

## Simultaneous Analysis of Minerals in Powdered Infant Formula

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### User Benefits

- ◆ Accurate simultaneous analysis of a range of analytes, from trace levels of Se to high-concentration alkali elements.
- ◆ Shorter analysis times by only using helium as the collision gas.
- ◆ Lower running costs due to Mini-Torch Plasma System that consumes around half the argon gas.

### Introduction

A well-balanced nutritional intake is vital for infant growth. Infant formulas contain an appropriate balance of nutrients necessary for infant growth and are sold in a variety of forms, including powdered and liquid. The CODEX STAN 72-1981<sup>1)</sup> standard was established to ensure these infant formulas are safe and meet quality requirements.

This article describes using the ICPMS-2030 to perform a simultaneous analysis of 12 elements in a certified standard reference infant formula.

The ICPMS-2030, equipped with a high-performance collision cell, provided accurate quantification of a range of analytes without needing hydrogen as a cell gas, from trace levels of Se to high-concentration alkali elements.

### Analyzed Sample

Certified Standard Reference Material NIST SRM 1849a Infant/Adult Nutritional Formula I (milk-based)

### Reagents

- High purity nitric acid (Ultrapur-100, Kanto Chemical Co., Inc.)
- 30 % hydrogen peroxide (super special grade, FUJIFILM Wako Pure Chemical Corporation)
- High purity methanol (Kanto Chemical Co., Inc.)

### Sample Preparation

Samples were prepared based on a method established by the AOAC.<sup>2)</sup>

0.20 g of certified standard reference material (NIST SRM 1849a) was weighed out in a digestion vessel, 5 mL of nitric acid, 2 mL of hydrogen peroxide, and 1 mL of pure water were added to the vessel, 0.5 mL of an internal standard element solution (5 mg/L of Ge and Te) was added, and digestion was performed with a microwave digestion system by following STEP 1 then STEP 2 of the digestion method shown in Table 1.

Table 1 Digestion Method

STEP 1	Time (min)	Temp. (°C)	STEP 2	Time (min)	Temp. (°C)
1	2	70	1	2	70
2	1	50	2	1	50
3	17	180	3	17	200
4	20	180	4	20	200

After digestion, 0.5 mL of high-purity methanol was added and the mixture was made up to 50 mL with pure water. All samples were prepared twice (n = 2) for analysis.

### Calibration Standard Solutions

Calibration standard solutions were prepared by combining commercially available single-element standard solutions. To mixtures of standard solutions were added 5 mL of nitric acid, 0.5 mL of high purity methanol, and 0.5 mL of the internal standard element solution (5 mg/L of Ge or Te), and the resulting mixture was made up to 50 mL with pure water. The concentrations of each element were chosen according to the AOAC-prescribed method.<sup>2)</sup> The concentrations of elements in each calibration solutions shown in Table 2.

Table 2 Elements, Mass, Internal Standard Element and Concentration of Calibration Standard Solutions

Element	m/z	Internal Standard Element (m/z)	The concentration of calibration standard solutions (mg/L)				
			STD0	STD1	STD2	STD3	STD4
Na	23	Ge (72)	0	0.5	2.5	10	20
Mg	24	Ge (72)	0	0.2	1	4	8
P	31	Ge (72)	0	0.5	2.5	10	20
K	39	Ge (72)	0	1	5	20	40
Ca	42	Ge (72)	0	1	5	20	40
Cr	52	Ge (72)	0	0.0008	0.004	0.016	0.032
Mn	55	Ge (72)	0	0.005	0.025	0.1	0.2
Fe	56	Ge (72)	0	0.05	0.25	1	2
Cu	65	Ge (72)	0	0.005	0.025	0.1	0.2
Zn	66	Ge (72)	0	0.02	0.1	0.4	0.8
Se	78	Te (130)	0	0.0004	0.002	0.008	0.016
Mo	95	Ge (72)	0	0.0008	0.004	0.016	0.032

## ■ Analytical Conditions

Analysis was performed on Shimadzu's ICPMS-2030 Inductively Coupled Plasma Mass Spectrometer using the analytical conditions shown in Table 3.

The ICPMS-2030 is equipped with a high-sensitivity, high-resolution collision cell that improves removal efficiency for polyatomic ions such as 40Ar/38Ar that interfere with 78Se and improves transmission efficiency for analyte ions.

As a result, the ICPMS-2030 can perform highly sensitive analysis of trace levels of Se using helium as the only collision gas. Analysis times can also be shortened since there is no need to switch between different analytical conditions, and running costs can be reduced due to Mini-Torch Plasma System that consumes only 9 L/min of plasma gas.

Table 3 Analytical Conditions

Instrument:	: ICPMS-2030
RF Frequency Power	: 1.2 kW
Sampling Depth	: 7.0 mm
Plasma Gas	: 9.0 L/min
Auxiliary Gas	: 1.10 L/min
Carrier Gas	: 0.70 L/min
Collision Gas	: He
Collision Gas Flowrate	: 8.0 mL/min
Torch	: Mini-torch
Sampling Cone	: Ni cone
Skimmer Cone	: Ni Cone
Chamber	: Electronically-cooled cyclone chamber
Nebulizer	: 07UES nebulizer

## ■ Analysis of Certified Standard Reference Material SRM 1849a

Table 4 shows mean results from three measurements performed on a digested solution of the certified reference material (n = 2) and instrumental limits of quantification (ILOQ) calculated from blank calibration standard solutions. Good results were obtained with an RSD of 1.2 % (n = 6) or less and all elements were measured within the range of the certified value. The ILOQ were also sufficiently low considering the concentrations of the analyte elements, showing the ICPMS-2030 is sensitive enough to use for the analysis of mineral elements in infant formula.

## ■ Conclusion

This article describes an analysis of 12 elements in infant formula. Using helium as the only collision gas, all elements were analyzed simultaneously with good sensitivity and accuracy from trace levels of Se to high-concentration alkali elements.

The ICPMS-2030 facilitates the quality control of mineral elements in foods from trace-level minerals to high concentrations and can also reduce costs associated with routine daily analysis.

## ■ References

- 1) CODEX STAN 72-1981: STANDARD FOR INFANT FORMULA AND FORMULAS FOR SPECIAL MEDICAL PURPOSES INTENDED FOR INFANTS
- 2) AOAC Official Method 2015.06 /ISO 21424 Minerals and Trace Elements in Milk, Milk Products, Infant Formula and Adult/Pediatric Nutritional Formula Final action 2017

Table 4 Analysis of NIST SRM 1849a

Element	Certified Value	Measured Value	ILOQ* <sup>3</sup>
	mg/kg	mg/kg	mg/kg
Na	4265 ± 83	4236	4
Mg	1648 ± 36	1638	0.14
P	3990 ± 140	4120	1.2
K	9220 ± 110	9167	4
Ca	5253 ± 51	5186	3
Cr	1.072 ± 0.032	1.056	0.007
Mn	49.59 ± 0.97	48.17	0.008
Fe	175.6 ± 2.9	172	0.5
Cu	19.78 ± 0.26	19.72	0.01
Zn	151.0 ± 5.6	145.6	0.04
Se	0.812 ± 0.029	0.79	0.009
Mo	1.707 ± 0.04	1.689	0.0014

\*3: ILOQ = 10 × Standard deviation of calibration standard blank (n = 10) × Calibration curve slope