

# CHARACTERIZATION OF INORGANIC COMPONENTS IN MULTIMINERAL/MINERAL SUPPLEMENTS

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## SUMMARY

Nowadays the use of multiminerals/mineral supplements is very common in order to supply their dietetic deficiencies as well as for the prevention and treatment of diseases. Consequently due to this demand, several brands or a great variety of supplements are commercially available on the market. In this work, supplement samples were analysed by neutron activation analysis in order to evaluate the composition of their inorganic essential components and also to compare these results obtained with those presented in labels or in the instructions for the use of the product. Samples of mineral supplements of Zn, Se, Ca and multiminerals vitamins sold in the form of capsules or tablets were analysed. The samples were irradiated with thermal neutron flux of the IEA-R1 nuclear reactor together with the elemental standards and the radioactivity measurements were carried out using a HGe detector coupled to a gamma ray spectrometry. Results obtained in these analyses, in general, presented a good agreement with the label values. Concentrations of toxic elements As and Sb were found in some supplements but in very low concentrations. The advantages of using neutron activation analysis to determine trace elements in supplement samples are discussed.

**Key words: Mineral supplements; Neutron activation analysis; Nutrients**

## INTRODUCTION

Recently, with the knowledge of the role of elements and for ensuring optimal function of human organisms, the use of multimineral/mineral supplements is very common in order to supply their dietetic deficiencies as well as for the prevention and treatment of diseases.

Besides the antioxidant vitamins E, A and C, there is an other group of substances, known as antioxidant enzymes that control the formation of free radicals are used to prevent certain diseases. The elements known as oligo elements take part on the formation of these enzyme molecules or compete with formation of hydroxyl free radicals. The deficiency of these oligo elements injure the antioxidation process. The elements such as Cu, Zn, Mn and Se are the elements having antioxidation function. Therefore the dietetic deficiency of these elements can be supplemented to normalise the antioxidant levels of these enzymes.

The effects these supplements on the health have been also the subject of several researches and conferences in the field nutrition and medicine<sup>(1-4)</sup> and vitamins and mineral supplements are widely consumed by individuals mainly by older adults, lactating mothers, athletes, and pregnant women.

Therefore due to this demand of multimineral/mineral supplements several brands and variety of theses products are commercially available on the market. Consequently it is of great interest to check elemental composition in these kinds of products.

Several techniques have been used in elemental analysis of tablets or pharmaceutical preparations. Lozak and Fijalek<sup>(5)</sup> compared the results obtained in the analyses of Se in pharmaceutical preparations using the method of adsorptive stripping voltametry and atomic absorption spectrometry. In an other paper, Lozak and Fijalek<sup>(6)</sup> analysed tablets and therapeutical nutritions using inductively coupled plasma-mass spectrometry, adsorptive stripping voltametry and atomic absorption spectrometry. Sandhya and Subramanian<sup>(7)</sup> analysed trace amounts of zinc in pharmaceutical preparations using radiometric method.

In this paper instrumental neutron activation analysis was applied in order to evaluate the composition of inorganic essential components in mineral and multimineral supplements to compare these results obtained with those presented in labels or in the instructions for the use of product.

## MATERIALS AND METHOD

### Samples of supplements analysed

The supplements analysed were provided from shops of natural products and from drugstores. The samples analysed were: Natural zinc, Zinc lozenges, Chelated zinc, Cal assimilate plus, Multimineral complex without iron, Vegetarian selenium, Mega multi vitamins and minerals, One source multivitamins and Calcium with vitamin D.

All the supplements were solid form in capsules, tablets or lozenges. The supplement of each capsule or tablet was weighted to obtain the mean values of mass per tablet or capsule. For the analyses, these samples ( 6 or 7 tablets or pills) were mixed and homogenised by grinding in an agate mortar.

