

Application News

Analysis of Trace Elements in Table Salt Using ICPMS-2050

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User Benefits

- ◆ Elements in table salt can be accurately analyzed over a long period of time.
- ◆ Reduced running costs due to a mini-torch that uses less argon gas
- ◆ Avoids a complex investigation of conditions by using the analytical conditions from preset methods.

Introduction

The Codex general standard for contaminants and toxins in food and feed (Codex) establishes requirements for levels of toxic metal elements in foods¹⁾. Inductively coupled plasma mass spectrometry (ICP-MS) systems can perform highly sensitive quantitative determination of multiple elements simultaneously, making them suited to the analysis of even trace amounts of toxic metal elements in foods.

Food samples are typically high-matrix samples that can often impact the clogging of torch or interfaces such as skimmer cone, non-spectral interference, and long-term stability in ICP-MS analysis.

This Application News uses the ICPMS-2050 (Fig. 1) to analyze table salt, a food with a particularly high matrix concentration. This analysis was performed using a mini-torch that features low argon gas consumption. The long-term stability and spike recovery were assessed based on the Food and Drug Administration (FDA) Elemental Analysis Manual (EAM) Section 4.7²⁾.

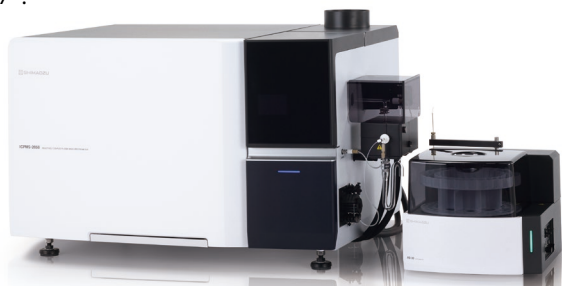


Fig. 1 ICPMS-2050 and AS-20 Equipment

Sample Preparation

The robustness of the ICPMS-2050 to sample matrix effects was evaluated using table salt, a food with a particularly high matrix concentration.

● Unspiked Sample

An unspiked sample was prepared by weighing out approx. 0.2 g of table salt, adding 2.5 mL of nitric acid, 0.25 mL of hydrochloric acid, and making the mixture up to 50 mL with pure water. The final concentration of table salt in the unspiked sample was approx. 0.4 %, the nitric acid concentration was 5 % v/v, and the hydrochloric acid concentration was 0.5 % v/v.

● Spiked Sample

A spiked sample was prepared by weighing out approx. 0.2 g of table salt, adding 2.5 mL of nitric acid, 0.25 mL of hydrochloric acid, adding commercially available single-element standard solutions of Al, Cr, Mn, Ni, Cu, Zn, As, Se, Mo, Cd, Sn, Hg, Tl, and Pb, then making the mixture up to 50 mL with pure water.

● Method Blank

A method blank was prepared by combining 2.5 mL of nitric acid with 0.25 mL of hydrochloric acid and making the mixture up to 50 mL with pure water.

Standard Samples

● Calibration Standards

Calibration standards were prepared by combining commercially available single-element standard solutions of Al, Cr, Mn, Ni, Cu, Zn, As, Se, Mo, Cd, Sn, Hg, Tl, and Pb 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

An internal standard solution was prepared by combining commercially available single-element standard solutions of Sc, Ge, Rh, Ir, and Bi and adding nitric acid to 5 % v/v and hydrochloric acid to 0.5 % v/v. The final concentration of Sc, Ge, Rh, Ir, and Bi in the internal standard solution was 2 mg/L.

● Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Samples

ICV and CCV samples were prepared with the same target element concentrations as STD3 in Table 1.

● Continuing Calibration Blank (CCB) Sample

A CCB sample was prepared with the same target element concentrations as STD1 in Table 1.

Table 1 Target Element Concentrations in Calibration Standards

Elements	Calibration Standard (µg/L)			
	STD1	STD2	STD3	STD4
Cr, Ni, As, Se, Mo, Cd, Sn, Tl, Pb	0	1	5	10
Hg	0	0.1	0.5	1
Al, Mn, Cu, Zn,	0	10	50	100
Nitric acid	5 % v/v			
Hydrochloric acid	0.5 % v/v			

Equipment Configuration and Analytical Conditions

The configuration of the ICP-MS system is shown in Table 2. To reduce running costs, analysis was performed with a mini-torch that consumes less argon gas than a typical plasma torch. To reduce the labor involved in sample preparation, the internal standard sample was added online using an online internal standard kit.

