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Sensitive and Rapid Analysis of Inorganic Arsenic in Food and Animal Feed Samples Using LC-ICP-MS

Kana Matsuno and Kosuke Naka
Shimadzu Corporation, Analytical & Measuring Instruments Division, Japan

1. Introduction

- Inorganic arsenic (iAs) is more toxic than organic arsenic, and its monitoring in food and feed is required worldwide.
- Because organic arsenic and chlorine in food matrices can interfere with iAs determination, effective chromatographic separation is essential. This study quantified iAs in various foods and feeds by LC-ICP-MS, following EN 16802:2016[1)] and EN 17374:2020[2)].

2. Instrument Configuration

- Samples were analyzed using the LC-ICP-MS system combining the ICPMS-2040/2050 with a Nexera™ XS inert, controlled by LabSolutions™ ICPMS TRM software (Fig. 1). The column was HAMILTON PRP-X100 (10.0 µm, 250 mm × 4.1 mm), and the eluent was 100 mM ammonium carbonate + 3% v/v methanol + 400 mg/L sodium sulfate (pH 9.3).
- The mini torch for ICP-MS reduces argon gas consumption to 11 L/min, roughly two-thirds that of a conventional torch, lowering running costs in lengthy LC-ICP-MS analyses.

Fig. 1 Workflow of this study

3. Regulatory Values and Detection Limits

- The limit of detection (LOD) were sufficient even for the strictest iAs limit under the Baby Food Safety Act 2021 (Table 1).

Table 1 Regulatory Values and Detection Limits

Samples	Regulatory Values (mg/kg)				LODs in samples (mg/kg)	LOD in analytical solution (µg/L)
	(EU) 2023/915[3)]	(EU) 2025/1891[4)]	The Baby Food Safety Act of 2021[5)]	DIRECTIVE 2002/32/EC[6)]		
Brown Rice	0.25	-	-	-	0.0004	0.02
Hijiki	-	-	-	-	0.04	
Rice Cereal	0.02	-	0.015	-	0.0004	
Apple Jelly	0.02	-	0.01	-	0.0004	
Strained Corn	0.02	-	0.01	-	0.0004	
Infant Formula	0.02	-	0.01	-	0.0004	
Sea Bream	-	0.1	-	-	0.0004	
Tuna	-	0.1	-	-	0.0004	
Squid	-	0.05	-	-	0.0004	
Ham	-	-	-	-	0.0004	
Orange Juice	0.02	-	-	-	0.0002 (mg/L)	
Japanese Sake	-	-	-	-	0.00004 (mg/L)	
Animal Feed	-	-	-	2	0.002	

4. Oxidation from As(III) to As(V)

- Without pretreatment, As(III) appeared as a broad peak, but after pretreatment a peak was obtained at the same retention time as As(V).
- When the 5 µg/L As(III) check standard (prepared from As(III)) was quantified using the iAs calibration curve prepared from As(V) standards, the measured concentration was 4.83 µg/L. These results confirm that As(III) is oxidized to As(V) by the pretreatment (Fig. 2).