### Preparation of synthetic standard of elements.

The standard for comparative neutron activation analysis were prepared by pipetting aliquots of multielemental or single standard solutions onto small pieces of Whatman No. 40 filter paper and drying in a desiccator at room temperature of about 23 °C. These standard solutions containing one or more elements were prepared from standard solutions provided by Spex Chemical. The quantities of the elements irradiated, in µg ( in parentheses) were the followings: Standard 1 contained As(1.5), Cr (2.0), Sb (0.6), Mo(3.0) and Cu (100.0; Standard 2 had Ca(1001.0), Zn (35.0), Rb (10.0) and Fe (350.0), Standard 3 with K (1001.0), La (0.56), Cs (0.56), Sc (0.075), Co (0.150) and Cd (1.0). The quantities of the elements in single standards were the following: Br (5.0), Ba (50.0), Mn (30) and Na (25.0). After drying at room these sheets of filter paper were placed in a clean polyethylene envelopes and irradiated together with the samples. These envelopes were prepared using heat sealer and polyethylene foils previously cleaned using diluted nitric acid solution and distilled water.

## Instrumental neutron activation analysis (INAA)

Samples of about 100 mg and standards were sealed in polyethylene envelopes were irradiated at the IEA-R1 research nuclear reactor for 3 min and 16 h under a thermal neutron flux of  $10^{12}$  n cm<sup>-2</sup> s<sup>-1</sup>. After adequate decay times, the gamma activities of the samples and the elemental standards were measured using a EG & G Ortec hyperpure Ge detector with a resolution 0.90 keV for 122 keV gamma-ray of <sup>57</sup>Co and 1.98 keV for 1332 keV of <sup>60</sup>Co. This detector is connected via TRUMP card to a microcomputer and an electronic system. The gamma spectra were processed using VISPECT2 software developed in our laboratory. The comparative method was used for calculating the concentrations of the elements in the samples. The radioisotopes used in

this study were: <sup>76</sup>As, <sup>131</sup>Ba, <sup>47</sup>Ca, <sup>51</sup>Cr, <sup>60</sup>Co, <sup>59</sup>Fe, <sup>42</sup>K, <sup>56</sup>Mn, <sup>99</sup>Mo, <sup>24</sup>Na, <sup>122</sup>Sb, <sup>46</sup>Sc, <sup>76</sup>Se, <sup>85</sup>Sr and <sup>65</sup>Zn.

## RESULTS AND DISCUSSION

Elemental concentrations obtained in the analyses of zinc supplements are presented in Table 1. From these results, the masses of element per tablet or pill were calculated in order to compare with those results presented in the labels of the products. These results indicate a good agreement between the data of Zn obtained and those values presented in labels or in the instructions for the use of the product.

Table 1. Concentrations of elements obtained in Zn supplements

Samples	This work			Values from the labels (mg)
	[Zn], mg g <sup>-1</sup>	Mean values of the tablet mass (g)	Mass of Zn per tablet or lozenge (mg)	
Natural zinc	91.7 ± 1.7	0.5476 ± 0.142	50.2	50
Zinc lozenger	15.16 ± 0.15	1.4929 ± 0.0082	22.6	23
Chelated zinc	70.2 ± 0.3	0.7396 ± 0.0070	51.9	50

Table 2 shows the concentrations of elements determined in supplements of Ca, Se and in multimineral supplements. Toxic elements such as As and Sb were also found in these analyses, however in very low concentrations. From the results obtained in these analyses, the quantities of nutritional elements present in each tablet were calculated and presented in Table 3. Comparing these results obtained with ones indicated in the labels, it can be concluded that there is a good agreement for most of elements. The major discrepancies occurred in the case of Cr and Se in the samples Multimineral complex without iron and Once source multivitamins, respectively. The quantity of Cr in Multimineral complex was lower than presented in the label of the product and in the case of Se in the sample One source multivitamins its quantity was 34 % higher than the value indicated in the product.

