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Professional paper

Application of an ICP-MS method for determination of metal content in whey protein supplements

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Abstract

The aim of our study was to determine the metal content of manganese (Mn), copper (Cu), iron (Fe), chromium (Cr), selenium (Se), zinc (Zn), arsenic (As), cadmium (Cd), nickel (Ni), lead (Pb), and mercury (Hg) using inductively coupled plasma mass spectrometry (ICP-MS) in different whey protein supplements (WPS), commercially available on the market in North Macedonia. The standard MKC EN 17851:2023 method for determination of various metals in foodstuffs by ICP-MS after microvawe digestion, was verified prior application on real samples. Method's accuracy and precision were assessed using certified reference materials from FAPAS proficiency testing (PT) program. Results indicated that the verified method could be successfully applied for routine determination of the selected metals in WPS with satisfactory analytical performance. The determined content of physiologically important metals Mn, Cu, Fe, Cr, Se, and Zn served to assess the contribution of these metals through WPS use to their recommended daily intake. The results obtained from the determination of potentially toxic elements in these products (As, Cd, Ni, Pb, and Hg) did not exceed the maximum permissible levels given in the European regulation. However, the widespread use of the WPS, particularly in young people, requires continuous monitoring of metals either essential or present as contaminants to avoid health risks.

Key words: whey protein, metals, ICP-MS, method verification

Introduction

Over the last years, the interest in healthy lifestyle and fitness has been growing continuously. Different products that supplement dietary nutritional requirements, especially products that help build muscle mass and enhance physical performance, promote weight loss or are used as meal replacement are very popular (Bandara et al., 2020; Ring et al., 2021). Protein supplements, especially those obtained from whey proteins, are one of the mostly used products. They are classified as complete proteins that contain all nine essential amino acids, particularly branched-chain amino acids (BCAA) like valine, leucine, and isoleucine, are readily absorbed from the body and have a low percentage of lactose. Consumption of whey proteins has a robust ability to stimulate muscle protein synthesis to a greater degree than other proteins such as casein and soy (Bethencourt-Barbuzano et al., 2023; Devries and Phillips, 2015; Kårlund et al., 2019; Van Loon, 2013). Therefore, in recent years, especially in developed countries, the consumption of whey proteins supplements (WPS) has increased (Elgammal et al., 2019; Ring et al., 2021).

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Despite the nutritional benefits, WPS can be contaminated from external sources, including contamination of the food chain from agricultural, anthropogenic activities as well as contamination of the product itself during the manufacturing process. Toxic metals can be found in WPS if milk assimilates toxic metals from animal feed. Since many metals are important for the proper functioning of the human body, WPS, on one hand can be considered a rich source of minerals. On the other hand, the intake of heavy metals present in these products may lead to many toxic effects for the human health such as deterioration of the heart, liver, kidneys, nerves, blood, and lungs as well as chronic accumulation (Elgammal et al., 2019, Ghasemidehkordi et al., 2018). Besides the recommendations of international organizations such as the European Food Safety Authority (EFSA) or the International Olympic Committee (IOC), food supplements to be regulated as individual class of products, which will provide stricter limits for potential contaminants, they are still regulated as food allowing free distribution on the market. As a consequence, the number of reported cases of adulteration and toxic effects of WPS are continuously rising (Gonzalez-Weller et al., 2023, Jiao et al., 2019; Song et al., 2025).

Given the potential implications of widespread consumption of WPS on human health, it is important to develop or apply sensitive and accurate analytical methods that can determine and quantify element levels in WPS (Ring et al., 2021; Guefai et al., 2022). Inductively coupled plasma mass spectrometry (ICP-MS) is an instrumental technique widely used for determination of various metal concentrations in different food samples due to its exceptional sensitivity, specificity, accuracy, and the ability to analyze multiple elements simultaneously (Puente De La Cruz et al., 2024).

This study describes the performance verification and application of the standard MKC EN 17851:2023 method for determination of As, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Se, Hg, and Zn in foodstuff by ICP-MS after microvawe digestion. Metal content in WPS available on the market in R. North Macedonia was determined. The obtained concentrations of the investigated metals in this study were used to calculate the estimated daily intake (EDI) for each metal through WPS consumption.