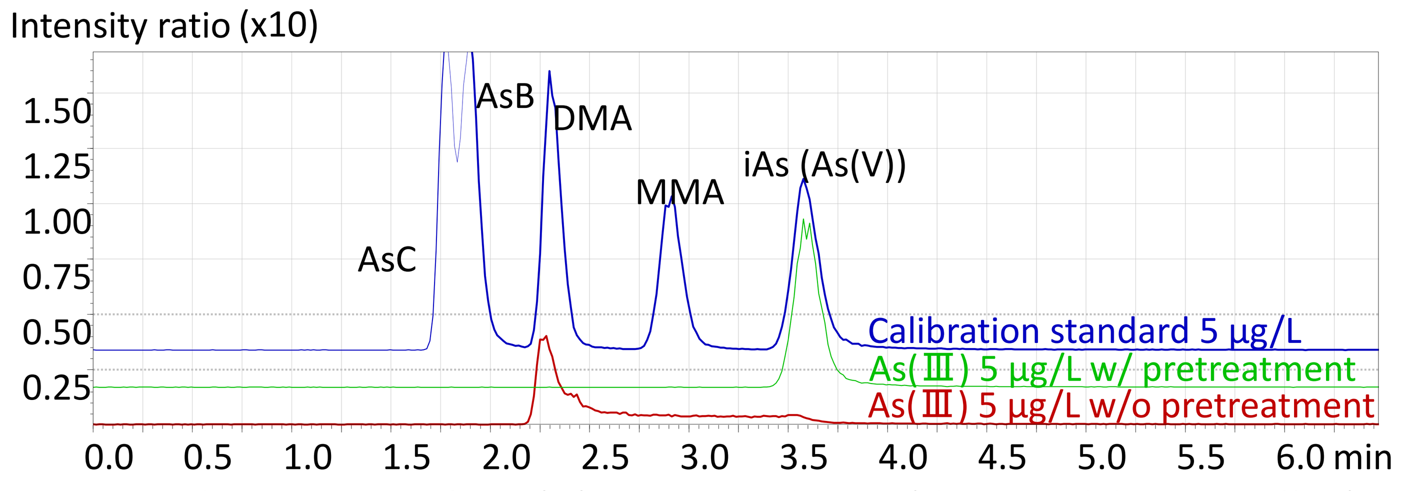

Fig. 2 Chromatograms for As(III) Standard Solutions (shown with baseline offset)

5. Removal of Cl Interference

- The Cl and iAs peaks were resolved under the chromatographic conditions used.
- The use of helium collision mode effectively suppressed $^{40}Ar^{35}Cl$ interference at m/z 75 (Fig. 3).

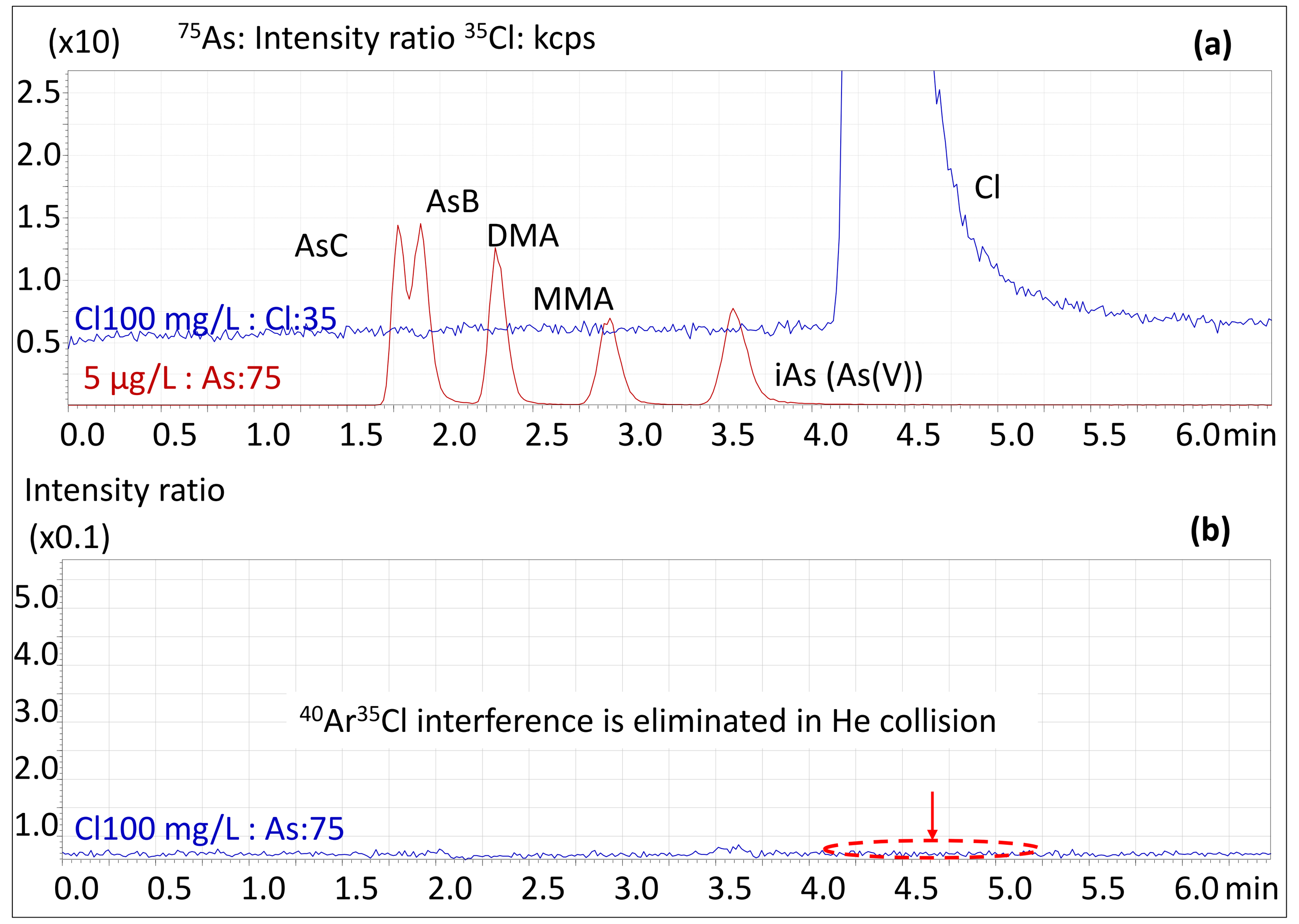

Fig. 3 Removal of Interference derived from Cl
(a) Separation of iAs from Cl, (b) Removal of $^{40}Ar^{35}Cl$ Interference in He Collision

