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Application News

Analysis of Heavy Metals in Baby Food Using ICP-MS

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User Benefits

- Heavy metals in various baby foods can be analyzed simultaneously with sufficient sensitivity.
- Stable analysis can be performed even for baby foods with high matrix content.
- Using the standard LabSolutions™ ICPMS rinse function, the time to clean the sample introduction system is minimized, which prevents carryover.

■ Introduction

Infants' metabolic pathways are still developing, and because of their lower body weight, they are known to be more susceptible to the effects of harmful substances. In the United States, the Baby Food Safety Act 2021[1], and in the EU, Commission Regulation (EU) 2023/915[2] and Commission Regulation (EU) 2024/1987[3] require the control of toxic metals contained in baby foods. ICP-MS is suitable for the analysis of trace amounts of toxic metal elements because it can perform multi-element simultaneous analysis with high sensitivity.

However, baby foods vary in matrix composition, which raises concerns about non-spectral interferences and long-term stability during ICP-MS analysis. This Application News describes the analysis of five baby foods with different matrices using the ICPMS-2040/2050 (Fig. 1). Additionally, spike recovery tests and long-term stability were assessed based on the Food and Drug Administration (FDA) Elemental Analysis Manual (EAM) Section 4.7[4].

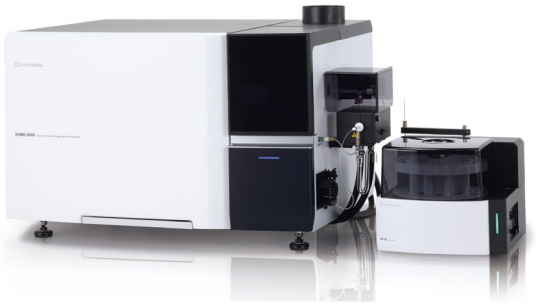


Fig. 1 ICPMS-2040/2050 and AS-20 Autosampler

■ Samples

Five baby foods with different matrices were selected:

- Infant formula
- Rice cereal
- Strained corn
- Strained pumpkin and sweet potato
- Apple jelly

■ Sample Preparation

The sample preparation procedure is shown in Fig. 2.

(1) Place approximately 0.5 g of the sample, 4 mL of nitric acid, 1 mL of hydrogen peroxide, and 0.5 mL of hydrochloric acid into each digestion vessel. To perform spike recovery tests, standard solutions of the target elements are added to some of the vessels.

(2) Heat in a microwave digestion system at 200 °C for 20 minutes.

(3) Cool to room temperature, then dilute to 25 mL with pure water (50 times dilution).

To confirm contamination levels for each element during digestion, a method blank solution was prepared using the same method as for the sample solution.

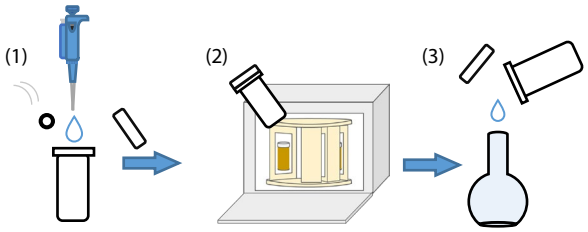


Fig. 2 Sample Pretreatment Procedure

■ Standard Samples

- Calibration Samples: Prepared by combining commercially available single-element standard solutions and adding nitric acid and hydrochloric acid. The concentration of the target elements in each calibration standard is shown in Table 1.
- Internal Standard Solution (ISTD): Prepared by combining commercially available single-element standard solutions of Ga, Te and Ir and adding nitric acid to 8 % v/v and hydrochloric acid to 0.5 % v/v. The concentrations in the internal standard solution were 200 µg/L. Additionally, isopropyl alcohol (IPA) was prepared to a concentration of 20 % v/v to suppress any impact on analysis values caused by organic substances in the samples.
- Initial Calibration Verification Sample (ICV) and Continuous Calibration Verification Sample (CCV): Prepared to match the concentrations of STD3.
- Continuous Calibration Verification Blank Sample (CCB): Prepared to match the concentrations of STD1.