Material and methods

Chemicals, reagents and materials

All chemicals and reagents used were of analytical grade and prepared by using high quality ultrapure water (Direct-Q 5UV system (Merck, Germany). Conductivity values were less than 0.05 μS/cm. Nitric acid (65% trace metal grade, ρ=1.4 kg/m^3) and hydrogen peroxide (30%, extra pure) were purchased from Thermofisher Scientific. High purity grade argon gas (99.999%), high purity helium gas (99.999%) and high purity hydrogen gas were purchased from Messer Technogas, Skopje.

Multielement ICP-MS standard traceable to NIST containing 36 elements (10 mg/L each), as well as mercury referent standard (1000 mg/L) were obtained from Agilent Technologies, USA and CPA Chem, Bulgaria, respectively. Daily optimization of the instrument's settings was performed using ICP-MS tuning solution (containing Ba, Be, Ce, Co, In, Pb, Th, Tl, Mg in 2% HNO_3, 10 mg/L each). The tuning solution, 1 μg/L (1 ppb) was prepared before analysis using 1% HNO_3. Grade A polymethylpentene volumetric flasks, as well as beakers, graduated cylinders, 15 mL and 50 mL polypropylene (PP) sample tubes and disposable pipettes were used during entire analysis. Certified reference materials (CRMs) from the FAPAS proficiency testing program were used as quality control in this study: Nutrional Food Supplement 07509 (powdered whey protein), and Rice powder 07419.

Whey protein samples were purchased from food supplement and sport nutrition stores in Skopje, R. North Macedonia. A total of nine samples were included in the study, purchased during 2024. The samples were coded with marks from P1-P9 and all samples were kept at 25 °C until analysis.

Instrumentation

The microwave digestion of whey proteins was conducted using high-performance microvawe digestion system (Milestone Ethos Up, Sorisole, Italy). This system was equipped with 15 tetrafluoromethoxy (TFM) vessels with a volume of 100 mL and suitable for all applications.

An inductively coupled plasma mass spectrometer (ICP-MS), PlasmaQuant MS Q (Analytic Jena, Germany) was used for the determination of the investigated metals in WPS. The ICP-MS operating conditions are presented in Table 1. The instrument was operated in the H_2 mode for the Se determination and in the He mode for the Cr determination.

Preparation of standard solutions

The calibration standards were prepared by dilution of the certified Agilent ICP/MS multielement standard solution to obtain stock standard concentrations of 100 ppb and 10 ppb, using 5% HNO_3. Nine calibration standards for As, Cd, Cr, Cu, Mn, Mo, Ni, Pb, Se, and Zn with concentrations of 0.5, 1, 2, 5, 10, 20, 50, 100, and 200 ppb were prepared in 5% HNO_3. For the Hg determinations, standard solutions containing 0.1, 0.2, 0.5, 1, 1.5, 2.5, 5, 7.5, and 10 ppb were prepared.

Sample preparation

The WPS sample preparation was performed according to the standard method EN 13805:2015. In brief, approximately 0.3 – 0.5 g of each homogenized sample was accurately measured into the TFM vessel and 3 mL HNO_3, 65%, were added followed by addition of 1 mL H_2O_2, 30%. The vessels were closed and the digestion program was performed according to the manufacturer's instructions. A

Table 1. ICP-MS (PlasmaQuant MS Q, Analytik Jena) operating conditions

Spray chamber	scott type double pass glass
Nebulizer	micromist concentric glass
RF power (W)	1300 W
Argon nebulizer gas flow (L/min)	7.5-10.5 L/min
Measures	
Scan mode	peak hopping
Resolution (amu)	0.8
Replicate time (s)	9.78
Dwell time (s)	50
Sweeps/reading	20
Intergration time (ms)	1000
Replicates	3
Isotopes	^{52}Cr, ^{55}Mn, ^{57}Fe, ^{60}Ni, ^{65}Cu, ^{66}Zn, ^{75}As, ^{78}Se, ^{111}Cd, ^{202}Hg, ^{206,207,208}Pb

blank solution containing only 3 mL HNO_3 65% and 1 mL H_2O_2, 30% was also prepared. The digestion procedure was performed in two steps. In the first one, the power was set at 1600 W for 15 min until the temperature reached 200 °C. In the second step, the power was set again at 1600 W for 15 min to hold the temperature at 200 °C. After cooling, the digest was quantitatively transferred in 50 mL plastic calibrated volumetric vessel and these solutions were used for the metal determinations. All samples were analyzed in triplicate.