Table 2. Concentrations of elements in multiminerall supplements

Elements	Samples							
	Cal Assimilate Plus	Multiminerall Complex without Iron	Vegetarian Selenium	Mega multi Vitamins & Minerals	One Source Multivitamins	Calcium with vitamin D		
As, $\mu\text{g g}^{-1}$	0.29 ± 0.03*	n.d.**	0.17 ± 0.01	n.d.	n.d.	0.45 ± 0.02		
Ba, $\mu\text{g g}^{-1}$	106 ± 24	99.0 ± 9.3	n.d.	n.d.	n.d.	40.5 ± 3.6		
Ca, %	13.9 ± 0.2	13.1 ± 0.4	14.3 ± 0.5	10.4 ± 0.4	8.22 ± 0.16	31.7 ± 1.3		
Co, $\mu\text{g g}^{-1}$	0.20 ± 0.01	0.076 ± 0.03	0.22 ± 0.01	1.73 ± 0.08	1.20 ± 0.04	0.41 ± 0.01		
Cr, $\mu\text{g g}^{-1}$	4.2 ± 0.2	77.6 ± 0.2	2.1 ± 0.2	2.03 ± 0.15	92.1 ± 0.7	1.96 ± 0.05		
Fe, $\mu\text{g g}^{-1}$	158.1 ± 6.6	53.7 ± 6.6	185.8 ± 6.4	124 ± 8	9358 ± 86	1090 ± 17		
K, %	n.d.	2.99 ± 0.03	0.445 ± 0.013	3.18 ± 0.03	4.33 ± 0.06	0.022 ± 0.005		
Mn, $\mu\text{g g}^{-1}$	552.8 ± 10.2	3847 ± 77	n.d.	2401 ± 19	4125 ± 83	n.d.		
Mo, $\mu\text{g g}^{-1}$	n.d.	47.3 ± 1.5	n.d.	n.d.	n.d.	n.d.		
Na, $\mu\text{g g}^{-1}$	4795 ± 111	2667 ± 13	430.2 ± 2.3	n.d.	n.d.	2661 ± 61		
Sb, $\mu\text{g g}^{-1}$	0.093 ± 0.008	0.80 ± 0.02	0.117 ± 0.005	n.d.	0.205 ± 0.014	0.35 ± 0.02		
Sc, $\mu\text{g g}^{-1}$	0.046 ± 0.002	0.035 ± 0.002	0.056 ± 0.001	36.6 ± 2.4	76.7 ± 3.8	0.268 ± 0.003		
Se, $\mu\text{g g}^{-1}$	n.d.	89.3 ± 0.7	139.7 ± 1.8	226.4 ± 1.4	157.8 ± 0.6	0.25 ± 0.04		
Sr, $\mu\text{g g}^{-1}$	322 ± 11	n.d.	87.0 ± 9.3	n.d.	n.d.	n.d.		
Zn, $\mu\text{g g}^{-1}$	2614 ± 24	13.3 ± 0.1	33.9 ± 0.8	14535 ± 88	7838 ± 47	4.8 ± 0.5		

\* - the uncertainty was calculated using statistical counting errors of standard and sample

\*\* - n.d. indicates not detected

Table 3. Comparison between the masses of elements obtained per capsule with those values presented in the labels of the products

Element	Sample	This work	Value from the label
Ca, mg	Cal Assimilate Plus	126	125
	Multimineral complex without iron	76	62
	Mega multi vitamins & minerals	105	100
	One Source multivitamins	164	165
	Calcium with vitamin D	499.8	500
Cr, mg	Multimineral complex without iron	0.045	0.090
	One Source multivitamins	0.140	0.120
Fe, mg	One Source multivitamins	16	18
K, mg	Multimineral complex without iron	17	15
	One source multivitamins	74	80
Mn, mg	Cal Assimilate Plus	0.501	0.500
	Multimineral complex without iron	2.23	2.5
	Mega multi vitamins & minerals	2.4	2.5
	One Source multivitamins	7.0	7.5
Mo, mg	Multimineral complex without iron	0.027	0.025
Se, mg	Multimineral complex without iron	0.052	0.050
	Vegetarian selenium	0.108	0.100
	Mega multi vitamins & minerals	0.229	0.200
	One source multivitamins	0.269	0.200
Sr, mg	Cal Assimilate Plus	0.292	0.250
Zn, mg	Cal Assimilate Plus	2.3	2
	Multimineral complex without iron	7.7	7.5
	Mega multi vitamins & minerals	14.7	15
	One source multivitamins	13	15

Mean mass of the tablets or pills, in grams: Cal Assimilate Plus =  $0.906 \pm 0.008$ ; Multimineral complex without iron =  $0.581 \pm 0.089$ ; Mega multi vitamins & minerals =  $1.014 \pm 0.013$ ; One Source multivitamins =  $1.707 \pm 0.010$ ; Calcium with vitamin D =  $1.576 \pm 0.010$ ; Vegetarian selenium =  $0.773 \pm 0.014$

Results obtained in this work show that the INAA can provide important information about the composition of essential nutrients present in supplements and this technique can be used to control the composition of the elemental nutrients. This technique presents advantage of the non destructive character and the possibility of simultaneous evaluation of several elements present in a large range of concentration.

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