solution on a hot plate until it reaches 3 ml.
4. After cooling, add 2 ml of hydrochloric acid and then stir the solution.
5. Dilute the solution with distilled water to a volume of 50 ml.
6. Examine the solution using ICP optical emission spectrometry (ICP-OES).

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2.2 CPSC Acid Extraction Test

(This test determines the amount of lead that is extracted from the test substance after it is placed in a gastric acid-like solution.)

Test method:

1. Measure the mass of the test sample.
2. Suspend the test sample in a beaker using a Teflon tube.
3. Add 0.07 mol/l hydrochloric acid. The acid solution should be equivalent to 50 times the weight of the test sample.
4. Heat the container to 37° C.
5. Stir the solution for 1 hour.
6. Move the sample from the beaker to a separate plastic container.
7. Add the 0.07 mol/l hydrochloric acid.
8. Stir the solution for 2 hours.
9. Move the sample from the beaker to a separate plastic container.
10. Add the 0.07 mol/l hydrochloric acid.
11. Stir the solution for 3 hours.
12. Individually analyze the resulting three solutions using ICP-OES.

1 2 3 4 5

Before dissolution → After dissolution

After filing

3. Results

3.1 Lead Content in Accessories

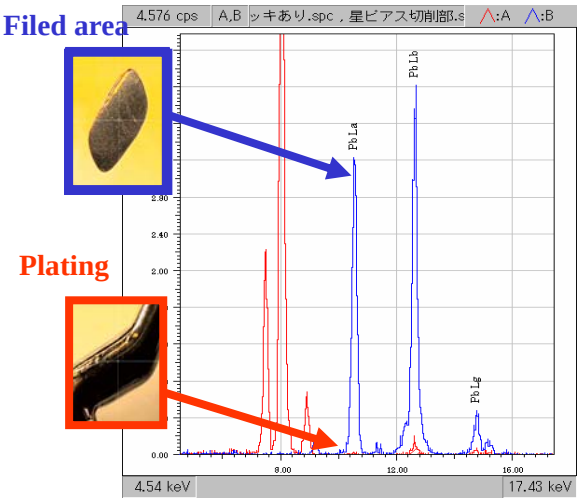
[Comparison of lead content]

ICP-OES was used to measure the completely dissolved items and fluorescent X-rays were used to measure the plating and filed area.

	Lead content (%)		
	1. Plating before dissolution	4. Plating after dis-solution	5. Filed area
Micro Element Monitor 5120A	2.9	15.4	87.4
ICP-OES SPS3100		83.1	

The results above suggest that as the plating melts, the lead under the plating is detected, which increases the lead content results. The ICP-OES results of the completely dissolved test samples and the results of the X-ray fluorescence analysis of the filed area were roughly the same.

The diagram below shows the measurement results from the SEA5120A Micro Element Monitor of the filed areas and plating. As can be seen, the fluorescent X-ray intensity of the plating is much lower than the intensity of the filed area.



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3.2 Lead Extraction Test

[Comparison of measurement values]

The concentrations of the lead in the solutions (1, 2 and 3 hours) from the acid extraction test were measured using ICP-OES.

	Mass of extracted lead (ppm)
1hr	0.010
2hr	0.013
3hr	0.019

The results above show that very little of lead leached out, even when the accessory was suspended in a gastric acid-like solution for 3 hours.

4. Conclusion

The lead content in metal accessories was measured using the SEA5120A Micro Element Monitor and SPS3100 ICP emission spectrometer. The results revealed that a very minute amount of lead leached out during the CPSC acid extraction test, which simulates ingestion. However, a high lead concentration (approximately 85%) was found when the lead content of the entire sample was measured. This suggests that if the sample were scratched, lead could leak out and cause severe harm.

Since very little pre-processing is necessary, X-ray fluorescence spectrometry is effective for performing quick measurements during the analysis of a large number of test substances. X-ray fluorescence spectrometer can measure target elements after simple pre-processing so they are well suited for RoHS element analysis involving a large number of test substances.

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