6. Separation of iAs from Organic As in Samples

- Although tuna and squid contained large amounts of organic arsenic (AsB), the analysis of iAs in the 5 µg/L spiked seafood samples was not affected (Fig. 4).

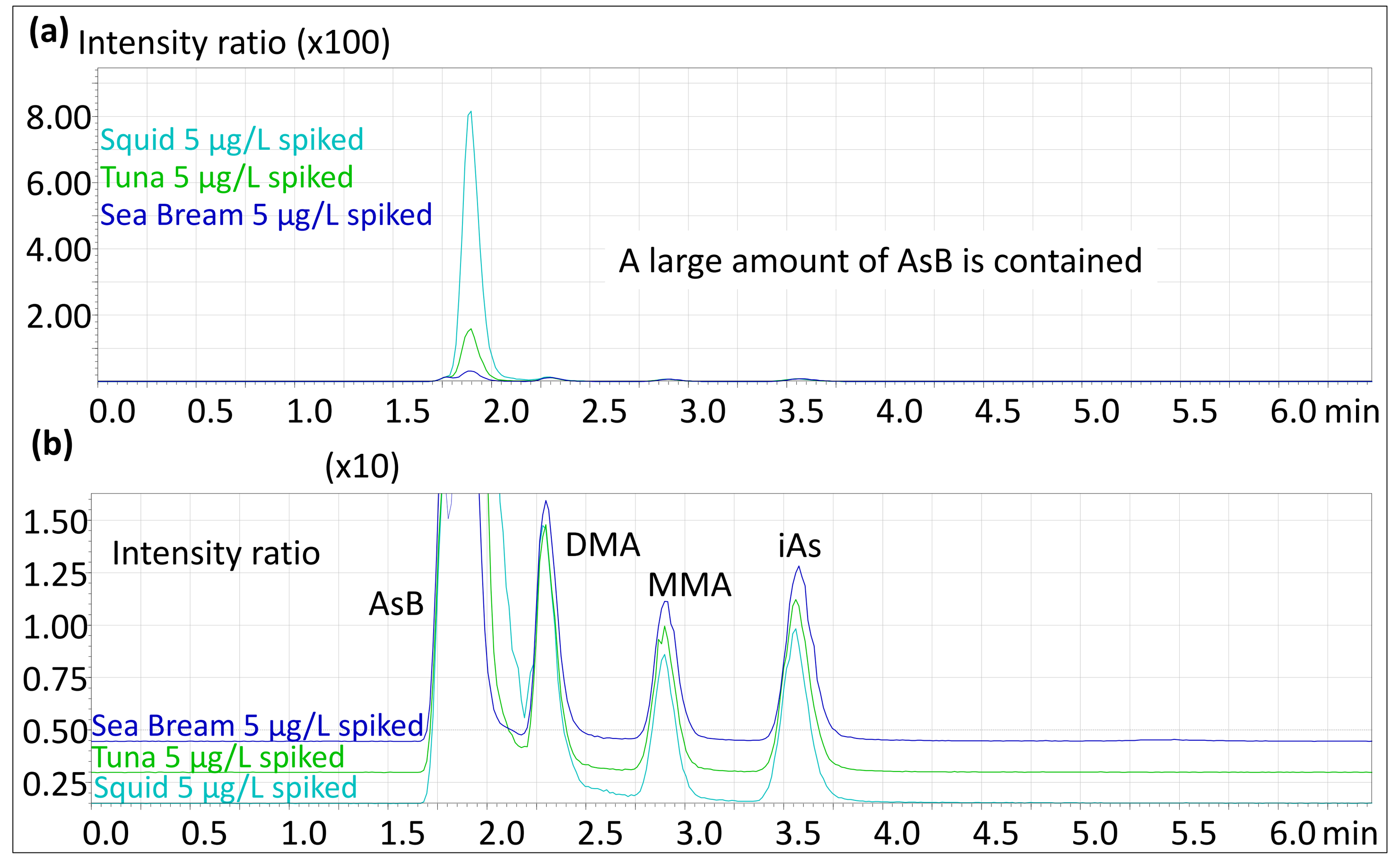

Fig. 4 Chromatograms of Seafood Spiked Sample
(a) Chromatograms of Seafood Spiked Sample, (b) Enlarged Chromatograms of Seafood Spiked Sample (shown with baseline offset)

7. Validation and Quantitative Analysis

- Quantified values for all three Certified Reference Materials (CRM) fell within their certified ranges (Table 2), confirming method validity.

