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Application News

No.J120

Inductively Coupled Plasma Mass Spectrometry

Analysis of Minerals and Harmful Elements in Formula Milk Powder Using ICPMS-2030

Introduction

Formula milk powder is formulated to contain a good balance of minerals necessary for infant growth. Japan's Health Promotion Law includes stipulations on the essential mineral content, including calcium (Ca), iron (Fe), and copper (Cu), of food for special dietary use (e.g., formulated milk powder for infants) and requires the labeling of their content. [1)]

Harmful elements like lead (Pb) have a negative effect on infant development, and require strict safety controls from raw materials to the finished product.

We describe using Shimadzu's ICPMS-2030 inductively coupled plasma mass spectrometer to perform a simultaneous analysis of elements present in formula milk powder (National Metrology Institute of Japan certified reference material [NMIJ CRM]). In addition to being highly sensitive, the ICPMS-2030 uses a helium gas collision system that greatly reduces the spectral interference caused by argon and chlorine.

[References]
1) Permission standards for labeling and component composition of breast milk and formulated milk powder for infants (Permission for labeling of food for special dietary use, Food Labeling Division, Consumer Affairs Agency, No. 221 of March 31, 2016)

Sample

NMIJ CRM milk powder (for trace element analysis)
(NMIJ CRM 7512-a: No. MI-040)

Sample Preparation

Compared to the normal wet digestion method, the microwave sample decomposition method has the advantages of completing decomposition in a shorter period of time and within a closed system, which results in little volatilization loss of As and other analytes. We used the ETHOS One (Milestone General K.K., Japan) to perform sample decomposition.

After placing a sample 0.25 g, hydrochloric acid 0.5 mL, and nitric acid 6.5 mL in a quartz decomposition vessel, decomposition was performed using a microwave sample pretreatment system.

After decomposition, pure water was added to make up 250 mL of solution for analysis (1000-fold dilution). At this point, Sc, Co, Ga, Y, In, and Ti internal standard elements were added (to an analytical solution concentration of 10 µg/L).

Table 1 shows the decomposition conditions.

Table 1 Decomposition Conditions for Microwave Digestion System

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

Shimadzu's ICPMS-2030 inductively coupled plasma mass spectrometer was used for analysis. Analytical conditions are shown in Table 2.

In addition to being highly sensitive, the ICPMS-2030 uses a helium gas collision system that greatly reduces the spectral interference caused by argon and chlorine.

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
Sample introduction	: Nebulizer 10
Chamber	: Cyclone chamber (electronic cooling)
Plasma torch	: Mini-torch
Collision gas	: He

Analysis

Minerals and harmful elements present in formula milk powder were measured simultaneously using a calibration curve method.
To confirm the validity of measured values, a spike and recovery test sample was prepared by adding a standard solution of analyte elements (Cd, Cr, Pb, As) after sample decomposition treatment.

Analytical Results

Table 3 shows the analytical results. The results for mineral elements were within the range of certified levels, and good spike and recovery was obtained for trace amounts of harmful elements.

Conclusions

We successfully used the ICPMS-2030 to simultaneously measure mineral constituents present in high concentrations in formula milk powder along with trace amounts of harmful elements.

Table 3 Analytical Results for Certified Reference Material in Formula Milk Powder (NMIJ CRM 7512-a)

	Unit	Measured Value (in Powder)	NMIJ Certified Value	Expanded Uncertainty	Spike and Recovery (%)
Ca	g/kg	8.5	8.65	0.38	-
Fe		0.102	0.104	0.007	-
K		8.3	8.41	0.33	-
Mg		0.82	0.819	0.024	-
Na		1.81	1.87	0.09	-
P		5.4	5.62	0.23	-
Mn	mg/kg	0.91	0.931	0.032	-
Mo		0.230	0.223	0.012	-
Sr		5.7	5.88	0.20	-
Zn		41	41.3	1.4	-
Cd		<0.005	-	-	100
Cr		<0.06	-	-	101
Pb		<0.03	-	-	100
As		<0.03	-	-	108

Spike and recovery (%) = { (Spike and recovery test sample analysis result - Measured value) / Spiked concentration} × 100

First Edition: Sep. 2016

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