APPLICATION NOTE

Atomic Absorption

Authors:

Lee Davidowski, Ph.D.

Lorraine Foglio

PerkinElmer, Inc.
Shelton, CT 06484 USA

The Determination of Minerals and Metals in Multi-Mineral/Multi-Vitamin Tablets by Flame Atomic Absorption Spectroscopy

Introduction

There are many mineral dietary supplements available in today's marketplace to ensure that mineral deficiencies do not occur in one's diet. The mineral content of these products must be verified for quality control (QC) purposes. Furthermore, the Nutritional Labeling and Education Act of 1990 mandates accurate labeling of all food supplements sold in the U.S. which means accurate testing of the products is mandatory. In many labs, this task is accomplished by the technique of flame atomic absorption spectroscopy (FAAS). FAAS has the advantages of lower initial cost, low cost per analysis, and requires less operator training than many other trace elemental techniques. The objective of this work is to demonstrate the applicability of FAAS using the PerkinElmer® PinAAcle™ 900T to accomplish this task. Seven elements are determined in two commercially available multi-mineral tablets, a NIST® Standard Reference Material, and a commercial reference material which simulates a mixed food diet.

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Experimental

Instrumentation

The PinAAcle 900T flame and longitudinal Zeeman furnace atomic absorption spectrometer controlled by WinLab32™ for AA software, running under Microsoft® Windows® 7, was used for all analyses (PerkinElmer, Inc., Shelton, CT). A high-sensitivity nebulizer (Part No. N3160112) with a spacer was employed. Single-element Lumina™ hollow cathode lamps (HCLs) were used and the instrumental operating conditions for each element in this application are shown in Table 1. A four-second integration time and three replicates were used for all elements.

Figure 1. PinAAcle 900T atomic absorption spectrometer.

Sample and Standard Preparation

One NIST® SRM 3280 Multi-vitamin/Multi-mineral Tablet and two over the counter multi-vitamin/multi-mineral tablets were coarsely ground. Nominally, 0.75 g were weighed out and rinsed into a 1000 mL volumetric flask containing about 20 mL of ASTM® Type I deionized water (>16MΩ • cm). Four mL of HCl (trace metal grade), two mL of HNO_3 (trace metal grade), and a magnetic stirring bar were then added to the flask. This sample digestion solution was stirred for approximately one hour and warmed, not to boiling, on a hot plate. Deionized water was then added to the volumetric mark after removal of the magnetic stir bar. The sample digestion solution was then filtered through Whatman® 46 paper (passes < 8 micron) to remove waxy undissolved material, presumably from the tablet's coating. The filtrate was then analyzed, after appropriate dilution, against single-element aqueous standards (PerkinElmer Pure) for elemental quantitation. A Certified Reference Material Mixed Food Diet (CRM-MFD) (High Purity Standards, Charleston, SC) was prepared by dissolution, following the manufacturer's instructions, to an appropriate concentration for individual elements.

All tablets also contained about 2 mg/tablet of silicon, much of which may have remained undissolved. For an accurate measurement of the silicon, HF (trace metal grade) and plastic volumetric flasks should be employed for the dissolution.

For calcium and magnesium determinations, $La(NO_3)_2$ (reagent grade) was added to all standards, blank and samples to a concentration level of 0.2% to remove chemical inferences from other matrix elements. For potassium determinations, CsCl (reagent grade) was added to all test solutions to a concentration level of 0.2% as an ionization buffer.

Results

All elements showed acceptable calibration criteria. The results obtained for the FAAS analyses of the multi-vitamin/multi-mineral tablets are shown in Table 2. The values are corrected for laboratory sample dissolution steps and dilutions. The commercial tablets were calculated and reported in mg/tablet as this was the unit shown and guaranteed on each label. All elements were found to be within the certified range for the NIST® 3280 tablet. Both commercial tablets showed good correlation to labeled quantity guaranteed by the manufacturer.

Table 1. **PinAAcle 900T Instrumental Parameters.**

	Element						
Parameter	**Ca**	**Mg**	**K**	**Fe**	**Zn**	**Cu**	**Mn**
Wavelength (nm)	422.7	285.2	766.5	248.3	213.9	324.8	279.5
Slit Width (nm)	0.7	0.7	0.7	0.2	0.7	0.7	0.2
Oxidant Flow (L/min)	10	10	10	10	10	8.7	10
C_2H_2 Flow (L/min)	2.5	2.5	2.5	2.2	2.5	2.0	2.5
Working Range (mg/L)	5	1	4	4	1	4	2
HCL Part Nos.	N3050114	N3050144	N3050139	N3050126	N3050191	N3050121	N3050145
Standard Part Nos.	N9303763	N9300179	N9303779	N9303771	N9300178	N9300183	N9303783

Table 2. **Corrected FAAS results for one certified reference material and two commercial multi-vitamin/multi-mineral samples.**

	NIST® 3280 (mg/g)			Commercial Tablet 1 (mg/Tab)			Commercial Tablet 2 (mg/Tab)		
Element	**Certified**	**Found**	**SD**	**Label**	**Found**	**SD**	**Label**	**Found**	**SD**
Ca	110.7	107	0.8	162	163	0.6	200	211	0.6
Mg	67.8	69.1	0.2	100	107	0.4	100	109	0.4
K	53.1	53.8	0.3	80	83.8	0.4	80	90.7	0.4
Fe	12.35	12.9	0.05	18	19.1	0.08	n/a	0.23	0.006
Zn	10.15	10.1	0.02	15	15.5	0.04	15	15.2	0.03
Cu	1.4	1.42	0.01	2	2.19	0.01	2	2.04	0.01
Mn	1.44	1.48	0.01	2	1.91	0.01	2	2.04	0.01

The results for the FAAS analysis of the Certified Reference Material – Mixed Food Diet from High Purity Standards – are given in Table 3. All elements were found to be within 5% of the certified value, showing excellent agreement with the reference material.

Table 3. **Mixed Food Diet CRM results using aqueous standards and FAAS on a PinAAcle 900T.**

	CRM-MFD (mg/L)		
Element	**Certified**	**Found**	**SD**
Ca	40	39.8	0.2
Mg	12	11.8	0.03
K	160	159	0.005
Fe	0.8	0.802	0.005
Zn	0.3	0.290	0.001
Cu	0.06	0.061	0.001
Mn	0.2	0.191	0.003

Conclusions

The mineral content of multi-vitamin/multi-mineral tablets must be determined in order to ensure the quality of commercial dietary supplements. This method demonstrates the ability of the PinAAcle 900T flame atomic absorption system to accurately measure minerals in both commercial tablets and food simulation diets. It is an efficacious method that allows for cost effectiveness and ease of use, with less operator training than other analytical methods. The PinAAcle 900H (Flame and Deuterium Furnace) and PinAAcle 900F (Flame only) spectrometers can also be used for this application.

PerkinElmer, Inc.
940 Winter Street
Waltham, MA 02451 USA
P: (800) 762-4000 or
(+1) 203-925-4602
www.perkinelmer.com

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