Table 1 Concentrations of Calibration Standards

Element	STD1 (µg/L)	STD2 (µg/L)	STD3 (µg/L)	STD4 (µg/L)	STD5 (µg/L)	STD6 (µg/L)	STD7 (µg/L)
Cr, Ni, As, Se, Mo, Cd, Sn, Tl, Pb	0	10	20	50			
Hg	0	0.2	0.4	1			
Mn, Cu, Zn	0	10	20	50	1000	2000	5000
Nitric acid	8 v/v%						
Hydrochloric acid	0.5 v/v%						

■ Equipment Configuration and Analysis Conditions

The equipment configuration is shown in Table 2. To reduce running costs, a mini-torch with lower argon gas consumption compared to conventional torches was used for analysis. Additionally, to streamline sample preparation, an Online Internal Standard Kit was utilized for online addition.

The analysis conditions are detailed in Table 3. After analyzing high-concentration samples, the sample introduction system can influence the analysis values of subsequent samples (carryover). If carryover occurs, re-measurement is necessary, which consumes additional time and samples. With LabSolutions ICPMS, rinse solutions can be set for each sample, enabling effective cleaning in minimal time. Normal cleaning was performed using pure water (Rinse solution 1), and an additional acid rinse solution (Rinse solution 2) was introduced only after high-concentration samples.

For example, Table 4 shows the difference in carryover concentration when measuring a blank sample after introducing a sample with a CCV concentration, comparing the case where rinse solution 2 is added for an additional 240 seconds to the case where it is not. Among the elements measured this time, Se and Hg tend to have a higher risk of carryover, but by introducing rinse solution 2, carryover was reduced to below the detection limit.

Moreover, even if unexpectedly high-concentration samples are introduced, the Extended Rinsing function allows for automatic additional rinsing (Fig. 3). This eliminates the need to repeatedly check analysis values or to set a uniform cleaning time uniformly, thus reducing the verification effort, the amount of rinse solution required, and the analysis time while preventing carryover.

Furthermore, to minimize time spent cleaning the sample introduction system, ProActive Rinsing was used (Fig. 4). When measuring multiple samples, starting the rinse in advance during measurement reduces the measurement time for subsequent samples.

Table 2 Equipment Configuration

Instrument:	ICPMS-2040/2050
Nebulizer:	Nebulizer DC04
Chamber:	Cyclone chamber
Torch:	Mini-torch
Sampling Cone:	Nickel
Skimmer Cone:	Nickel
Autosampler:	AS-20
Internal Standard Elements:	Online Internal Standard Kit (sample: internal standard = about 9: 1)

Table 3 Analysis Conditions

RF Power:	1.20 kW	
Sampling Depth:	5 mm	
Pump Speed:	15 rpm	
Plasma Gas Flowrate:	9.0 L/min	
Auxiliary Gas Flowrate:	1.10 L/min	
Carrier Gas Flowrate:	0.45 L/min	
Dilutions Gas Flowrate:	0.40 L/min	
Rinse Solution 1	Pure water	
Rinse Solution 2	HNO_3 8 % v/v HCl 2 % v/v	
Cell Condition:	He	H_2*
Cell Gas:	He	H_2
Cell Gas Flowrate:	7.0	7.0
Cell Voltage:	-40	-30
Energy Filter:	7.0	7.0

* The cell condition H_2 is available only for ICPMS-2050.

Table 4 Difference in Carryover Depending on whether Rinse Solution 2 is Introduced

Element	Cell condition	IDL (µg/L)	Without rinse 2 (µg/L)	With rinse 2 (µg/L)
^{78}Se	He	0.01	0.12	<
^{202}Hg	He	0.0006	0.001	<