Method verification

Prior application of the standard method on real samples, verification was performed for the following parameters: linearity, limit of detection (LOD), limit of quantification (LOQ), accuracy and precision, according to ISO 17025 requirements (ISO 17025, 2012).

Estimated metal daily intake (EDI)

The metal levels determined in the investigated samples were used to calculate the EMDI which characterizes the metal intake resulting from each WPS consumption.

$$EMDI_{WPS} = C \times RDI$$

Where: EMDI – estimated metal daily intake (mg/day or µg/day); C – concentration of each metals found in the sample (mg/kg or µg/kg); RDI – recommended daily intake of WPS (g/day) according to the manufacturer's instructions. The daily intake ranging from 25 g to 35 g WPS is common. For the essential metals, the calculated intake was compared to the recommended daily intake in order to evaluate the contribution of the investigated WPS to the total metal intake per day.

Results and discussion

In this study, the content of eleven selected metals in WPS freely available on the market in R. North Macedonia was determined. Since food supplement industry does not imply strict regulation of contaminants during the manufacturing process and these products are mainly regulated as food, trace amounts of diverse metals (essential or toxic) present in these products can pose serious health threat.

Prior application on real samples, the performance verification of the standard MKC EN 17851:2023 method was performed. The standard method does not include the determination of Hg and Fe, however, these elements were included in our study.

Linearity – Linearity was confirmed by analysis of the calibration curves of the examined elements. All calibration curves showed good linearity where the correlation coefficient values (R^2) were higher than 0.998. Limit od detection (LOD) as well as limit of quantification (LOQ) were calculated based on the analysis of blank samples (Millour et al., 2011; Pinto et al., 2020). The results are presented in Table 2.

The method's accuracy and precision was investigated by analyzing samples with reliably known content of the analyzed elements. CRMs comparable to the investigated samples in terms of matrix and concentration range, such as Nutrional Food Supplement 07509 (powdered whey protein) was used. The accuracy of Hg, Pb, Cd, and As determination was additionally estimated using Rice powder 07419 CRM. The results from method's accuracy and precision determination using the CRMs are presented in Table 3.

Table 2. Results from the determination of linearity, LOD, and LOQ

Element	Linear range (µg/L)	LOD (µg/L)	LOQ (µg/L)	Correlation coefficient (R^2)	Intercept (a)	Slope (b)	RSD* (%)
Mn	0.5 - 200	1×10^{-6}	3×10^{-6}	0.9997	0.99	0.95	1.48
Fe	0.5 - 200	3×10^{-3}	1×10^{-2}	0.9992	0.98	2.35	2.05
Ni	0.5 - 200	2×10^{-6}	8×10^{-6}	0.9997	0.99	0.86	2.38
Cu	0.5 - 200	6×10^{-6}	1.7×10^{-5}	0.9997	0.99	0.92	2.39
Zn	0.5 - 200	8×10^{-6}	2.5×10^{-4}	0.9992	0.99	1.92	4.01
As	0.5 - 200	1×10^{-6}	3×10^{-6}	0.9999	0.99	0.62	3.16
Cd	0.5 - 200	3×10^{-7}	1×10^{-6}	0.9998	0.99	0.71	3.24
Hg	0.1 - 10	8×10^{-7}	4×10^{-6}	0.9983	1.09	0.11	3.40
Pb	0.5 - 200	1×10^{-6}	7×10^{-6}	0.9999	0.99	0.45	2.62
Cr	0.5 - 200	2×10^{-5}	5.1×10^{-5}	1.0000	0.99	0.04	4.37
Se	0.5 - 200	1×10^{-5}	2.1×10^{-5}	0.9995	0.98	0.30	3.24

*RSD calculated from 3 determinations

Table 3. Results obtained for method's accuracy and precision using CRM 07509 and CRM 07419

