

Application News

Inductively Coupled Plasma Mass Spectrometer

Determination of Metal Elements in Edible Oils Using ICP-MS

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User Benefits

- ◆ It is possible to accurately analyze metal elements in edible oils.
- ◆ Edible oils can be analyzed using only dilution with organic solvents, which simplifies sample preparation and improves throughput.
- ◆ The use of solvent-resistant peristaltic pump tubing enables the addition of internal standard elements in-line, even during the analysis of organic solvents, thereby eliminating the need for manual addition.

Introduction

Edible oils are essential in our diet. Analyzing metal elements in edible oils is crucial for ensuring food safety and preserving oil quality. The Codex General Standard for Contaminants and Toxins in Food and Feed¹⁾ (hereinafter referred to as CODEX) requires the management of heavy metals (such as arsenic and lead) present in edible oils. Additionally, metal ions such as copper, iron, and manganese in edible oils can act as oxidation catalysts, affecting the deterioration of the oil.

For the analysis of metal elements in edible oils, methods involving the acid digestion of the oil followed by analysis using ICP Optical Emission Spectrometry (ICP-OES) or ICP Mass Spectrometry (ICP-MS) are known. While digestion with acid can remove the organic matrix, it also carries the risk of volatilizing or contaminating the analytes during the complex sample preparation process, which can lead to errors in the analysis results. Furthermore, the complex sample preparations are time-consuming and limit throughput. Another method involves diluting edible oils with organic solvents and analyzing them by ICP-OES. However, with this method, it can be challenging to achieve sufficient sensitivity for the analysis of harmful trace elements such as As and Pb.

In this Application News, the ICPMS-2050 (Fig. 1) was used to analyze 25 metal elements by simply diluting edible oils with an organic solvent. Spike recovery tests are also performed to verify the validity of the analytical results.

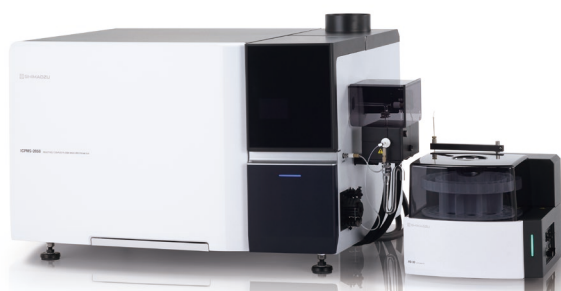


Fig. 1 ICPMS-2050 and AS-20

Samples and Reagents

- Samples
Three types of commercially available edible oils were prepared as samples: vegetable oil, rice oil, and olive oil.

- Diluent
PremiSolv (CONOSTAN) was used as the diluent.

- Reagents for standards
The oil-based mixed standard solution S-21 and the oil-based single-element standard solutions for Li, P, K, Co, As, Se, Sb, Be, Y, In, and Bi (CONOSTAN), the oil-based single-element standard solution for Sc (LGC Standards), and the toluene-based alkyl mercury standard solution (Fujifilm Wako Pure Chemical Corporation) were used.

Preparation of Samples and Standards

- Sample preparation
Approximately 0.4 g of each edible oil was weighed and diluted 10 times (w/w) with PremiSolv. Similarly, spiked samples were prepared by adding standard solutions.
- Calibration standards
S-21 and single-element standard solutions were mixed and diluted with PremiSolv to prepare the calibration standards. The concentrations of each element in the calibration standards are shown in Table 1.
- Internal standard solution
Oil-based single-element standard solutions of Be, Sc, Y, In, and Bi were mixed and diluted with PremiSolv. The concentration of Be and Sc in the internal standard solution was 250 µg/kg, while the concentration of Y and In was 25 µg/kg, and Bi was 5 µg/kg.

Configurations and Analytical Conditions

The configuration of the ICP-MS system is shown in Table 2. The "Organic Solvent Injection System" was used as the sample introduction system. By using a platinum sampling cone, damage to the cone from prolonged exposure to organic solvents can be minimized. Additionally, the use of solvent-resistant peristaltic pump tubing enables the in-line addition of internal standard elements, thereby streamlining the sample preparation process.

