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Application News

Spectrophotometric Analysis No.A402

Toy Analysis by Atomic Absorption

■ Introduction

Recent revelations regarding toxicity in toys and metallic accessories has spurred increased concern about the safety of these items. In particular, lead (Pb) is known to be toxic to both the nervous system and blood system, and remarkably more so in infants than in adults. Toys that contain such harmful elements could be hazardous to the health of infants if put in the mouth or swallowed. To address these concerns as well as to establish compliance with the ISO standard, the Japan's Ministry of Health, Labour and Welfare revised parts of Article 78 of the Food Sanitation Law Enforcement Regulation and the Food and Additives Standards on March 31, 2008.

The Food Sanitation Law, first implemented in January 1, 1948 with the aim of preventing the occurrence of harm due to food and drink consumption, is a law under the jurisdiction of the Ministry of Health, Labour and Welfare. It establishes standards pertaining to food and additives, apparatus and containers, and rules governing labeling, testing, etc. It comprises the following chapters and supplemental provisions.

Chapter 1 General Provisions (Article 1-4)
Chapter 2 Food and Food Additives (Article 5-14)
Chapter 3 Apparatus and Containers/Packaging (Article 15-18)
Chapter 4 Labeling and Advertising (Article 19-20)
Chapter 5 The Japanese Standards for Food Additives (Article 21)
Chapter 6 Principle and Plan of Inspection and Guidance (Article 22-24)
Chapter 7 Examination (Article 25-30)
Chapter 8 Registered Laboratories (Article 31-47)
Chapter 9 Business (Article 48-56)
Chapter 10 Miscellaneous Provisions (Article 57-70)
Chapter 11 Penal Provisions (Article 71-79)

Although this revision relates to apparatus and containers and packaging, Article 78 of the Food Sanitation Law Enforcement Regulation includes a definition of toys. In addition, since the Food and Additives Standards, also referred to as the "Ministry of Health, Labour and Welfare Notification No. 370", also show the criteria values and preparation procedures, there are many opportunities to see these when conducting analysis of foods and additives. The main revision of this Notification No. 370 is in "Chapter 4 Toy Parts", in which criteria values are set for all paints, including vinyl chloride resin paints, requiring that testing be conducted on all product painted parts. In addition, the dissolution test is based on the ISO standard, with "heavy metals" replaced by "Pb", and the upper limits of dissolution with respect to 1 kg of paint coating and metal were set at 90 mg/kg of Pb, 75 mg/kg of Cd and 25 mg/kg of As.

The standard for metallic toy accessories (restricted to those that are small enough to be swallowed by an infant) was newly established, with the upper limit set at 90 mg/kg of Pb by the dissolution test.

The atomic absorption spectrophotometry and ICP emission methods are employed in the analysis of toxic metal coatings and metallic toy accessories, but the ICP emission analysis method is effective because it allows simultaneous multi-elemental analysis with high sensitivity when the matrix contains many analytes. On the other hand, when there are few analytes, the efficient atomic absorption spectrophotometric method is adopted in many official methods for food and drainage, etc. due to the high sensitivity and low cost with which analysis can be conducted.

This Application News introduces the dissolution testing of Pb from toy paint coating using the furnace atomic absorption method.

Sample preparation for the paint coating dissolution test is described in the Food and Additives Standards specifies as follows: (1) Shave off some coating and grind the particles sufficiently to pass through a 0.5 mm mesh, (2) accurately weigh out about 0.1 g, (3) add 5 mL of 0.07 mol/L hydrochloric acid and shake for 1 minute in the dark at 37 ˚C, and (4) set aside for 1 hour (for metallic toy accessories, perform dissolution using the same solution and dissolution temperature, but set aside for 2 hours).

Here, 0.07 mol/L hydrochloric acid was used for filtration of the processed solution, washing of the filter paper, and for dilution at measurement.

■ Basic Data for Pb

Atomic weight: 207.2
Melting point: 328 ˚C ($PbCl_2$ 501 ˚C, $PbSO_4$ 1070 ˚C)
Boiling point: 1740 ˚C ($PbCl_2$ 954 ˚C)
Oxidation number: +2 (e.g. PbO, PbS, $PbCl_2$, etc.)
+4 (e.g. PbO_2, PbS_2, $PbCl_4$, etc.)
Degree of aqueous solubility: $PbCl_2$ 10.8 g/L (25 ˚C)
$PbSO_4$ 40 mg/L (15 ˚C)

[References]
New Information on Element 111, Dictionary of Physics and Chemistry

■ Measurement Wavelengths of Pb

Wavelength (nm)	Sensitivity Ratio
283.3	0.4
217.0	1.0

Note: Although the sensitivity at 283.3 nm is 1/2.5 that at 217.0 nm, it will be less susceptible to background effect due to the longer wavelength. In addition, baseline noise is lower and more stable than at 217.0 nm due to the greater light intensity.

