

Application News

No. J123

Inductively Coupled Plasma Mass Spectrometry

Analysis of Toxic Elements in Supplements as per USP 2232 Using the ICPMS-2030

■ Introduction

The United States Pharmacopeia (USP) 2232 designates permitted daily exposure (PDE) levels for four elements for which toxicity is a concern in dietary supplements. PDE values need to be converted to concentrations when evaluating metallic impurities in formulations or their structural components. Compliance with these regulations is also obligatory for supplements imported to the U.S. from other nations.

Here, we introduce a quantitative analysis performed on arsenic, cadmium, mercury, and lead in supplements using the Shimadzu ICPMS-2030 ICP mass spectrometer.

■ Sample

Four commercially available supplements
 (1) One 0.331 g tablet (three tablets/day)
 (2) One 0.350 g tablet (one tablet/day)
 (3) One 0.201 g tablet (one tablet/day)
 (4) One 0.380 g tablet (six tablets/day)

■ 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.

One tablet 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.

Table 1 shows the sample decomposition conditions using the microwave digestion system for sample preparation.

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

STEP	Temperature (°C)	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 mass spectrometer with standard attachments as shown in Table 2 was used for measurements.

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	: 0.65 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 quantitative analysis of the elements arsenic, cadmium, mercury, and lead.

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

Table 3 shows the permitted daily exposure (PDE) levels, maximum permitted concentrations, and detection limits. The analysis results and spike recovery rates are shown in Table 4. These show favorable recovery rates and confirm that the results obtained were appropriate. The sensitivity was also evidently sufficient, even at the maximum intake of 10 g per day.

■ Conclusion

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

[Reference]

USP 2232 Elemental Contaminants in Dietary Supplements

Table 3 PDE Levels, Maximum Permitted Concentrations, and Detection Limits for Elemental Impurities Specified in USP 2232

Name of Element	As	Cd	Hg	Pb
PDE (µg/day)	15	5	15	10
Maximum Permitted Concentration ^{*1} (µg/g)	1.5	0.5	1.5	1
Maximum Permitted Concentration in Measurement Solution ^{*2} (µg/g)	3	1	3	2
Detection Limit ^{*2} (µg/L)	0.009	0.003	0.003	0.001

*1: When the maximum intake per day is 10 g

*2: When a 0.2 g sample is pretreated and diluted to a volume of 100 mL

Table 4 Analytical Results for Supplements (concentration in solid sample, µg/g)

Name of Element Sample name	As	Cd	Hg	Pb
①	0.21	0.029	N.D.	0.035
②	0.08	0.018	N.D.	0.009
③	0.11	0.008	N.D.	0.037
④	0.29	0.071	N.D.	0.095
Recovery rate (%)	97	98	92	101

N.D.: Not detected

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

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