The analytical conditions are shown in Table 3. To prevent carbon deposits from forming on the interface due to the organic solvents, a mixed gas of argon (70 %) and oxygen (30 %) was introduced into the quadrupole structure organic solvent torch. The analytical conditions used for edible oils can be easily registered from the preset methods of LabSolutions™ ICPMS (Version 2.10 and later), eliminating the need for complex condition testing.

Table 1 Concentrations of Analytes in Calibration Standards

Elements	Calibration Standards (µg/kg)					
	STD0	STD1	STD2	STD3	STD4	STD5
Li, V, Cr, Mn, Fe, Co, Ni, Cu, As, Se, Mo, Cd, Sn, Sb, Pb	0	1	5	10	50	100
Na, Mg, Al, Si, K, Ca, Ti, Zn	0			10	50	100
Hg	0	0.1	0.5	1	5	
P	0			1000	5000	10000

Table 2 ICP-MS System Configuration

Instrument	: ICPMS-2050
Nebulizer	: Nebulizer DC04
Chamber	: Cyclone Chamber
Torch	: Organic Solvent Torch
Sampling Cone	: Platinum
Skimmer Cone	: Nickel
Auto Sampler	: AS-20 (Rinse station for organic solvents)
Peristaltic Pump Tubing	: I.D. 0.76 mm for sample ^{*1} I.D. 0.38 mm for internal standards ^{*2} (materials: Solva PVC)
Internal Standard Elements	: Online Internal Standard Kit (for Organic Solvents) ^{*3} (sample: internal standard = about 4:1)

I.D.: Internal Diameter

*1: P/N: S018-31558-61

*2: P/N: S018-31558-62

*3: P/N: S211-95010-42

Table 3 Analytical Conditions

RF Power	: 1.60 kW
Sampling Depth	: 8.0 mm
Plasma Gas Flowrate	: 20.0 L/min
Auxiliary Gas Flowrate	: 0.50 L/min
Carrier Gas Flowrate	: 0.60 L/min
Dilution Gas Flowrate	: 0 L/min
Ar-O ₂ Mixed Gas Flowrate	: 0.35 L/min
Chamber Temperature	: -5 °C
Pump Rotation Speed	: 15 r.p.m. (Low) / 45 r.p.m. (High)
Cell Gas	: No Gas He H ₂
Cell Gas Flowrate	: - 6.0 mL/min 7.0 mL/min
Cell Voltage	: - -40 V -35 V
Energy Filter	: - 7.0 V 7.0 V

■ Removal of Interference and Detection Limit

To analyze metal elements with high sensitivity in organic solvents, it is necessary to eliminate interferences caused by carbon from the organic solvent. For example, there are carbon-derived polyatomic ion interferences such as ¹²C¹²C⁺ on ²⁴Mg, ¹²C¹⁶O⁺ on ²⁸Si, and ⁴⁰Ar¹²C⁺ on ⁵²Cr. By using collision mode with helium gas or reaction mode with hydrogen gas, these interferences can be eliminated, enabling sensitive analysis. As an example, Fig. 2 shows the calibration curve for Cr under each condition. In the No Gas mode, the influence of ⁴⁰Ar¹²C⁺ results in a high background equivalent concentration (BEC), making it challenging to analyze trace amounts of ⁵²Cr. However, in collision mode and reaction mode, interferences can be eliminated, reducing the BEC and enabling the analysis of trace amounts of ⁵²Cr.

Additionally, the detection limits (DLs) are shown in Table 4. The detection limits were calculated as the concentration that gives a signal equivalent to three times the standard deviation (σ) of the calibration blank sample (STD0). Detection limits of arsenic and lead in edible oils were sufficient to monitor the CODEX standard values of 100 $\mu\text{g/kg}$ (0.1 mg/kg).

Table 4 Detection Limits (DLs)

