SHIMADZU
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Application News

No.J122

Inductively Coupled Plasma Mass Spectrometry

Analysis of Heavy Metals in Pet Food Using the ICPMS-2030

Introduction

In Japan, a law related to ensuring the safety of pet food (the Pet Food Safety Law) was enacted on June 1, 2009. Based on this law, standards are prescribed regarding the raising and safekeeping of domestic animals, show animals, farm animals, and test animals respectively to ensure the health and safety of the animals, and at the same time to prevent the animals from causing people problems or harm. The manufacture, sale, and import of pet food not in accordance with the regulations are prohibited, and manufacturers, vendors, and importers must comply with the component standards.
A ministerial ordinance regarding component standards for pet food was also enacted on March 1, 2012 in Japan. This ordinance specifies three heavy metals; cadmium, lead, and arsenic.
Here, we introduce a simultaneous analysis performed on toxic elements in pet food using the Shimadzu ICPMS-2030 ICP mass spectrometer.

Sample

Commercially available pet food

Sample Preparation

We used the microwave digestion method that could decompose samples faster than the typically used wet digestion method. Since this method uses a closed vessel, it also has the advantage that loss of volatile elements such as arsenic is minimal. In this study, the sample was decomposed using the Milestone General ETHOS-One.

0.15 g of the sample, 0.5 mL of hydrochloric acid, and 6.5 mL of nitric acid were added to a quartz vessel of the microwave digestion system for sample preparation. The mixture was then decomposed by the microwave digestion system.
After sample decomposition, pure water was added to bring the measurement solution to a volume of 30 mL. At this point, Ga, In, and Bi (at a 10 µg/L concentration in measurement solution) were added as the internal standard elements.
The sample decomposition conditions are shown in Table 1.

Table 1 Sample Decomposition Conditions Using the Microwave Digestion System for Sample Preparation

STEP	Temperature (℃)	Time (min)	Power (W)
1	50	2	1000
2	30	3	0
3	180	25	1000
4	150	1	0
5	180	4	1000
6	180	15	1000

Instrument and Analytical Conditions

The Shimadzu ICPMS-2030 ICP mass spectrometer was used for the measurements. Table 2 shows the measurement conditions.
In addition to providing high sensitivity, the ICPMS-2030 is equipped with a collision system using helium gas, which reduces interference from argon and chlorine significantly.

Table 2 Analytical Conditions

Instrument	: ICPMS-2030
High-frequency output	: 1.2 kW
Plasma gas flowrate	: 8.0 L/min
Auxiliary gas flowrate	: 1.10 L/min
Carrier gas flowrate	: 0.60 L/min
Nebulizer	: Nebulizer 10
Chamber	: Cyclone chamber (electronically cooled)
Plasma torch	: Mini torch
Collision gas	: He

Analysis

The calibration curve method was used for analysis of the three elements; cadmium, lead, and arsenic.
To verify the analysis results, a spike recovery test sample was created by adding a standard solution of measurement elements after the sample decomposition. Quantitative analysis was performed in the same way using this sample.

Analytical Results

Fig. 1 shows the calibration curves for the elements.
Table 3 shows the analysis results. The detection limit is 1/1000th or less of the standard value, so the sensitivity was evidently sufficient. Further, with the favorable spike recovery rates obtained, the validity of the quantitative values was confirmed.

Conclusion

Using the ICPMS-2030, it is possible to perform an analysis for controlling the heavy metals; cadmium, lead, and arsenic in pet food.

[Reference]
Ministerial ordinance regarding component standards for pet food (on April 28, 2009)
The Japan's Ministry of Agriculture, Forestry and Fisheries, and the Ministry of the Environment, Ordinance No.1)

Table 3 Analytical Results for Pet Food

	Unit	As	Cd	Pb
Standard value	µg/g	15	1	3
Detection limit	µg/g	0.002	0.0008	0.0003
Pet food quantitative result	µg/g	0.053	0.054	0.068
Spike recovery rate	%	99	100	102

Spike recovery rate (%) = { (Analysis value for the spike recovery test sample – Analysis value) / Spike concentration} × 100

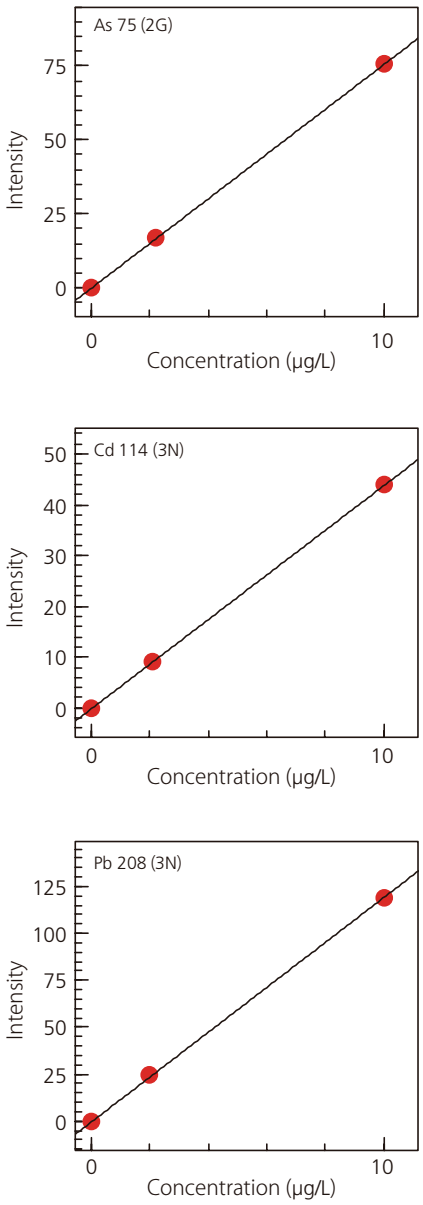

Fig. 1 Calibration Curves for As, Cd and Pb

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