<: below the limit of detection

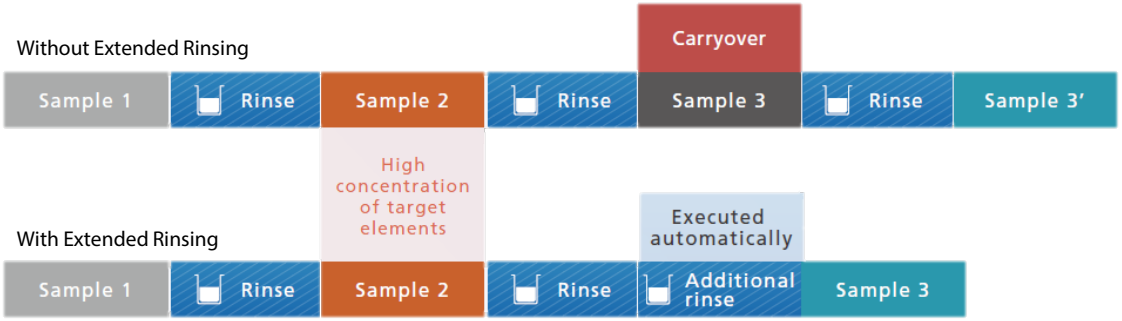
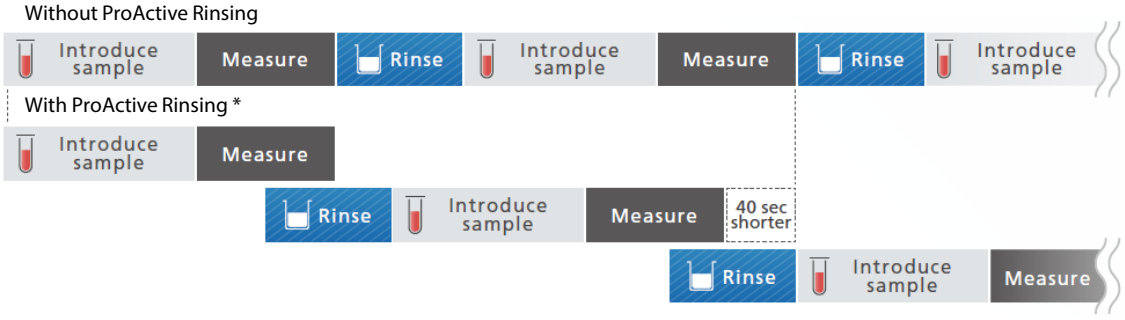


Fig. 3 Extended Rinsing



* The time available for pre-rinsing depends on the conditions and the number of elements to be analyzed.

Fig. 4 ProActive Rinsing

■ Analysis Results

Thirteen regulated elements, including As, Cd, Pb, Hg, Ni, and Sn, were analyzed simultaneously in five baby foods with different matrices. As shown in Table 5, the detection limits were less than one-tenth of the regulatory values.

To evaluate the impact of the matrix on the analysis values, spike recovery tests were conducted. The results are shown in Table 7. Recovery rates of 88 - 110 % for all samples and elements were obtained, indicating that the impact of the matrix on analysis values was sufficiently minimized. This result falls within the EAM4.7 range of 80 - 120 %.

Table 5 Regulatory Values for Toxic Metals in Baby Foods and Detection Limits

Element	Baby Food Safety Act 2021 (µg/kg)	EU 2023/915 (µg/kg)	EU 2024/1987 (µg/kg)	IDL (µg/kg)
Inorganic As	10 (Non-Cereal)	20		0.2 *1
	15 (Cereal)			
Cd	5 (Non-Cereal)	20 (placed on the market as powder and manufactured from soy protein isolates, alone or in a mixture with cows' milk proteins)		0.1
	10 (Cereal)	40 (Baby food and processed cereal-based food)		
Pb	5 (Non-Cereal)	20		0.05
	10 (Cereal)			
Hg	2	10		0.03
Ni			3000 (Processed cereal-based food for infants and young children)	1
			500 (others)	
Sn		50000		0.4

IDL: Instrument detection limit: 3σ × calibration curve gradient × {25 (mL) / 0.5 (g)}
*1: as Total As

Table 6 Results of Baby Food Analysis

Element	Cell condition	ISTD	IDL (µg/kg)	Quantitative results in the sample (µg/kg)				
				Infant formula	Rice cereal	Pumpkin and sweet potato	Corn	Apple jelly
^{52}Cr	He	^{71}Ga	1	33	171	6	9	15
^{55}Mn	He	^{71}Ga	0.2	379	9500	422	881	168
^{60}Ni	He	^{71}Ga	1	9	123	10	23	13
^{65}Cu	He	^{71}Ga	0.2	3620	2190	238	247	128
^{66}Zn	He	^{71}Ga	2	33000	74500	1100	2800	90
^{75}As*1	He	^{128}Te	0.2	5.4	110	1.3	0.9	3.3
^{78}Se	He	^{128}Te	0.5	91.5	130	<	<	<
^{78}Se	H_2 *2	^{128}Te	0.1	86.7	129	0.3	0.5	<
^{95}Mo	He	^{71}Ga	0.2	54.0	654	39.5	18.5	3.8
^{111}Cd	He	^{71}Ga	0.1	5.9	7.5	1.8	1.1	1.0
^{118}Sn	He	^{71}Ga	0.4	1.9	2.7	1.6	6.5	99.9
^{202}Hg	He	^{193}Ir	0.03	<	1.49	<	<	0.26
^{205}Tl	He	^{193}Ir	0.1	0.4	0.4	2.5	0.6	0.8
^{sum}Pb	He	^{193}Ir	0.05	3.30	0.90	0.90	1.50	2.55