The analytical conditions used are shown in Table 3. A complex investigation of conditions was avoided by using the analytical conditions from a preset method in LabSolutions™ ICPMS.

Table 2 ICP-MS System Configuration

System:	ICPMS-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 Analytical Conditions

RF Power:	1.20 kW
Plasma Gas Flowrate:	9.0 L/min
Auxiliary Gas Flowrate:	1.10 L/min
Carrier Gas Flowrate:	0.35 L/min
Dilution Gas Flowrate:	0.55 L/min
Cell Gas:	He/H ₂

Quantitative Analysis

A calibration curve was prepared using the calibration standards shown in Table 1. The unspiked sample, spiked sample, method blank, ICV sample, CCV sample, and CCB sample were quantitatively analyzed by this calibration curve.

Detection Limits (DLs)

Detection limits are shown in Table 4. The detection limits were calculated using the standard deviation (σ) of the calibration blank (STD1). Detection limits for the target elements in table salt were all at least one order of magnitude below the reference levels listed in the Codex.

Table 4 Detection Limits

Element	Cell Gas	Internal Standard Element	IDL ($\mu\text{g/L}$)	DL in Table Salt (mg/kg)	Codex Reference Level (mg/kg)
²⁷ Al	H ₂	⁴⁵ Sc	0.09	0.02	–
⁵² Cr	He	⁷⁴ Ge	0.1	0.03	–
⁵⁵ Mn	He	⁷⁴ Ge	0.07	0.02	–
⁶⁰ Ni	He	⁷⁴ Ge	0.09	0.02	–
⁶⁵ Cu	He	⁷⁴ Ge	0.09	0.02	–
⁶⁶ Zn	He	⁷⁴ Ge	0.2	0.04	–
⁷⁵ As	He	⁷⁴ Ge	0.08	0.02	0.5
⁷⁸ Se	H ₂	⁷⁴ Ge	0.04	0.009	–
⁹⁵ Mo	He	¹⁰³ Rh	0.02	0.005	–
¹¹¹ Cd	He	¹⁰³ Rh	0.03	0.007	0.5
¹¹⁸ Sn	He	¹⁰³ Rh	0.07	0.02	–
²⁰² Hg	He	¹⁹³ Ir	0.009	0.002	0.1
²⁰⁵ Tl	He	²⁰⁹ Bi	0.009	0.002	–
sumPb	He	²⁰⁹ Bi	0.008	0.002	1

IDL: Instrument detection limit ($3\sigma \times$ calibration curve gradient)
sumPb: Measured as the sum of ²⁰⁶Pb, ²⁰⁷Pb, and ²⁰⁸Pb

Spike Recovery Test

A spike recovery test was performed to assess the effect of the sample matrix on analysis. The results are shown in Table 5. Good recovery of between 95 and 106 % was achieved for all elements, showing the sample matrix effect on the analysis remained allowably small. All recovery levels remained within 80 to 120 % as required by EAM Section 4.7.

Table 5 Spike Recovery Test

Element	Unspiked Sample ($\mu\text{g/L}$)	Spiked Conc. ($\mu\text{g/L}$)	Spiked Sample ($\mu\text{g/L}$)	Spike Recovery (%)
²⁷ Al	N.D.	10	10.5	105
⁵² Cr	N.D.	1	0.99	99
⁵⁵ Mn	5.47	10	15.5	100
⁶⁰ Ni	N.D.	1	1.05	105
⁶⁵ Cu	N.D.	10	9.68	97
⁶⁶ Zn	N.D.	10	9.5	95
⁷⁵ As	N.D.	1	1.05	105
⁷⁸ Se	0.12	1	1.09	97
⁹⁵ Mo	0.05	1	1.10	105
¹¹¹ Cd	N.D.	1	0.96	96
¹¹⁸ Sn	N.D.	1	0.98	98
²⁰² Hg	N.D.	0.1	0.106	106
²⁰⁵ Tl	N.D.	1	1.01	101
sumPb	0.741	1	1.77	103

Spike recovery (%) = (Spiked sample - Unspiked sample) / Spiked concentration \times 100
N.D.: Below instrument detection limit (IDL)

Evaluation of Long-Term Stability

The long-term stability was assessed by the analysis for around 10 hours. The analytical sequence used was the calibration standards followed by the ICV sample, method blank, then test samples. The CCV sample and CCB sample were assayed every 10 test samples to confirm the validity of the calibration curve over the course of the evaluation. The analytical sequence is shown in Fig. 2.

