



Determination of macro and trace elements in multivitamin dietary supplements by high-resolution continuum source graphite furnace atomic absorption spectrometry with slurry sampling

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ABSTRACT

In this research, three different commercially available multivitamin dietary supplements were analyzed by high-resolution continuum source graphite furnace atomic absorption spectrometry (HR-CS GFAAS) with slurry sampling. The concentrations of Cr, Cu, Fe, Mn, and Se were determined and compared to the amounts stated by producers. The safety of multivitamin dietary supplements depends on various factors including the manufacturing process and the purity and origins of the raw ingredients. For this reason, this research determined concentrations of several toxic elements (As, Cd, and Pb). Microwave-assisted high pressure Teflon bomb digestion was used to determine total amounts of elements in samples. Samples were prepared as slurries at a concentration of 0.1% (m/v) for macro elements (Cr, Cu, Fe, Mn, and Se) and at a concentration of % (m/v) for trace elements (As, Cd, and Pb) in acidic media (3 M HNO₃). The influence of acid concentration, Triton X-100 addition, sonication time, and sonication power on absorbance was investigated. The accuracy of this method was validated by analyses of NRCC LUTS-1 (Lobster hepatopancreas), NRCC DORM-1 (Dogfish Muscle), NRCC DOLT-2 (Dogfish Liver), NBS SRM 1570 (Spinach Leaves) and NBS SRM 1573 (Tomato Leaves) certified reference materials. The measured elements contents in these reference materials (except NRCC DOLT-2) were in satisfactory agreement with the certified values according to the *t*-test for a 95% confidence level.

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1. Introduction

To compensate for deficiencies of macro and trace elements in the human diet, a number of multivitamin preparations have appeared on the market. These dietary supplements are recommended for application in the treatment and prophylaxis of the deficiency of macro and trace elements. The intake of these preparations has increased significantly in recent decades. Regulation No. 178/2002 of the European Parliament and the Council hold supplement manufacturers and distributors responsible for the content of the dietary preparations [1]. These supplements should contain only what is on the label and should not contain any harmful or undesirable substances, such as toxic metals. The safety of multivitamin dietary supplements depends on various factors including the manufacturing process and the purity and origins of the raw ingredients. Several studies proved that multivitamin materials may contain high levels of certain elements, especially Pb and As [2–4].

Graphite furnace atomic absorption spectrometry (GFAAS) is one of the most frequently used analytical techniques for the determination of selected elements in multivitamin dietary supplements. Bourgoin et al. determined Cd and Pb in calcium supplements and other calcium-rich matrices by anodic stripping voltammetry, flame atomic absorption spectrometry (FAAS), GFAAS, and inductively coupled plasma mass spectrometry (ICP-MS) [5]. Kovács et al. investigated chromium content in foodstuffs and nutrition supplements by GFAAS [6]. Sołyk et al. determined Cr and selected elements (Ca, Cr, Cu, Fe, Mg, Mn, Mo, P, Se, and Zn) in multivitamin preparations and in pharmaceutical raw material using GFAAS and ICP-MS [7].

The direct analysis of solids by GFAAS or inductively coupled plasma atomic emission spectrometry (ICP-OES) offers several advantages over conventional wet oxidation and dry ashing sample preparation technique. The slurry sampling technique reduces sample preparation time, decreases the possibility of loss of analyte, and minimizes the risk of contamination [8]. This technique was used for the determination of Cu and Mn in antibiotic tablets (ciprofloxacin and cephalexin) [9]. Amin et al. proposed slurry sampling electrothermal atomic absorption spectrometry with a molybdenum tube atomizer for the determination of Mn [10] and Cu [11] in herbal medicine samples. Silveira et al. determined

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manganese in antihypertensive drugs by GFAAS with slurry sampling [12]. This technique was also used for the determination of selected elements in sewage sludge [13,14] as well as soils and sediments [15,16]. High-resolution continuum source flame atomic absorption spectrometry (HR-CS FAAS) with slurry sampling was used for the determination of Zn in yogurt [17], Fe in fortified milk powder [18], as well as Ca and Mg in dairy products [19]. Other authors used high-resolution continuum source atomic absorption spectrometry with atomization in a graphite furnace for the determination of Cd and Fe in sewage sludge [20] and Cd in fertilizer samples [21]. The slurry sampling technique was also developed and optimized for rapid multi-elemental analysis of multivitamin preparations using ICP-OES [22,23]. Trace elements (Cr, Mo, Pd, Cd, Pt, and Pb) were also determined in drug tablets by ultrasonic slurry sampling electrothermal vaporization inductively coupled plasma mass spectrometry (USS-ETV-ICP-MS) [24].

The aim of this work was to improve on the analytical potential of graphite furnace atomic absorption spectrometry by using sequential multi-element determination of macro (Cr, Cu, Fe, Mn, and Se) and trace (As, Cd, and Pb) elements in multivitamin dietary supplements in liquid and slurry samples.

