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Application News

No.X246A

X-ray Analysis

EDXRF Analysis of Arsenic and Lead in Dietary Supplement

In recent years, dietary supplements have become widely available in convenience stores and supermarkets. They are defined as food products that promote and maintain health and are used to improve disease prevention and enhance immunity. They are available in various types and forms, including tablet and powdered supplements, and processed herbal products, etc. Among these are products that are subject to safety standards that address the presence and concentrations of heavy metals, etc.[1)]

Analysis of toxic heavy metals such as As and Pb is typically conducted using an emission spectrophotometer (ICP) or atomic absorption spectrophotometer (AA), however, these require time-consuming preparation procedures. For analyte quantities ranging from a few to tens of ppm, measurement can be conducted using an X-ray fluorescence spectrometer, which permits very easy sample preparation.

Using an energy dispersive X-ray fluorescence spectrometer, we conducted quantitative analysis of As and Pb in a dietary supplement (herbal medicine), and evaluated their lower limit of detection and quantitation, respectively.

1) Example: Japan Health and Nutrition Food Association (JHNFA)

Standard Samples

Seven standard samples were prepared by mixing herbal powder with a standard solution used for atomic absorption analysis. The elements and standard values are shown in Table 1, and the preparation procedure is shown in Fig. 1.

Table 1 Standard Values

No.	As	Pb
(1)	50	0
(2)	30	5
(3)	20	10
(4)	10	20
(5)	5	30
(6)	0	50
(7)	0	0

Unit: ppm

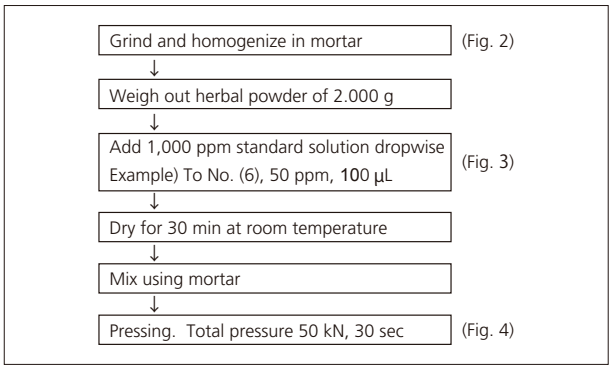

Fig. 1 Preparation Procedure

Fig. 2 Homogenization by Pulverizing

Fig. 3 Blend in Standard Solution

Fig. 4 Formed Briquette

Calibration Curves

The calibration curves for As (Kα line) and Pb (Lβ line) are shown in Fig. 5 and 6, respectively. Correction by the dj method was conducted for As, which is overlapped by Pb. We also generated those calibration curves with the internal standard which line is the RhKα C scattering (Compton) (figure not shown). Table 2 shows the accuracy of the respective calibration curves with and without internal standard correction. Accuracy refers to the variation of the calibration point using a numerical value indicated as 1 σ.

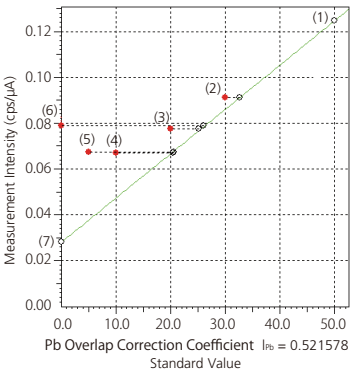

Fig. 5 Calibration Curve for As

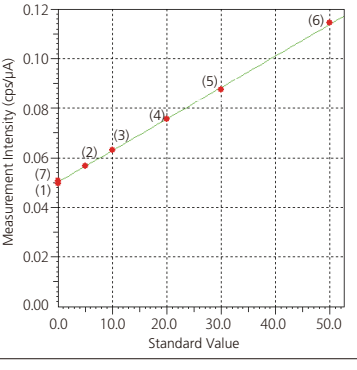

Fig. 6 Calibration Curve for Pb

Table 2 Accuracy of Calibration Curve

Internal Standard Correction	Without		With	
Internal Standard Line	–		RhKαC	
Element	As	Pb	As	Pb
Analytical line	Kα	Lβ1	Kα	Lβ1
Accuracy (1 σ)	0.23	0.42	0.60	0.83

Unit: ppm

Profile

Fig. 7 shows the profile overlap of standard sample No. (4) (As: 10 ppm, Pb: 20 ppm) and No. (7) (Blank). Since the AsKα line and PbLα line are adjacent, one or a combination of the following processing methods is selected.