Recovery rates from the ICV samples and CCV samples are plotted in Fig. 3. Recoveries from all ICV and CCV samples were within 90 and 110 % (red dashed lines) as required by EAM Section 4.7. All elements in the CCB sample were also assayed below the analytical solution quantification level (ASQL, $30\sigma \times$ calibration curve slope³).

The change in signal intensity of the internal standard elements over the course of the evaluation is shown in Fig. 4, where the signal intensity of internal standard elements in STD1 is taken as 100 %. The change in signal intensity of the internal standard elements during the evaluation spanning around 10 hours remained within 60 to 120 % (red dashed lines) as required by EAM Section 4.7.

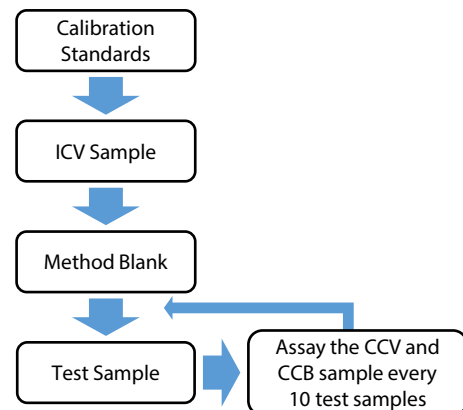


Fig. 2 Analytical Sequence

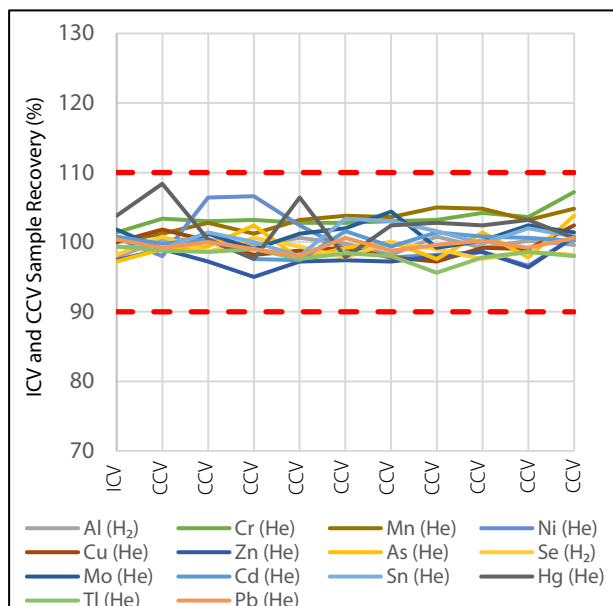


Fig. 3 ICV and CCV Sample Recovery

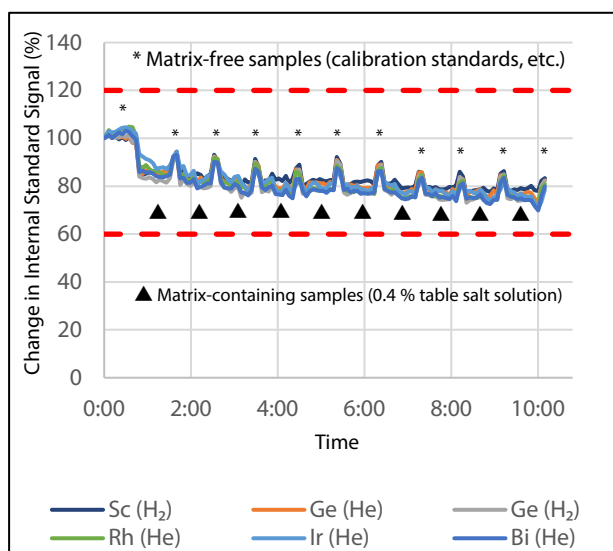


Fig. 4 Change in Signal of Internal Standard Elements during Approx. 10 Hours of Analysis

<References>

- 1) Codex general standard for contaminants and toxins in food and feed (CODEX STAN 193-1995)
- 2) 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)
- 3) U.S. Food and Drug Administration Elemental Analysis Manual 3.2 Terminology, Version 3.0 (December 2021)

■ Conclusion

This Application News describes the use of the the ICPMS-2050 with a mini-torch to measure trace elements in table salt. The detection limits were sufficient to measure trace elements in table salt. Spike recovery testing showed good results even with high matrix concentration samples, confirming the accuracy of the analysis. The stability was also confirmed over the course of 10 hours of analysis.

The ICPMS-2050 performs highly sensitive, accurate, and stable analysis. The ICPMS-2050 can also reduce running costs by using a mini-torch that consumes less argon gas. Analytical conditions can also be entered from preset methods, simplifying analysis by avoiding a complex investigation of conditions.

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