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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 Feed[1] (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.

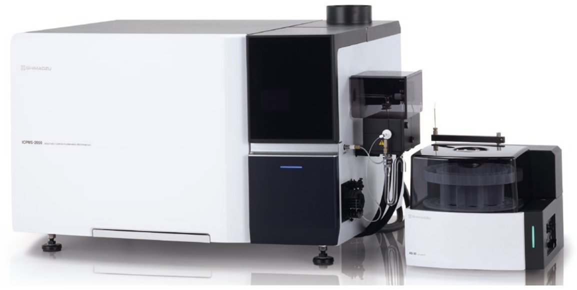

Fig. 1 ICPMS-2050 and AS-20

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.

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 $^{12}C^{12}C^{+}$ on ^{24}Mg, $^{12}C^{16}O^{+}$ on ^{28}Si, and $^{40}Ar^{12}C^{+}$ on ^{52}Cr. 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 $^{40}Ar^{12}C^{+}$ results in a high background equivalent concentration (BEC), making it challenging to analyze trace amounts of ^{52}Cr. However, in collision mode and reaction mode, interferences can be eliminated, reducing the BEC and enabling the analysis of trace amounts of ^{52}Cr.

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 µg/kg (0.1 mg/kg).

Table 4 Detection Limits (DLs)

Elements	Cell Gas	Internal Standards	IDL (µg/kg)	DL in edible oil (µg/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
^{44}Ca	H_2	^{45}Sc	2	20
^{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) × slope of calibration curve
DL in edible oil: IDL × dilution factor (10 times)

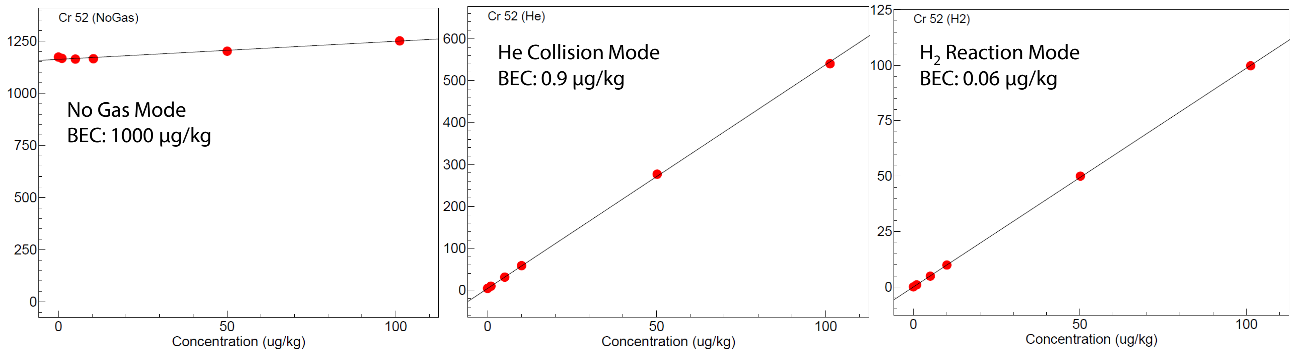

Fig. 2 Calibration Curve of Cr under Each Cell Condition (vertical axis: intensity ratio; horizontal axis: concentration)

■ 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 120 % 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.

<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
^{44}Ca	2	10	55.8	542	(103)	N.D.	N.D.	100	67.6	675	(120)
^{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)

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