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Analysis of the mineral content in dietary supplements for athletes*

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Abstract

Dietary supplements are becoming increasingly popular, yet these products are subject to less stringent quality regulations than pharmaceuticals. The present study employed the ICP-OES method to analyze a selection of elements (Ca, Mg, P, Al, and Zn) in dietary supplements. Commercially available products, purchased in Poland through online sales, were selected for the analysis. The study was conducted to assess compliance between the actual composition of the supplements and the manufacturers' declarations. Additionally, it was used to estimate daily exposure to metals at the recommended intake. A number of supplements exhibited discrepancies between their declared and actual composition, suggesting variability resulting from manufacturing processes and inadequate quality control. In the majority of the analyzed supplements, the concentrations of elements derived from excipients were found to be lower than the stated values on the product labels. Additionally, the presence of certain elements, such as calcium (Ca) in two products and phosphorus (P) in one, was not declared despite their substantial concentrations. An evaluation of product exposure indicated that the Recommended Daily Intake (RDI) for zinc was surpassed in merely two products, which could potentially pose health concerns with prolonged use. The most significant variability in composition was observed in multi-ingredient supplements, particularly those containing caffeine, while creatine supplements demonstrated greater consistency. These findings underscore the need for enhanced quality control in dietary supplements, particularly with respect to the composition of excipients and the consistency between the declared and actual composition of the products.

Keywords: metals, sport supplements, ICP-OES, creatine, caffeine

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INTRODUCTION

A dietary supplement (DS) is defined by the Federal Food, Drug, and Cosmetic Act as a product intended to supplement the diet, containing one or more dietary ingredients such as vitamins, minerals, amino acids, plant substances or their extracts, metabolites, or concentrates (Maughan et al. 2018). Dietary supplementation is important for people with dietary restrictions caused by food allergies or intolerances. For example, individuals with cow's milk protein allergies may need to supplement calcium, vitamin D, and vitamin A. Among athletes, supplement use is particularly significant due to the body's increased nutrient requirements resulting from intense physical activity. In many cases, it is difficult to meet nutritional needs, especially for protein, through a conventional diet alone, which makes the use of concentrated forms of nutrients a viable option. However, dietary supplements cannot replace a balanced diet as the primary source of nutrients (Alfieri et al. 2023). Their use is often based on beliefs rather than knowledge of the mechanisms of action, bioavailability, and metabolic interactions of the active ingredients (Maughan et al. 2007).

The value of the dietary supplement market in Poland in year 2020 was forecasted to reach 5.04 billion PLN (around 1 billion USD) with annual growth of 0.35. The global market of dietary supplements in the mentioned year was valued at 140.3 billion USD, indicating a rapid growth in the dietary supplement market. (Hys et al. 2022) A study conducted in 2025 by Brodnicka et al. states that 35% of adult Poles use dietary supplements on a daily basis. The frequency of use of dietary supplements by athletes ranges from 51% to 98%, depending on type of sport and gender. In the above study, results from 659 Polish participants showed that 91.1% of surveyed athletes reported using dietary supplements. More than 45% of professional as well as non-professional athletes use caffeine and creatine as a performance supplement (Broniecka et al. 2025). Despite the increasing availability of dietary supplements for athletes and their widespread use, the mineral composition of these products and the accuracy of their declared elemental content remain insufficiently studied, particularly in creatine- and caffeine-based supplements. These substances are among the most commonly used active ingredients in competitive and recreational sports (Maughan et al. 2007); however, the excipients and fillers may serve as undeclared sources of minerals, including both macro-minerals and potential metallic contaminants.

In the United States, dietary supplements are regulated by the Food and Drug Administration (FDA) under the Dietary Supplement Health and Education Act of 1994 (DSHEA). According to its provisions, dietary supplements are classified as a distinct category of food, not as medicinal products, meaning manufacturers are not required to obtain an FDA approval before marketing their products. The manufacturer is solely responsible for safety and proper labeling. As dietary supplements are subject to less stringent quality

control requirements than medicinal products, there is a legitimate risk of discrepancies between the declared and actual composition. This includes elements introduced into the product not as active ingredients, but as byproducts of manufacturing processes or through the use of fillers as well as contaminants (Fu et al. 2009, Abernethy et al. 2010).