Elements	Cell Gas	Internal Standards	IDL ($\mu\text{g/kg}$)	DL in edible oil ($\mu\text{g/kg}$)
⁷ Li	No Gas	⁹ Be	0.06	0.6
²³ Na	No Gas	⁴⁵ Sc	2	20
²⁴ Mg	H ₂	⁴⁵ Sc	0.4	4
²⁷ Al	He	⁴⁵ Sc	0.7	7
²⁸ Si	H ₂	⁴⁵ Sc	3	30
³¹ P	He	⁴⁵ Sc	100	1000
³⁹ K	H ₂	⁴⁵ Sc	0.7	7
⁴⁰ Ca*	H ₂	⁴⁵ Sc	0.2	2
⁴⁷ Ti	He	⁴⁵ Sc	0.5	5
⁵¹ V	H ₂	⁴⁵ Sc	0.007	0.07
⁵² Cr	H ₂	⁴⁵ Sc	0.02	0.2
⁵⁵ Mn	He	⁴⁵ Sc	0.07	0.7
⁵⁶ Fe	H ₂	⁴⁵ Sc	0.09	0.9
⁵⁹ Co	He	⁴⁵ Sc	0.01	0.1
⁶⁰ Ni	He	⁴⁵ Sc	0.1	1
⁶³ Cu	He	⁴⁵ Sc	0.03	0.3
⁶⁶ Zn	He	⁴⁵ Sc	0.2	2
⁷⁵ As	He	⁸⁹ Y	0.03	0.2
⁷⁸ Se	H ₂	⁸⁹ Y	0.04	0.4
⁹⁵ Mo	He	⁸⁹ Y	0.007	0.07
¹¹¹ Cd	He	¹¹⁵ In	0.02	0.2
¹¹⁸ Sn	He	¹¹⁵ In	0.02	0.2
¹²¹ Sb	He	¹¹⁵ In	0.01	0.1
²⁰² Hg	He	²⁰⁹ Bi	0.006	0.06
²⁰⁸ Pb	He	²⁰⁹ Bi	0.02	0.2

IDL (Instrument Detection Limit): 3σ (standard deviation of STD0) \times slope of calibration curve

DL in edible oil: IDL \times dilution factor (10 times)

*: Analysis of Ca with a mass number of 40 is permitted exclusively in LabSolutions™ ICPMS Ver2.10 and later.

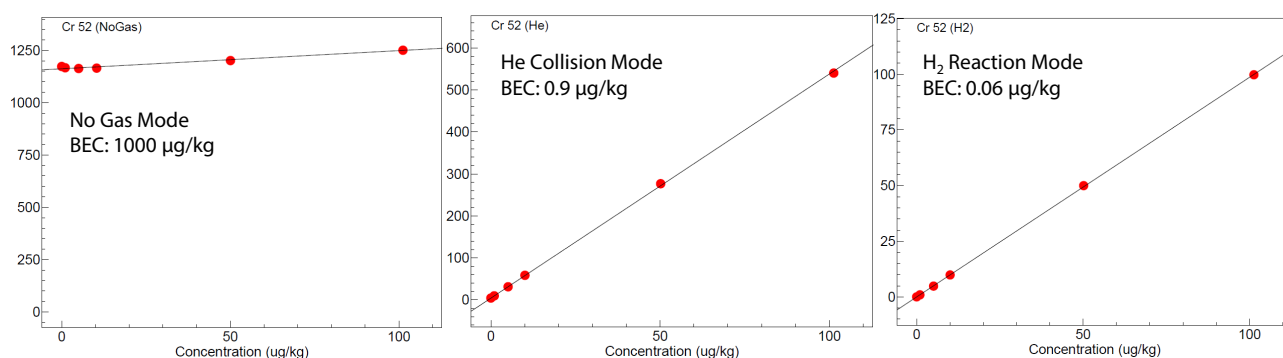


Fig. 2 Calibration Curve of Cr under Each Cell Condition (vertical axis: intensity ratio; horizontal axis: concentration)

■ Quantitative Analysis and Spike Recoveries

The trace metal elements in edible oils were quantitatively analyzed using the calibration standards shown in Table 1. The quantitative results were converted to the concentration in edible oils by multiplying the dilution factors (vegetable oil: 9.72 times, rice oil: 9.78 times, olive oil: 9.98 times). Additionally, to confirm the accuracy of the measurement, spike recoveries were evaluated. The results of the quantitative analysis and the spike recoveries are shown in Table 5.

Good recoveries between 87 % and 112 % were obtained, demonstrating that trace metal elements in edible oils can be accurately analyzed using simple dilution with organic solvents.

■ Conclusion

In this Application News, the analysis of metal elements in edible oils was performed using the ICPMS-2050 with the organic solvent injection system. Good spike recoveries were achieved, confirming that trace metal elements in edible oils can be accurately quantified with a simple sample preparation involving only dilution with organic solvents. Since it is possible to analyze without the complex procedures required for acid decomposition, the risk of volatilization or contamination of the analytes during the sample preparation process is reduced. Additionally, the use of solvent-resistant peristaltic pump tubing enables the in-line addition of internal standard elements, further reducing the time required for sample preparation.

