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Application News

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Atomic Absorption Spectrophotometer AA-7000F / AAC / GFA-7000A / ASC-7000

Measurement of Trace Heavy Metals in Food with Nutrient Function Claims by Microwave Digestion-Furnace Method

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User Benefits

- Easy measurement of trace heavy metals in "foods with nutrient function claims" (FNFCs) is possible by atomic absorption spectrophotometer (AA).
- Sample preparation can be conducted quickly with minimal environmental pollution by using microwave digestion.
- Data can be acquired with high sensitivity at the ppb order in measurements by the furnace method.

■ Introduction

Foods with nutrient function claims (FNFCs) are food products which are used to supplement designated nutritional components and display the functions of the nutritional components concerned. Many people use FNFCs to maintain health or to supplement nutritional components. The components which can be shown in product labeling and their content ranges are regulated, and include five types of minerals (zinc, calcium, iron, copper, magnesium) and 12 types of vitamins. In many cases, substances extracted from natural products and food additives are used as raw materials for manufacturing FNFCs.

However, there is a possibility of contamination by toxic heavy metals contained in the raw materials or in the manufacturing process. Japan has set standard values for heavy metals in unpolished rice, drinking water, and food additives, and international organizations and various foreign countries have also set standard values for heavy metals in many products.

This article introduces an example in which a commercially-available vitamin product was decomposed by the microwave digestion method, and the heavy metals lead (Pb), arsenic (As), and cadmium (Cd) were measured by the furnace method.

The Microwave digestion method has various advantages, including a high decomposition capacity which enables fast decomposition treatment, small consumption of reagents, and minimal loss of volatile metals elements.

The furnace method enables highly sensitive measurement of numerous elements in small samples.

■ Sample Preparation

First, 0.5 g of the vitamin product was weighed and transferred to a microwave digestion container which had been washed with dilute nitric acid in advance, 6 mL of nitric acid and 2 mL of hydrogen peroxide were added, and the container was sealed, after which microwave digestion was carried out under the conditions [1] in Table 1.

Table 1 Microwave Digestion Conditions

Step	Time (min)	Temperature (˚C)	Output (W)
1	0	0	0
2	2	70	500
3	5	50	0
4	20	200	500
5	30	200	500

After cooling, the digestion solution was transferred to a beaker and heated with a hot plate until immediately before exsiccation. The dried and hardened sample was then dissolved by adding 0.25 mL of nitric acid and a small amount of water, and diluted in a 50 mL measuring flask by adding pure water up to the marked line.

■ Instrument Configuration and Measurement Conditions

The instrument used here was a Shimadzu AA-7000F/AAC atomic absorption spectrophotometer. A graphite furnace atomizer and autosampler were used in the furnace method.

The furnace method enables higher analytical sensitivity in comparison with the flame method.

The measurement was carried out by the calibration curve method. Table 2 shows the measurement conditions and atomization conditions of the instrument used.

Table 2 Measurement Conditions

Element	Pb	As	Cd
Analysis wavelength	283.3 nm	193.7 nm	228.8 nm
Slit width	0.7 nm	0.7 nm	0.7 nm
Lighting mode	BGC-D2	BGC-D2	BGC-D2
Lamp current	10 mA	12 mA	8 mA
Incineration temperature	700 ˚C	900 ˚C	500 ˚C
Atomization temperature	2000 ˚C	2100 ˚C	2000 ˚C
Tube type	Pyro-coated graphite tube	Platform tube	Pyro-coated graphite tube
Pd 50 ppm addition	None	2 µL	2 µL
Sample injection volume	10 µL		
Number of repeated measurements	2 times (maximum 3 times)		

■ Measurement Results

Table 3 (a) to (c) show the measurement results for the standard solutions of Pb, As, and Cd, respectively. Figs. 1 to 3 show the corresponding calibration curves. A satisfactory correlation (r = 0.999 or higher) was obtained for all calibration curves.

Table 4 shows the measurement results. None of the target elements Pb, As, and Cd was detected from the samples. The limit of quantitation (LOQ) was expressed by the value of 10 σ calculated from the standard deviation (SD) obtained by 10 repeated measurements of a blank sample. The LOQs of Pb, As, and Cd in the solution were 0.3 ppb, 3 ppb, and 0.05 ppb, respectively.