CRM type					
07509 Nutritional Food Supplement (whey protein)					
Element	Assigned value (mg/kg)	Amount found* (mg/kg)	Recovery (%)	RSD (%)	Z - score
Mn	11.90	10.90 ± 0.48	91.61	4.37	0.76
Fe	85.30	83.65 ± 4.92	98.07	5.88	0.24
Ni	1.37	1.52 ± 0.08	111.27	5.32	0.74
Cu	7.84	6.75 ± 0.37	85.74	5.52	1.22
Zn	24.50	19.67 ± 0.48	80.29	2.54	2.00
As	57.70	61.67 ± 3.41	106.87	5.53	0.31
Cd	0.03	0.03 ± 0.88	85.66	3.11	0.65
Hg	0.02	0.02 ± 1.37	80.22	9.25	1.03
Pb	0.05	0.05 ± 3.81	103.41	7.69	0.16
Cr	0.54	0.64 ± 51.19	119.64	7.97	1.63
Se	0.29	0.25 ± 13.43	86.45	5.29	0.71
07419 Rice powder					
Element	Assigned value (µg/kg)	Amount found (µg/kg)	Recovery (%)	RSD (%)	Z - score
As	211.00	247.25±9.08	117.18	4.18	1.49
Cd	96.00	105.00±3.82	109.16	3.65	0.42
Hg	106.00	119.00±2.72	112.58	2.28	0.57
Pb	291.00	289.00±3.23	99.36	1.12	0.03

*Results are expressed as mean± SD

As it can be seen from the results (Table 3), the obtained values for the recoveries ranged 80.22 - 119.64% and the RSD values between 1.12 - 9.25%, indicating good accuracy of the method. All RSD values were below 10%, indicating that the method is precise (Ghuniem et al., 2019).

Method application

After the method's verification, it was applied for determination of the As, Cd, Cr, Cu, Mn, Mo, Ni, Pb, Se, Hg, and Zn levels in nine different WPS available on the market. The measured levels for the different WPS are presented in Table 4 and 5. Table 4 shows the elements present in mg/kg range whereas Table 5 shows the elements present in μg/kg range. All eleven investigated elements were determined in all investigated samples.

Manganese, an element belonging to the group of heavy elements, is essential for the proper functioning of the human body. The concentration of this element in the investigated samples ranged from 0.10 - 13.16 mg/kg. If we take a look at the recommended daily intake for this element through food (2 mg/day) (Official Gazette, 67/2018, 2018) and calculated EMDI for Mn (Table 6) based on determined Mn content in each WPS (Table 4) and the recommended daily portion of the WPS manufacturer (approximately 30 g WPS/ day), it can be assumed that there is no risk for the consumer.

Fe is another essential element required for many vital body functions. However, excess intake can cause various adverse effects such as increased risk for cardiovascular problems, cancer, and endocrine problems as well as liver disease (Ghuniem et al., 2019). Measured Fe content in investigated WPS ranged 20.48 - 66.10 mg/kg. The highest level of Fe determined was 66.10 mg/kg (P1) (Table 4) and the calculated EMDI for this WPS was 2.18 mg/kg Fe (Table 6). Using the recommended daily intake that is 14 mg (Official Gazette, 67/2018, 2018), the Fe EMDI for P1 is 15.6%, indicating safe consumption for the consumer.

Cu is found in the human body mostly bound to several metalloproteins, in which he has a structural role and acts as a cofactor for the catalytic activity. Cu levels in our samples ranged from 0.710 - 11.63 mg/kg. It is apparent that there is one product which provides 38% of the recommended daily intake for this element of 1 mg (Official Gazette, 67/2018, 2018). However, the maximum daily intake for Cu is limited to 3 mg and if this product is carefully used without exceeding the recommended serving portion by the manufacturer (app. 30 g product), there is no risk for the consumers. For the other investigated WPS, the EMDIs were significantly lower (Table 6) with no risk of any Cu-related side effect.

The measured values of Zn ranged from 1.93 - 70.06 mg/kg. The same WPS with the highest Cu content contained highest levels of Zn among the investigated samples (Table 4) which corresponds to 23% of the recommended daily intake for Zn (RDI of 10 mg) (Official Gazette, 67/2018, 2018). The maximum daily intake of Zn that is set up at 15 mg allows safe use of the products according to the manufacturer's recommendations.

