SHIMADZU

Application News

Spectrophotometric Analysis No.A415A

Determination of Lead in Sugar by Electrothermal Atomization

■ Introduction

The Japanese pharmacopeia specifies that analysis of lead content in refined white sugar be conducted by the electrothermal atomization (furnace) method. Since high sensitivity sample analysis is possible even at micro levels, this method is effective for trace level analysis even for toxic elements other than lead.
Here we introduce an example of such an analysis based on the method specified in the Japanese pharmacopeia.

■ Pretreatment

The sample used for the analysis consisted of commercially available granulated sugar. Since atomic absorption spectrometry requires that the sample be in solution in order to conduct measurement, pretreatment of the refined sugar is necessary. Here we used a high pressure acid digestion vessel as indicated in the pretreatment procedure prescribed in the pharmacopeia. The high pressure acid digestion vessel consists of an internal PTFE vessel and an external metal or ceramic high pressure vessel. In the actual preparation, 0.050 g of sample was first accurately weighed into the internal PTFE vessel. To this, 0.5 mL of nitric acid was added for measurement of the toxic metals. The vessel was then placed in the external high pressure vessel, and this was heated for 5 hours in a 150 ˚C thermostatic chamber. After cooling, purified water was accurately added to bring the volume to 5 mL, and this was used as the measurement solution.
In addition, a blank solution was prepared by adding purified water to 0.5 mL of the above-mentioned nitric acid to bring the volume to 5 mL.

■ Analytical Method and Conditions

Quantitation was conducted by the standard addition method. The injection volumes of the prepared measurement solution, dilution-blank solution and Pb standard solution (20 ppb) were adjusted respectively using the autosampler. Tables 1 – 3 indicate the instrument used for the analyses and the main analytical conditions.

Table 1 Instrument and Optical Parameters

Instrument	Atomic absorption spectrophotometer unit: AA-7000 Atomizer unit: GFA-7000
Analysis wavelength	283.3 nm
Slit width	0.7 nm
Current	10 mA
Lamp mode	BGC-D2

Table 2 Furnace Program

	Temperature (˚C)	Time (s)	Mode	Sensitivity	Gas Flow Rate (L/min)
1	60	3	RAMP	REGULAR	0.10
2	120	20	RAMP	REGULAR	0.10
3	250	10	RAMP	REGULAR	0.10
4	600	10	RAMP	REGULAR	1.00
5	600	10	STEP	REGULAR	1.00
6	600	3	STEP	HIGH	0.00
7*	2200	3	STEP	HIGH	0.00
8	2500	2	STEP	REGULAR	1.00

7* Atomization stage
Graphite tube: Pyrolysis tube

Table 3 Autosampler Standard Addition Parameter Settings

Addition Concentration	Sample	Diluent	Pb: 10 ppb	Total
0 ppb	14 µL	6 µL	0 µL	20 µL
1 ppb	14 µL	4 µL	2 µL	20 µL
2 ppb	14 µL	2 µL	4 µL	20 µL
3 ppb	14 µL	0 µL	6 µL	20 µL

■ Results

The measurement results were under the quantitation limit. Fig. 1 shows an overlay of some typical peak profiles, and Fig. 2 shows the calibration curve using the blank solution. The quantitation limit in this measurement is 0.2 ppb for the concentration in aqueous solution, which converts to 0.03 ppm in the solid sample. This easily satisfies the criterion value of 0.5 ppm.

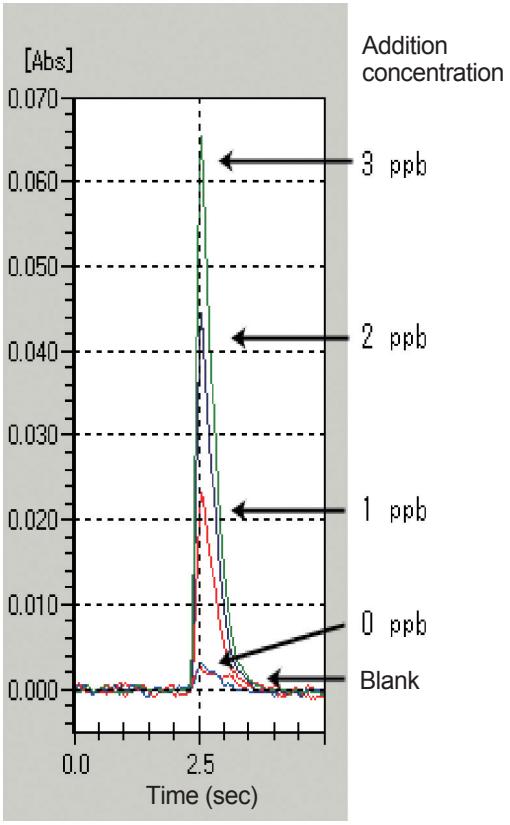

Fig. 1 Peak Profiles (partial)