Table 3(a) Measurement Results of Pb Standard Solution

Set concentration (ppb)	Absorption (Abs)	%RSD	SD
0	0.0010	22.33	0.0002
4	0.0334	0.00	0.0000
10	0.0796	2.02	0.0016
20	0.1432	1.04	0.0015

Table 3(b) Measurement Results of As Standard Solution

Set concentration (ppb)	Absorption (Abs)	%RSD	SD
0	0.0064	18.93	0.0012
4	0.0376	6.21	0.0023
10	0.0798	0.44	0.0004
16	0.1207	1.64	0.0020
20	0.1506	3.24	0.0049

Table 3(c) Measurement Results of Cd Standard Solution

Set concentration (ppb)	Absorption (Abs)	%RSD	SD
0	0.0036	5.81	0.0002
0.4	0.0950	0.22	0.0002
1	0.2046	0.73	0.0015
2	0.4028	0.83	0.0033

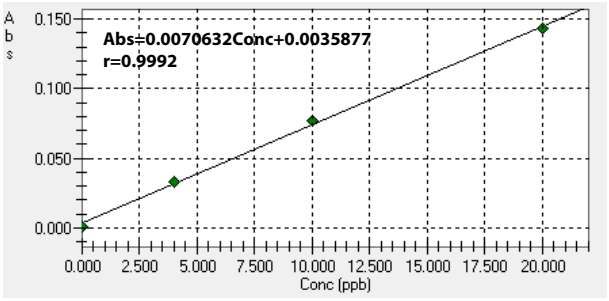

Fig. 1 Calibration Curve of Pb

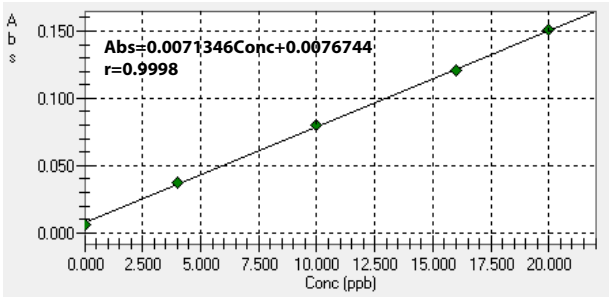

Fig. 2 Calibration Curve of As

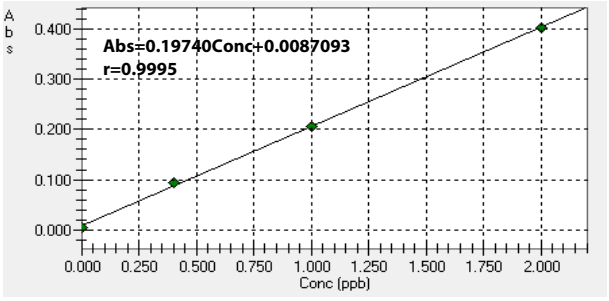

Fig. 3 Calibration Curve of Cd

Table 4 Measurement Results of Sample

Element	Absorption (Abs)	SD	%RSD	Actual concentration (mg/kg)
Pb	0.0020	0.0001	12.86	<0.03
As	0.0108	0.0023	21.51	<0.3
Cd	0.0032	0.0002	6.53	<0.005

Table 5 shows the recovery rates of the samples. In the spike-and-recovery test, each element was spiked with a standard solution of a set concentration, and the concentration was measured. Good results were obtained for all elements, as the recovery rates were within 100 ±10 %.

Fig. 4 shows the excerpts of the peak profiles of the measurements.

Table 5 Recovery Rates of Samples

Element	Spike amount (ppb)	Measurement value (ppb)	Recovery rate (%)
Pb	4	3.88	97 %
As	10	9.58	96 %
Cd	1	1.08	108 %

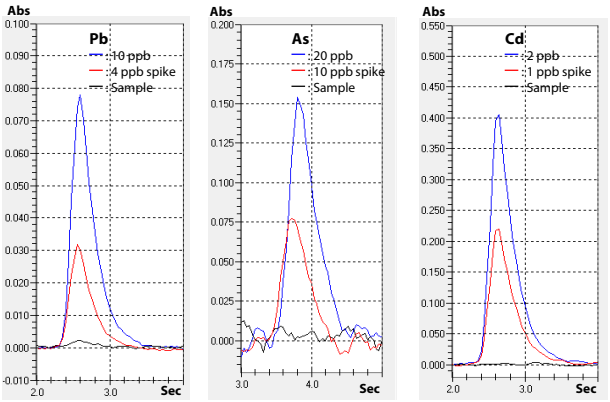

Fig. 4 Excerpts of Peak Profiles (BG Omitted)

■ Conclusion

In a measurement of trace amount of heavy metals in a Food with Nutrient Function Claims (vitamin product) by atomic absorption spectrophotometry (AA), treatment in a short time in comparison with the conventional wet digestion and dry digestion methods was possible by using the microwave digestion method in sample preparation. Among other advantages of the microwave digestion method, consumption of reagents is small, and volatilization of metallic elements is minimal.

In measurement of As, measurement with good repeatability was possible by using a platform tube. In addition, data could be acquired with high sensitivity by measuring As and Cd by the furnace method using palladium nitrate as a matrix modifier.

<Reference>

(1) Standard Tables of Food Composition in Japan – 2015 – (Seventh Revised Edition), Analysis Manual

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