SHIMADZU

Application News

Measurement of Minerals in Dietary Supplements by Atomic Absorption

■ Introduction

Recently, the development and sales of a variety of dietary supplements have increased dramatically against the background of rising public interest regarding health. Here we introduce the method of analysis of minerals in dietary supplements as specified in the Pharmacopoeia of the United States (USP 32), where the supplement market now stands at about three trillion yen (33 billion dollars).

As one example, in the case of tablets of oil -and water- soluble vitamins with minerals, the sample preparation and measurement methods are specified for the quantitation of the minerals Ca, Cr, Cu, Fe, K, Mg, Mn, Mo, Se, and Zn, in which flame atomic absorption spectroscopy is used for conducting the quantitation.

■ Sample Preparation

The sample preparation differs for (1) Ca, Cr, Cu, Fe, K, Mg, Mn, Zn and (2) Mo, Se in the above supplement. For the elements in group (1), at least 20 tablets are crushed and a quantity corresponding to 5 tablets are transferred to a porcelain crucible. After ashing at 550 ˚C in a muffle furnace, hydrochloric acid is added and the contents are heated to dissolve the residue. Adjust the final solution to 0.125 N hydrochloric acid. For the elements in group (2), at least 20 tablets are crushed, and a quantity corresponding to 1000 μg of the measurement element is weighed. This is decomposed using nitric acid and perchloric acid, and is finally brought to a fixed 2 % solution of ammonium chloride.

■ Standard Concentrations of Elements

According to the USP, calibration curves are to be generated using standard solutions having the concentrations shown in Table 2, and quantitation is conducted using a calibration curve approximated by a straight line using a standard solution prepared for the concentration indicated in bold type in the Table. Examples of the target element calibration curves are shown in Fig. 1 – 10, but 0 μg/mL is not included in accordance with USP. In the case of Zn, since the high concentration of the standard solution causes curvature of the calibration curve at normal sensitivity, the angle of the burner was changed and measurement was conducted at lower sensitivity to improve the linearity.

■ Measurement Conditions

The measurement wavelength, type of flame, and matrix modifier used are shown in Table 1. The N_2O-C_2H_2 flame was used for measurement of Ca and Mo, and the air-C_2H_2 flame was used for all the other elements. La was added as a matrix modifier for measurement of Ca and Mg, and ammonium chloride was added for measurement of Mo and Se.

Table 1 Measurement Conditions

Element	Wavelength	Flame	Matrix Modifier
Ca	422.7 nm	N_2O-C_2H_2	0.1 % La
Cr	357.9 nm	Air-C_2H_2	
Cu	324.7 nm	Air-C_2H_2	
Fe	248.3 nm	Air-C_2H_2	
K	766.5 nm	Air-C_2H_2	
Mg	285.2 nm	Air-C_2H_2	0.1 % La
Mn	279.5 nm	Air-C_2H_2	
Mo	313.0 nm	N_2O-C_2H_2	2 % Ammonium Chloride
Se	196.0 nm	Air-C_2H_2	2 % Ammonium Chloride
Zn	213.8 nm	Air-C_2H_2	

Table 2 Element Concentrations for Calibration Curves

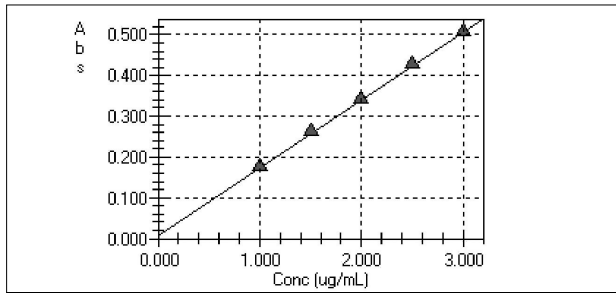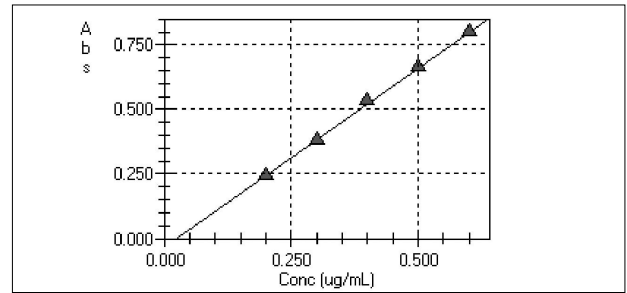| Element	STD (μg/mL)						
	1	2	3	4	5	6	8
Mn		0.5	0.75	**1**		1.5	2
K	0.5	**1**	1.5	2	2.5		
Zn	0.5	1	1.5	**2**	2.5		
Cu	0.5	1		**2**		3	4
Cr		**1**		2		3	4
Fe		2		4	**5**	6	8
Mg		0.2	0.3	**0.4**	0.5	0.6	
Ca		1	1.5	**2**	2.5	3	
Mo	5	**10**			25		
Se	5	**10**			25		