■ Furnace Analysis of Pb

Here we conducted comparative analysis of the same sample using the flame method and the furnace method. The measurement wavelength used was 217.0 nm in the flame method, and 283.3 nm in the furnace method.
Fig. 1 and Fig. 2 show the calibration curves obtained by the flame method and furnace method, respectively. Fig. 3 shows the heating conditions under which furnace analysis was conducted, using a high density tube.

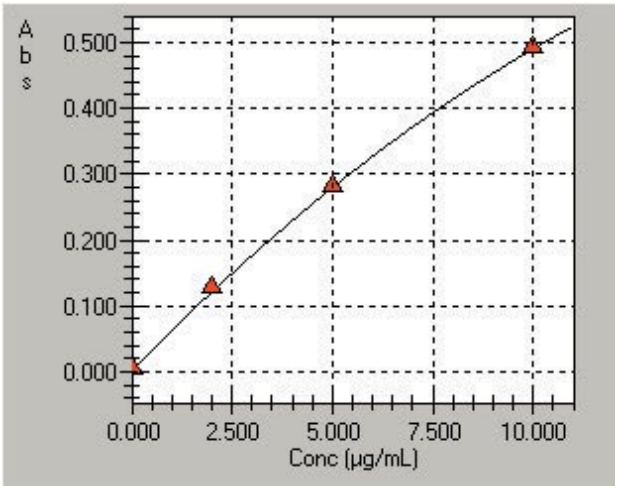

Fig.1 Calibration Curve (Flame)

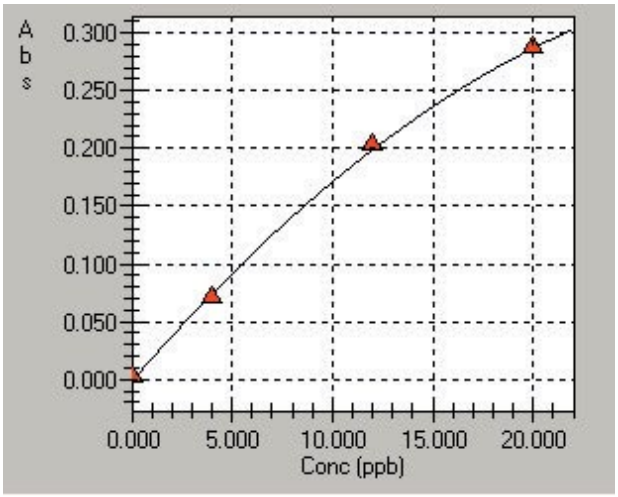

Fig.2 Calibration Curve (Furnace)

	Temperature (°C)	Time (Sec)	Heating
1	150	20	RAMP
2	250	10	RAMP
3	800	10	RAMP
4	800	10	STEP
5	2200	2	STEP
6	2400	2	STEP

Fig.3 Heating Conditions

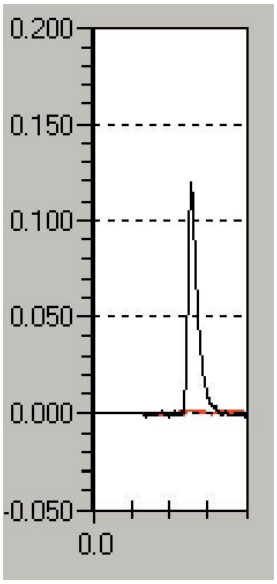

Fig.4 Peak Profile

■ Conclusion

Fig. 4 shows the peak profile when a solution of the actual sample was diluted 10-fold and measured by the furnace method. A result of 65 ppb was obtained using solution conversion. Since this concentration is lower than the quantitation lower limit of 0.1 ppm (217 nm) of Pb in flame measurement and the sensitivity level is not enough, it is evident that furnace measurement is essential for normal dissolution testing of Pb.

Here, 0.1 g of sample was processed, and the solution including that used for filter paper washing was brought to a volume of 20 mL. Therefore, using solid conversion, the result of 13 mg/kg was obtained from 65 ppb × 20 mL/0.1 g.

■ References

- Food Sanitation Law Enforcement Regulation (Ministry of Health and Welfare Ordinance No. 23, 1948)
- Partially Revised Food Sanitation Law Enforcement Regulation (Ministry of Health, Labour and Welfare Ordinance No. 66, 2008)
- Food and Additives Standards Ministry of Health, Labour and Welfare Notification No. 370, 1959
- Partially Revised Food and Additives Standards (Ministry of Health, Labour and Welfare Notification No.153, 2008)
- Regarding Partial Revision of Enforcement Regulation of Food Sanitation Law, and Food and Additives Standards (Food Safety Issue No. 0331007, March 31, 2008)
- Toy Safety Principles (The Japan Toy Association) (ST-2002, 4th Edition)

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