A) Intensity peak separation
B) Intensity overlap correction
C) Overlap correction of the dj method on Calibration curve

Here, we applied method C) only.

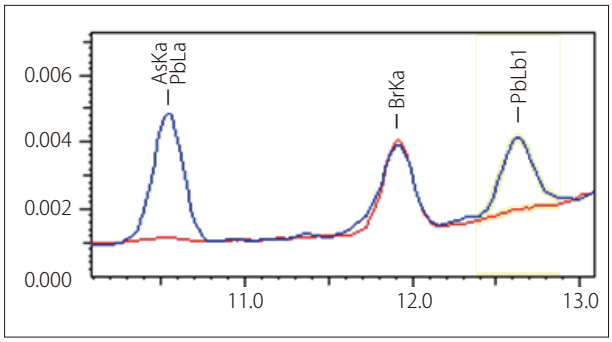

Fig. 7 Qualitative Profile Overlap of As and Pb
Blue: No. (4); Red: No. (7) (Blank)

Lower Limits of Detection and Quantitation

Ten times repeatability test of the No. (7) Blank were conducted, and the lower limit of detection (3σ) and lower limit of quantitation (10σ) were determined.

Table 3 shows the results obtained using 2 different sample preparation methods, pressing and simple compression. In the case of simple manual compression, quantitation was conducted using an internal standard calibration curve to correct for flatness and density effects. The powder preparation procedure used is shown in Fig. 8.

Table 3 Lower Limits of Detection and Quantitation for As and Pb

Preparation Method	Pressing		Powder, Sample Container	
Internal Standard Correction	Without		With	
Element	As	Pb	As	Pb
Average Value	(-0.08)	0.35	0.32	(-0.18)
Standard Deviation	0.18	0.26	0.18	0.26
Lower Limit of Detection (3σ)	0.53	0.77	0.55	0.78
Lower Limit of Quantitation (10σ)	1.8	2.6	1.8	2.6

Unit: ppm

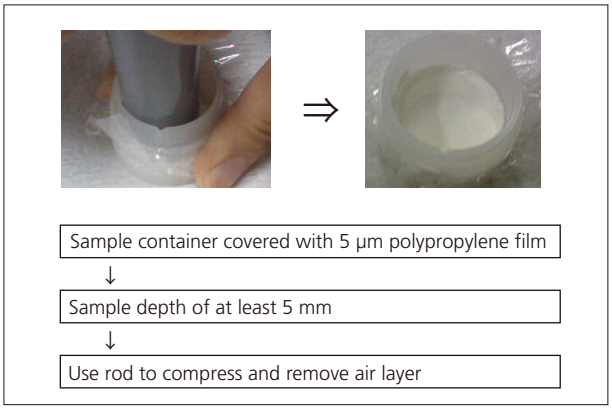

Fig. 8 Simple Powder Compression by Hand

Conclusion

Table 2, which shows the accuracy of the calibration curves, indicates that without conducting internal standard correction, accuracy improved 2.6 times for As, and 2.0 times for Pb. The cause is thought that the fluctuation of the RhKαC is added to the fluctuations in the respective intensities of AsKα and PbLβ1 .

On the other hand, both for As and Pb, Table 3 indicates that the lower limit of detection and lower limit of quantitation are the same for both formed briquette samples and manually compressed samples. The reason for this lack of any substantial difference is thought to be due to a zero net intensity for the blank.

Therefore, since either method is valid for measuring dietary supplements, using the pressing sample preparation method without the use of an internal standard or the easy compression (powder) sample preparation method with an internal standard is suitable.

Analytical Conditions

Table 4 Analytical Conditions

Instrument	: EDX-720 (EDX-GP)
X-Ray Tube	: Rh target
Tube Voltage	: 50 kV
Tube Current	: (Auto sensitivity control) µA
Filter	: #3 (EDX-720) #4 (EDX-GP)
Atmosphere	: Air
Measurement Diameter	: 10 mm
Measurement Time	: 1200 sec
Dead Time	: Max 40 %

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