Fig. 1 Calibration Curve of Ca

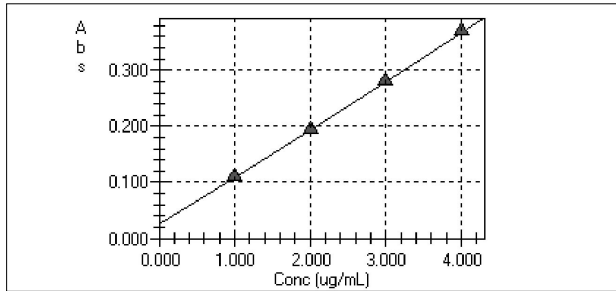**Fig. 6 Calibration Curve of Mg**

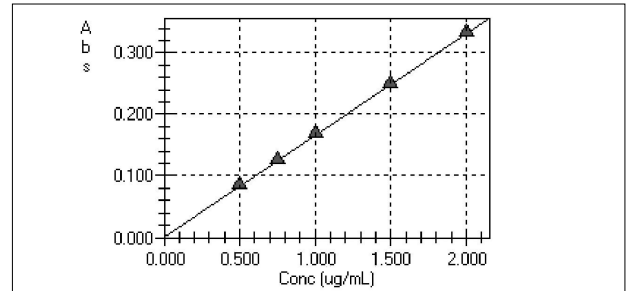**Fig. 2 Calibration Curve of Cr**
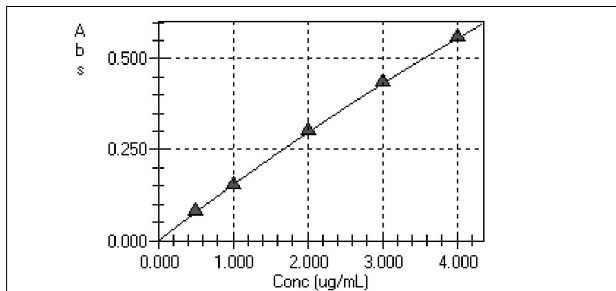

Fig. 7 Calibration Curve of Mn
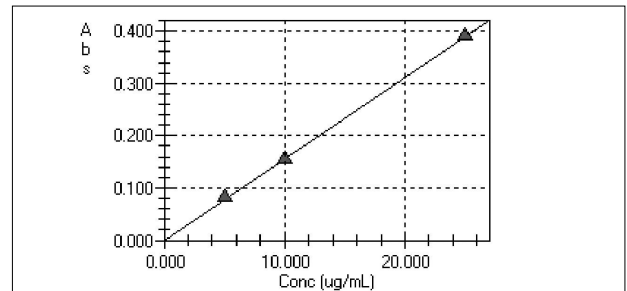

Fig. 3 Calibration Curve of Cu

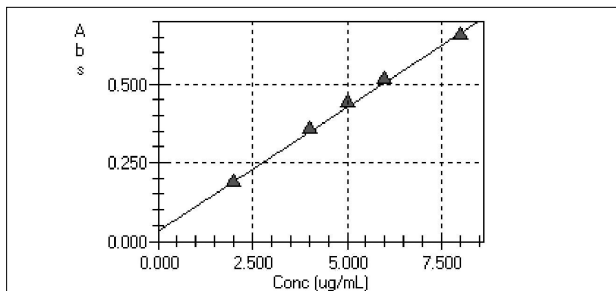**Fig. 8 Calibration Curve of Mo**

Fig. 4 Calibration Curve of Fe
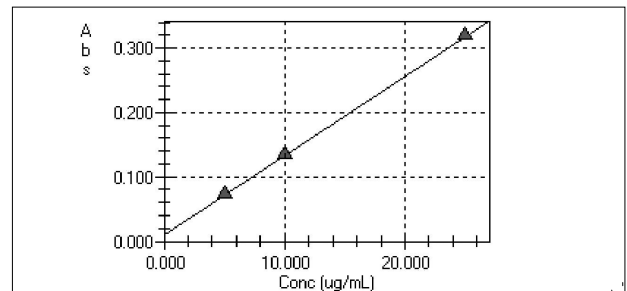
Fig. 9 Calibration Curve of Se
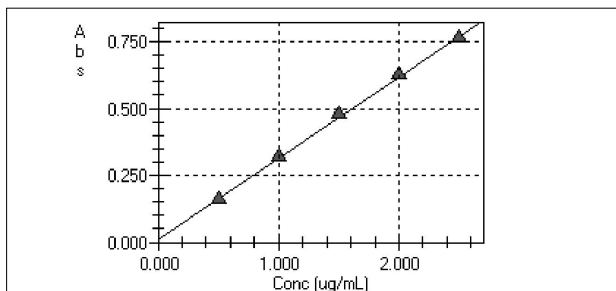
Fig. 5 Calibration Curve of K
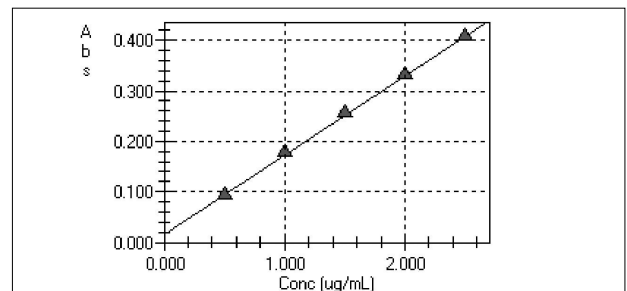
Fig. 10 Calibration Curve of Zn