IDL: Instrument detection limit: 3σ × calibration curve gradient × {25 (mL) / 0.5 (g)}
*1: as Total As
*2: The cell condition H_2 is available only for ICPMS-2050.
^{sum}Pb:Sum of ^{206}Pb, ^{207}Pb, and ^{208}Pb
<: below the limit of detection (Method blank subtracted)

Table 7 Spike Recovery Test

Element	Cell condition	ISTD	IDL (µg/L)	Spiked conc. (µg/L)	Spike recovery (%)				
					Infant formula	Rice cereal	Pumpkin and sweet potato	Corn	Apple jelly
^{52}Cr	He	^{71}Ga	0.02	20	110	98	103	101	101
^{55}Mn	He	^{71}Ga	0.003	1000	102	103	105	105	106
^{60}Ni	He	^{71}Ga	0.02	20	108	95	99	98	99
^{65}Cu	He	^{71}Ga	0.003	1000	108	95	98	98	98
^{66}Zn	He	^{71}Ga	0.03	1000	99	88	93	94	93
^{75}As *1	He	^{128}Te	0.004	20	101	99	103	102	103
^{78}Se	He	^{128}Te	0.01	20	100	99	103	101	102
^{78}Se	H_2 *2	^{128}Te	0.002	20	96	97	101	100	101
^{95}Mo	He	^{71}Ga	0.003	20	106	103	103	103	103
^{111}Cd	He	^{71}Ga	0.002	20	97	95	91	92	90
^{118}Sn	He	^{71}Ga	0.008	20	103	102	101	101	97
^{202}Hg	He	^{193}Ir	0.0006	1	92	99	95	95	93
^{205}Tl	He	^{193}Ir	0.002	20	93	101	104	102	103
^{sum}Pb	He	^{193}Ir	0.001	20	95	105	109	106	108

IDL: Instrument detection limit : 3σ × calibration curve gradient × {25 (mL) / 0.5 (g)}
*1: as Total As
*2: The cell condition H_2 is available only for ICPMS-2050.
^{sum}Pb:Sum of ^{206}Pb, ^{207}Pb, and ^{208}Pb
Spike recovery (%) = (Spiked sample - Unspiked sample) / Spiked concentration × 100

■ Long-Term Stability Evaluation

To evaluate long-term stability, particularly for the high-matrix Infant formula, 50 samples were analyzed consecutively. After measuring the calibration samples, we measured the ICV, then the samples. To confirm the validity of the calibration during the analysis, CCV and CCB were measured every ten samples.

The quantitation values for CCB were confirmed to be below the Analytical Solution Quantification Level (ASQL, 30σ × calibration curve slope[5]) for all elements. Using LabSolutions ICPMS, rinse solutions of different compositions can be set for each sample. This prevents carryover with minimal rinse time.

The recovery rates for all ICV and CCV measurements are shown in the Fig. 5. All ICV and CCV measurements achieved recovery rates within the acceptable range 90 - 110 % as specified by EAM4.7.

Additionally, Fig. 6 illustrates the variations in intensity of the internal standard elements during the analysis. The intensity of each internal standard element in STD1 is set at 100 %. The variations in intensity of the internal standard elements over approximately six hours of analysis remained within the EAM4.7 specified range of 60 - 120 %.

■ Conclusion

In this Application News, elemental analysis of baby food was conducted using the ICPMS-2040/2050. We achieved detection limits that met stringent standards. The spike recovery tests yielded good results for all samples with different matrices, confirming the validity of the analysis. Additionally, we analyzed 50 samples of infant formula with a high matrix over approximately six hours and confirmed good stability.

The ICPMS-2040/2050 offers high sensitivity, accurate analysis, and good stability. Its Extended Rinsing and ProActive Rinsing functions effectively clean the sample introduction system, preventing carryover. Therefore, the ICPMS-2040/2050 is a suitable instrument for elemental analysis in baby food.