WPS contained Se levels ranging from 5.12 - 1002 μg/kg (Table 5). Two of the investigated samples contained higher Se levels (Table 5) corresponding to 32% and 60% of the RDI (55 μg) (Official Gazette, 67/2018, 2018). The usual diet is relatively poor with Se, an element with important antioxidant properties. In this context, the use of WPS with higher Se content may be beneficial but careful use is suggested in order not to exceed the maximum daily intake (100 μg).

In the investigated WPS, the Cr concentration was 10.52 - 893.1 μg/kg. In four WPS, Cr levels respective to the range 12 - 20% of the RDI were calculated. However, considering the maximum daily intake of 150 μg (Official Gazette, 67/2018, 2018), these products are probably safe for consumption.

Table 4. Determined metal content in mg/kg of the investigated WPS

	Metal (mg/kg)				
Sample ID	Mn	Fe	Ni	Cu	Zn
P1	5.37 ± 0.16	66.10 ± 0.54	0.99 ± 0.05	5.26 ± 0.35	14.76 ± 0.75
P2	1.52 ± 0.05	33.62 ± 0.72	0.63 ± 0.06	1.87 ± 0.003	4.93 ± 0.64
P3	3.16 ± 0.09	42.13 ± 1.87	0.83 ± 0.05	2.56 ± 0.006	4.80 ± 0.59
P4	13.16 ± 0.82	42.66 ± 0.82	0.68 ± 0.08	11.63 ± 0.88	70.06 ± 6.45
P5	0.13 ± 0.004	20.48 ± 0.82	0.37 ± 0.06	0.71 ± 0.04	1.93 ± 0.82
P6	3.22 ± 0.06	45.52 ± 1.08	1.01 ± 0.05	3.48 ± 0.04	6.43 ± 0.44
P7	1.51 ± 0.004	20.26 ±1.75	0.44 ± 0.08	1.24 ± 0.03	13.63 ± 0.57
P8	0.12 ± 0.03	23.58 ± 2.24	0.68 ± 0.04	1.15 ± 0.36	4.39 ± 2.12
P9	0.10 ± 0.01	21.92 ± 2.25	0.41 ± 0.10	1.27 ± 0.13	3.95 ± 1.07

*Results are expressed as mean± SD (n=3)

Table 5. Determined metal content in μg/kg of the investigated WPS

	Metal (μg /kg)					
Sample ID	Cd	Hg	Pb	Cr	Se	As
P1	12.49±1.09	31.94±4.43	66.15±11.22	563.09±5.55	73.22±0.16	33.87 ± 1.01
P2	10.86±0.59	40.25±8.00	56.16±1.48	10.54±0.03	5.12±0.01	46.79 ± 2.11
P3	9.19±0.78	52.19±6.48	70.58±12.76	584.16±7.69	370.59±0.92	29.11 ± 0.07
P4	1.99±0.59	93.89±11.92	85.86±10.17	897.46±6.16	1002.86±2.91	73.29 ± 2.58
P5	1.04±0.52	22.57±3.36	26.95±10.72	328.94±4.48	310.62±0.59	38.74 ± 0.40
P6	8.10±0.64	14.67±1.59	46.64±10.42	635.70±10.78	529.830±0.59	26.63 ± 0.43
P7	1.12±0.53	10.54±1.17	30.66±10.97	409.81±6.36	154.30±0.50	28.69 ± 0.46
P8	3.26±1.45	19.27±2.23	47.88±25.02	309.97±0.78	359.26±0.38	23.58 ± 1.00
P9	2.43±1.02	17.31±1.64	29.25±12.70	311.31±0.22	378.75±1.25	22.73 ± 1.23

*Results are expressed as mean± SD (n=3)

Table 6. Estimated metal daily exposure (EMDI) to the metals present in WPS considering common serving size of approximately 30 g per day