The analytical conditions can be easily registered from preset methods, allowing for analysis without the need to consider complex analytical conditions for organic solvents.

<References>

- 1) Codex general standard for contaminants and toxins in food and feed (CODEX STAN 193-1995)

Table 5 Quantitative Results in Edible Oil and Spike Recoveries

Elements	IDL (μg/kg)	Spike Conc. (μg/kg)	Vegetable Oil			Rice Oil			Olive Oil		
			Unspiked Sample (μg/kg)	Results in Edible Oil (μg/kg)	Spike Recoveries (%)	Unspiked Sample (μg/kg)	Results in Edible Oil (μg/kg)	Spike Recoveries (%)	Unspiked Sample (μg/kg)	Results in Edible Oil (μg/kg)	Spike Recoveries (%)
⁷ Li	0.06	10	0.36	3.5	96	0.27	2.6	95	0.37	3.7	91
²³ Na	2	10	N.D.	N.D.	100	N.D.	N.D.	90	N.D.	N.D.	90
²⁴ Mg	0.4	10	17.6	171	96	N.D.	N.D.	95	9.2	92	92
²⁷ Al	0.7	10	N.D.	N.D.	92	N.D.	N.D.	102	2.3	23	99
²⁸ Si	3	10	5	50	90	6	60	100	12	120	90
³¹ P	100	1000	N.D.	N.D.	110	N.D.	N.D.	97	100	1000	110
³⁹ K	0.7	10	2.9	28	94	N.D.	N.D.	89	25.9	258	93
⁴⁰ Ca*	0.2	10	57.1	555	(99)	0.3	3	96	68.6	685	(106)
⁴⁷ Ti	0.5	10	N.D.	N.D.	105	0.6	6	91	N.D.	N.D.	98
⁵¹ V	0.007	10	0.156	1.52	102	0.087	0.85	100	0.019	0.19	100
⁵² Cr	0.02	10	0.87	8.5	93	N.D.	N.D.	101	N.D.	N.D.	99
⁵⁵ Mn	0.07	10	0.37	3.6	99	N.D.	N.D.	93	2.05	20.5	98
⁵⁶ Fe	0.09	10	0.66	6.4	100	0.21	2.1	99	40.9	408	(112)
⁵⁹ Co	0.01	10	0.09	0.9	99	0.04	0.4	95	0.03	0.3	93
⁶⁰ Ni	0.1	10	0.2	2	96	N.D.	N.D.	97	0.1	1	95
⁶³ Cu	0.03	10	0.12	1.2	99	0.99	9.7	93	0.18	1.8	90
⁶⁶ Zn	0.2	10	0.98	9.5	99	0.55	5.4	87	4.72	47.1	94
⁷⁵ As	0.03	10	0.84	8.2	106	0.97	9.5	100	0.05	0.5	99
⁷⁸ Se	0.04	10	0.26	2.5	102	0.43	4.2	106	N.D.	N.D.	104
⁹⁵ Mo	0.007	10	0.108	1.05	102	0.055	0.54	98	0.013	0.13	95
¹¹¹ Cd	0.02	10	0.08	0.8	104	0.03	0.3	97	0.02	0.2	97
¹¹⁸ Sn	0.02	10	0.26	2.5	107	0.16	1.6	103	0.04	0.4	94
¹²¹ Sb	0.01	10	0.40	3.9	108	0.18	1.8	104	0.12	1.2	101
²⁰² Hg	0.006	1	0.025	0.24	101	0.011	0.11	110	0.012	0.12	96
²⁰⁸ Pb	0.02	10	0.957	9.30	104	0.307	3.00	96	0.181	1.81	93

N.D.: Not Detected

Spike Recovery (%): (spiked sample – unspiked sample) / spike concentration × 100

(): Reference value (because spike concentration is much lower than the result in the unspiked sample)

*: Analysis of Ca with a mass number of 40 is permitted exclusively in LabSolutions ICPMS Ver2.10 and later.

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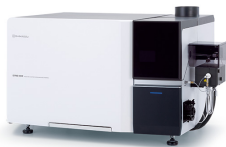
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