2. Experimental

2.1. Spectrometer

An Analytik Jena ContraAA 700 high-resolution atomic absorption spectrometer equipped with a 300W xenon short-arc lamp (Analytik Jena, Jena, Germany) as a continuum radiation source was used throughout these experiments. This new equipment comprises a compact high-resolution double echelle monochromator and a charge-coupled device (CCD) array detector with a resolution of about 2 pm per pixel in the far-ultraviolet range. In our research, a graphite furnace was used for atomization of analytes. After the measurement, data stored in the internal memory of the processing system were transferred to the computer for evaluation. The operating parameters of the HR-CS GFAAS instrument are summarized in Table 1.

2.2. Gases and reagents

Compressed high-purity argon obtained from Air Products (Warsaw, Poland) was used as a carrier gas.

Standard solutions of As, Cd, Cu, Cr, Fe, Mn, Pb, and Se were prepared from a 1000 mg l⁻¹ atomic absorption standard solutions (Merck, Darmstadt, Germany). Working standard solutions were prepared fresh each day by diluting appropriate aliquots of the stock solution in high-purity water.

All mineral acids (65% (v/v) HNO₃ and 40% HF (v/v)) and hydrogen peroxide 30% (v/v) of the highest quality (Suprapur, Merck, Darmstadt, Germany) were used.

Triton X-100 (Fluka, Buchs, Switzerland) was used.

The following chemical modifier solutions were used: palladium modifier stock solution, 10.0 ± 0.2 g l⁻¹ Pd(NO₃)₂; magnesium modifier stock solution, 10.0 ± 0.2 g l⁻¹ Mg(NO₃)₂; palladium/magnesium modifier stock solution, 10.0 ± 0.2 g l⁻¹ Pd + Mg(NO₃)₂; and phosphate modifier stock solution, 10.0 ± 0.2 g l⁻¹ NH₄H₂PO₄. All chemical modifier solutions were obtained from Merck, Darmstadt, Germany.

High-purity water was used throughout the research: deionized water (DEMIWA 5 ROSA, Watek, Czech Republic) and doubly distilled water (quartz apparatus, Bi18, Heraeus, Hanau, Germany). The resistivity of the water was 18 MΩ cm.

2.3. Reference materials and multivitamin dietary supplements

Validation of the method described in this work was performed using five certified reference materials. The following materials were chosen: LUTS-1 (Lobster hepatopancreas), DORM-1 (Dogfish Muscle), DOLT-2 (Dogfish Liver) supplied by the National Research Council of Canada (NRCC), Ottawa, Canada and NBS SRM 1570 (Spinach Leaves), NBS SRM 1573 (Tomato Leaves) supplied by the National Institute of Standards and Technology (NIST), Gaithersburg, USA. The certified reference values are available for elements for the assessment of the method accuracy. All solid reference materials were used as bottled, without further grinding and sieving.

It should be stressed that LUTS-1 is a slurred biological material obtained through mechanical homogenization of lobster hepatopancreas tissue.

In the research, three different commercially available multivitamin dietary supplements were analyzed.

To ensure homogeneity, it was necessary to grind the solid samples. This was achieved in an agate pestle and mortar by the manual grinding of solids and by a vibrational mixer mill Model S (Testchem, Pszów, Poland) equipped with 30 ml grinding chamber and rod (6 cm diameter), all made of tungsten carbide.

2.4. Microwave digestion system

A laboratory-built prototype of a high pressure-temperature focused microwave heated digestion system, equipped with a closed TFM-PTFE vessel (30 ml internal volume) based on a design outlined in detail by Matusiewicz [25], was employed for wet-pressure sample digestion.

2.5. Procedure

The analytical procedure consisted of various steps. It included: (1) sample preparation (closed-vessel digestion of the samples or slurry preparation), (2) graphite furnace atomization of selected elements and (3) measurement by HR-CS GFAAS.

Table 1
Instrumental specification and operating parameters for determination of selected elements by HR-CS GFAAS.

Parameter	Setting							
	As	Cd	Cu	Cr	Fe	Mn	Pb	Se
Wavelength (nm)	193.6960	228.8018	324.7540	357.8687	248.3270	279.4817	283.3060	196.0267
Lamp current (A)	9							
Spectral range (pixel)	200							
Dispersion (pm pixel ⁻¹)	2							
Read time (s)	5							
Delay time (s)	0							
Signal measurement	Peak area absorbance							
Modifier	Pd(NO ₃) ₂	Pd + Mg(NO ₃) ₂	Pd + Mg(NO ₃) ₂	None	Mg(NO ₃) ₂	Pd + Mg(NO ₃) ₂	NH ₄ H ₂ PO ₄	Pd(NO ₃) ₂
Modifier concentration (mg l ⁻¹)	2							
Modifier volume (μl)	5							

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