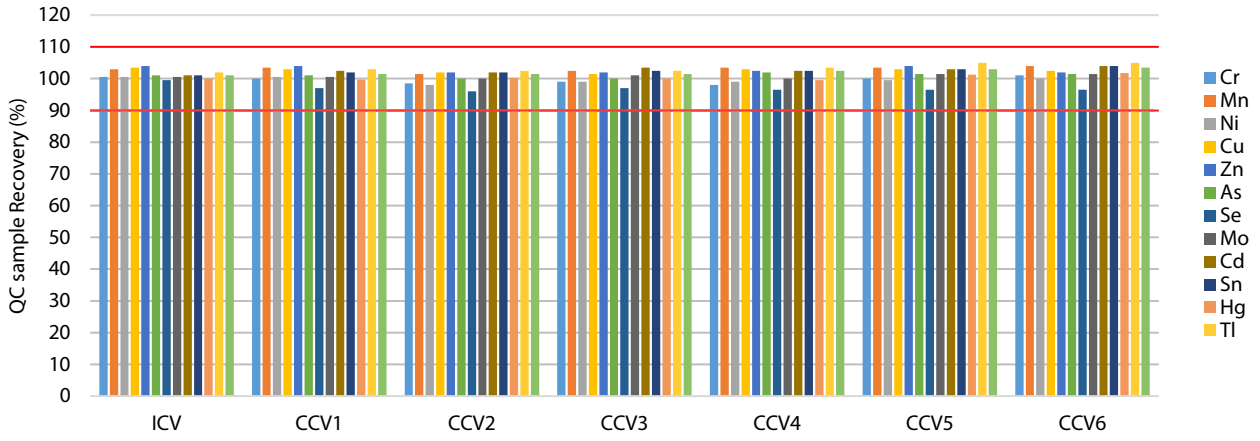


Fig. 5 ICV and CCV Recoveries

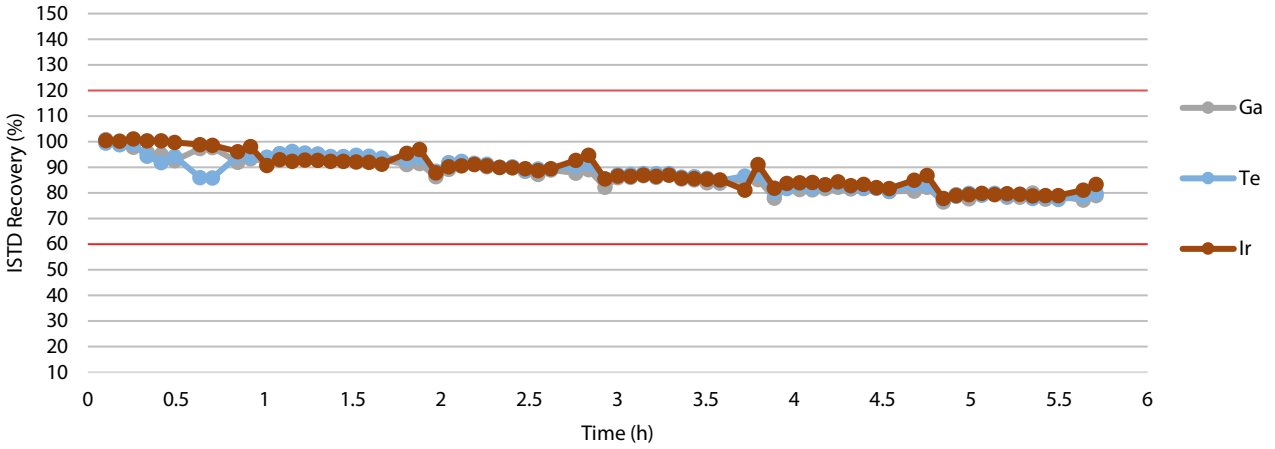


Fig. 6 Stability of ISTD

<References>

1) US House of Representatives, The Baby Food Safety Act of 2021, accessed May 2021
2) COMMISSION REGULATION (EU) 2024/1987 of 30 July 2024 amending Regulation (EU) 2023/915 as regards maximum levels of nickel in certain foodstuffs
3) COMMISSION REGULATION (EU) 2023/915 of 25 April 2023 on maximum levels for certain contaminants in food and repealing Regulation (EC) No 1881/2006
4) U.S. Food and Drug Administration Elemental Analysis Manual 4.7 Inductively Coupled Plasma-Mass Spectrometric Determination of Arsenic, Cadmium, Chromium, Lead, Mercury, and Other Elements in Food Using Microwave Assisted Digestion, Version 1.2 (February 2020)
5) U.S. Food and Drug Administration Elemental Analysis Manual 3.2 Terminology, Version 3.0 (December 2021)

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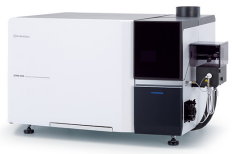

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