Table 2 Results of CRM (iAs)

Samples	LODs in samples (mg/kg)	Certified Values (mg/kg)	Quantitative Results (mg/kg)
Brown rice NMIJ 7532-a	0.0004	0.298±0.008	0.291
Brown rice NMIJ 7533-a	0.0008	0.530±0.016	0.527
Hijiki NMIJ 7405-b	0.04	24.4±0.7*1	24.7

*1: Certified value as As(V)

- For the samples other than the CRMs, spike-recovery tests yielded 96–113% iAs recovery across food and feed matrices (Fig. 5), confirming method accuracy and reliability.
- Table 3 shows the quantified iAs results. Most samples contained little or no detectable iAs, while rice cereal and animal feed showed higher levels. This may be related to their grain-based ingredients.

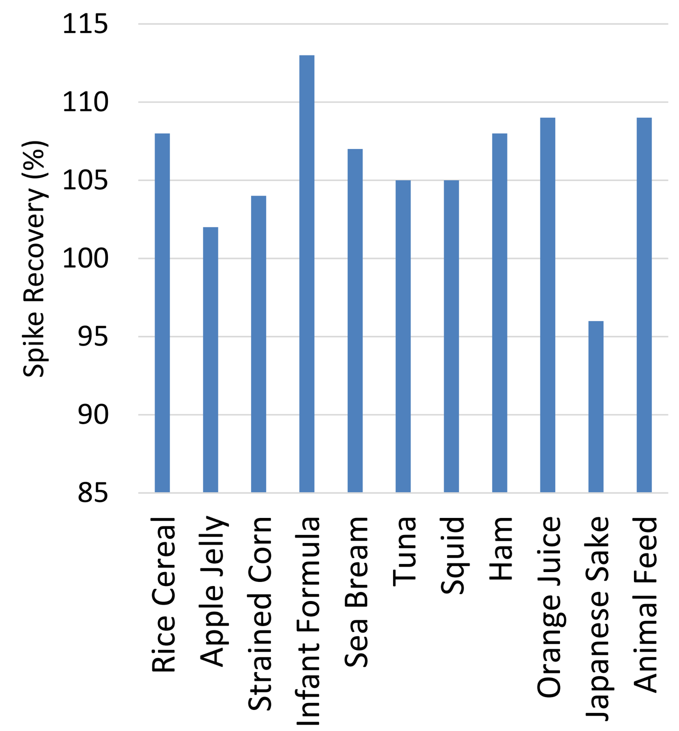

Fig. 5 Spike Recovery Test

Table 3 Quantitative Results of iAs in Samples

Samples	Quantitative Results in Samples (mg/kg)
Rice Cereal	0.0864
Apple Jelly	0.0024
Strained Corn	N.D.
Infant Formula	N.D.
Sea Bream	0.002
Tuna	N.D.
Squid	N.D.
Ham	N.D.
Orange Juice	N.D. (mg/L)
Japanese Sake	0.0011 (mg/L)
Animal Feed	0.013

N.D.: below the detection limit

8. Conclusion

- Quantitative analysis of inorganic arsenic using the Shimadzu LC-ICP-MS system was conducted on 14 different food and feed matrices.
- Inorganic arsenic was separated from organic arsenic and chlorine in various food and feed samples within 6.5 minutes.
- CRM analysis and spike recovery tests confirmed accurate inorganic arsenic analysis in a wide range of food and feed samples. Detection limits were low enough for even strict baby food regulations.

Reference

1) EN 16802:2016: Determination of inorganic arsenic in foodstuffs of marine and plant origin by anion-exchange HPLC-ICP-MS
2) EN 17374:2020: Determination of inorganic arsenic in animal feed by anion-exchange HPLC-ICP-MS
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) Commission Regulation (EU) 2025/1891 of 17 September 2025 amending Regulation (EU) 2023/915 as regards maximum levels of inorganic arsenic in fish and other seafood
5) US House of Representatives, The Baby Food Safety Act of 2021, accessed Jan 2026
6) DIRECTIVE 2002/32/EC OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 7 May 2002