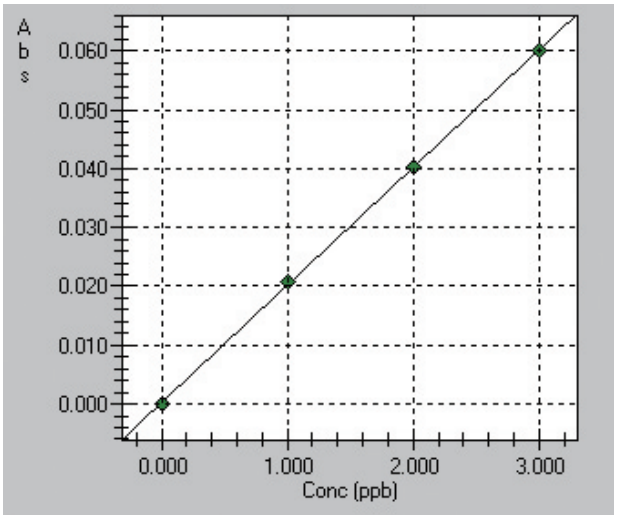

Fig. 2 Calibration Curve of Blank Solution

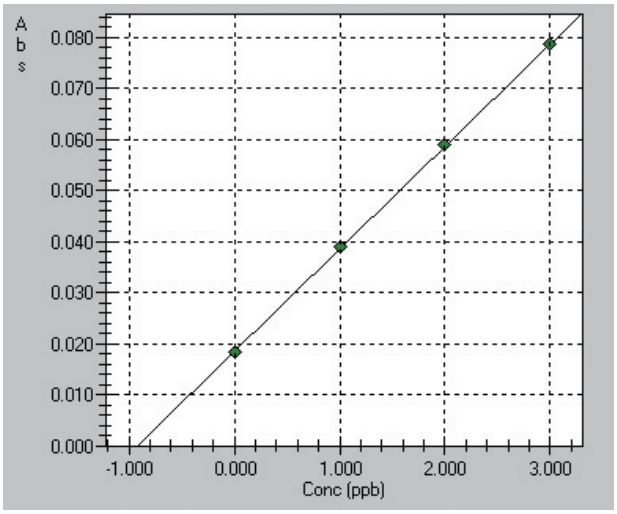

Fig. 3 Calibration Curve Using Sample Addition

In addition, Pb was added to the sample solution to bring the Pb concentration of the solution to 1.3 ppb (0.13 ppm in solid), and this was also measured as a sample. The results are shown in Fig. 3 and Table 4.
The value was 0.13 ppm in the solid, indicating good correlation.
The 1.43 dilution factor in Table 4 was calculated from the total injection volume of 20 µL with respect to the sample injection volume of 14 µL (20/14).

Table 4 Sample Addition Results and Concentration Computation

Sample ID	Conc. Setting (ppb)	Conc. (ppb)	Abs.	Sample Amt.	Total Volume	Dilution Factor	Coefficient	Actual Conc.	Actual Conc. Unit	%RSD	SD
Blank			0.0023							2.47	0.0001
Added Conc: 0 ppb	0.0000		0.0184							3.19	0.0006
Added Conc: 1 ppb	1.0000		0.0389							1.12	0.0004
Added Conc: 2 ppb	2.0000		0.0589							0.87	0.0005
Added Conc: 3 ppb	3.0000		0.0787							1.03	0.0008
Added Sample		0.9253		0.0500	5.00	1.43	0.001	0.13	ppm		

NOTES:
*This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.

SHIMADZU

SHIMADZU CORPORATION. International Marketing Division
3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax. 81(3)3219-5710
Cable Add.:SHIMADZU TOKYO

Printed in Japan 3295-12902-10A-IK