Sample ID	Investigated metal concentration per serving (33 g)										
	Mn	Fe	Ni	Cu	Zn	Cd	Hg	Pb	Cr	Se	As
EMDI	mg	mg	mg	mg	mg	μg	μg	μg	μg	μg	μg
P1	0.18	2.18	0.03	0.17	0.49	0.41	1.05	2.18	18.58	2.42	1.12
P2	0.05	1.21	0.02	0.06	0.16	0.36	1.33	1.85	0.35	0.17	1.54
P3	0.10	1.39	0.03	0.08	0.16	0.30	1.72	2.33	19.28	12.23	0.96
P4	0.43	1.41	0.02	0.38	2.31	0.07	3.10	2.83	29.62	33.09	2.42
P5	0.00	0.68	0.01	0.02	0.06	0.03	0.74	0.89	10.85	10.25	1.28
P6	0.11	1.50	0.03	0.11	0.21	0.27	0.48	1.54	20.98	17.48	0.88
P7	0.05	0.67	0.01	0.04	0.45	0.04	0.35	1.01	13.52	5.09	0.95
P8	0.00	0.84	0.02	0.04	0.14	0.11	0.64	1.58	10.23	11.86	0.78
P9	0.00	0.70	0.01	0.04	0.13	0.08	0.57	0.97	10.27	12.50	0.75

As, Cd, Ni, Pb, and Hg are highly toxic metals that can accumulate in different organs in the human body and therefore represent toxicological concern in a prolonged period of time. In the investigated WPS, the determined level of As were 22.73 - 46.79 μg/kg, the Cd level ranged 1.12 - 12.49 μg/kg, the Ni level ranged 0.37 - 1.01 mg/kg, the Pb level in the investigated samples ranged 26.95 - 85.86 μg/kg, and Hg content ranged 14.67 - 93.90 μg/kg. According to the maximum permitted levels for the investigated toxic elements in the European regulative for food, the obtained content for these elements in all WPS complied with the regulation (Regulation (EC) 2023/915; Regulation (EC) 2024/1987).

Conclusion

In our study, performance verification and the application of the standard MKC EN 17851:2023 method for determination of Mn, Cu, Fe, Cr, selenium (Se), zinc (Zn), arsenic (As), cadmium (Cd), nickel (Ni), lead (Pb), and mercury (Hg) in WPS is presented. The verified method was successfully applied for determination of these metals in WPS commercially available on the market. Considering the calculated EMDI values based on the determined content of physiologically important metals in the investigated WPS, they are safe when used according to

the manufacturers recommendations. The toxic metal content in all tested samples was in accordance with the European regulative requirements. However, it is suggested for the consumers to seek advice from dietitian or other specialized practitioner to ensure WPS safe consumption.

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Резиме

Примена на ICP-MS метод за определување на метали во производи од суруткини протеини

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Клучни зборови: суруткини протеини, метали, ICP-MS, верификација

Целта на нашето истражување беше определување на содржината на манган (Mn), бакар (Cu), железо (Fe), хром (Cr), селен (Se), цинк (Zn), арсен (As), кадмиум (Cd), никел (Ni), олово (Pb), и жива (Hg) со примена на индуктивно-спрегната плазма масена спектрометрија (ICP-MS) во производи од суруткини протеини и се комерцијално достапни на пазарот во Република С. Македонија. Стандардниот МКС EN 17851:2023 метод за определување на разни метали во храна со примена на ICP-MS, по микробранова дигестија на примерокот, беше верификуван пред да се примени на реални примероци. Точноста и прецизноста на методот беа проценети преку анализа на сертифицирани референтни материјали добиени од FAPAS програмата за меѓулабораториски споредби. Резултатите покажаа дека верификуваниот метод може успешно да се примени за определување на одредени метали во производи од суруткини протеини со задоволителни аналитички перформанси. Определената содржина на физиолошки значајните елементи Mn, Cu, Fe, Cr, Se и Zn беше применета за процена на дневниот удел на испитуваните метали од производите во исхраната имајќи го предвид препорачаниот дневен внес за секој метал. резултатите добиени од одредувањето на потенцијално токсичните елементи во овие производи (As, Cd, Ni, Pb и Hg) не ги надминаа максимално дозволените нивоа дадени во Европската регулатива. Сепак, многу е важно да се напомене дека масовната употреба на овие производи, особено кај младата популација, бара постојано следење на содржината на метали (есенцијални или токсични) како контаминенти, со цел да се избегнат ризиците по здравјето.