Contaminants in products are divided into three groups: organic, inorganic, and solvent residues. Metals are classified as inorganic impurities, and they are monitored in medications and their ingredients, as well as in food and dietary supplements. The toxicity of metals is well documented in scientific literature, and the toxic effect can be acute, chronic, or sub-chronic (Balaram 2016, Maithani et al. 2019, Chawla et al. 2020, Sheth and Patel 2020, Aleluia et al. 2023, Dimpal et al. 2024, Cordeiro et al. 2024, Mande et al. 2024). The most dangerous types of toxicity caused by these contaminants are neurotoxicity, nephrotoxicity, and hepatotoxicity. They affect the functioning of the cardiovascular system and contribute to risks related to reproduction and development through neurotoxicity, immunotoxicity, and carcinogenicity (Abernethy et al. 2010).

Dietary supplements may be contaminated with heavy metals, some of which are recognized as harmful to human health even at low exposure levels. Many trace elements are used in formation of active components that have therapeutic properties. However, these elements in high concentration can lead to toxicity. Exposure to heavy metals, such as lead, can cause a range of symptoms headache, irritability, abdominal pain and various neurological disturbances. Many minerals that are essential to human physiology in appropriate amounts become potentially harmful once a certain threshold of exposure is exceeded (Canbay, Doğantürk 2016).

Dietary supplements are intended for long-term and regular consumption; as a result, the contaminants they contain may accumulate in the body. This risk is further exacerbated when the actual mineral content of a product deviates from the composition declared on the manufacturers' label, which may result in consumers receiving intakes different from those intended, leading to deficient supplementation or excessive exposure. This underscores the importance of verifying label accuracy as an integral component of dietary supplement quality and safety assessment. Recent studies demonstrate that plant-based herbal products are more likely to contain heavy metals than conventional, highly processed products (Fugh-Berman 2009, Sakharkar 2017, Luo et al. 2021, Zuo et al. 2023, Bhalla, Pannu 2022). The aim of this study was to conduct quantitative analysis of the content of selected mineral elements (Ca, Mg, P, Al, Zn) in dietary supplements for athletes, based on caffeine and/or creatine, using the ICP-OES method, and to assess the consistency of the results obtained with the composition declared by the manufacturer on the product label.

MATERIALS AND METHODS

The study included conventionally formulated, highly processed dietary supplements ($n=26$) available for retail sale in Poland, purchased from sports nutrition stores and online retailers between 2024 and 2025. The selection criterion was the presence, as declared by the manufacturer, of the active ingredient: caffeine (CAF, $n=10$), creatine (Cr, $n=10$), or both ingredients combined (CAF & Cr, $n=6$). The selection of supplements aimed to include products commonly used by athletes that are representative of the Polish market in terms of form (tablets, capsules, powders) and diversity of manufacturers. Each supplement was represented by a single production batch, analyzed in three analytical replicates.

Approximately 0.5 g of each homogenized sample was subjected to microwave-assisted digestion using a microwave digestion system (Anton Paar, Graz, Austria). The samples were transferred to Teflon vessels previously acid-etched, to which 3 mL of nitric acid (HNO_3) and 1.5 mL of hydrogen peroxide (H_2O_2) were added. After the mineralization process was complete, the vessels were left to cool to room temperature. The resulting solutions were transferred volumetrically to polypropylene vials, diluted to a volume of 50 mL, and then analyzed using ICP-OES (Inductively Coupled Plasma Optical Emission Spectroscopy). This technique enables simultaneous multi-element determination of both light and heavy elements in complex matrices. The method is characterized by low limits of detection (LOD), short analysis times, and ease of sample preparation (Maćkiewicz et al. 2025). The content of the following elements was determined in each sample: calcium (Ca), magnesium (Mg), phosphorus (P), aluminum (Al), and zinc (Zn). Each sample was analyzed three times, and the results were expressed as the average of the three replicates. For each supplement, the declared content of the analyzed elements was specified by the manufacturer on the product label, and the experimentally determined values were subsequently compared with these declared values to assess label accuracy. Data were compiled and processed using Microsoft Excel 2013.

RESULTS AND DISCUSSION

The results of the multi-element ICP-OES analysis of calcium, magnesium, phosphorus, and aluminum across the groups: CAF (caffeine-based supplements), Cr (creatine-based supplements), and CAF & Cr (containing both substances) are presented in Figure 1*a-d*, while zinc content is shown before and after outlier removal in Figure 1*e* and 1*f*, respectively. The concentration distributions obtained allow an assessment of intra-group variability and evaluation of differences between groups.

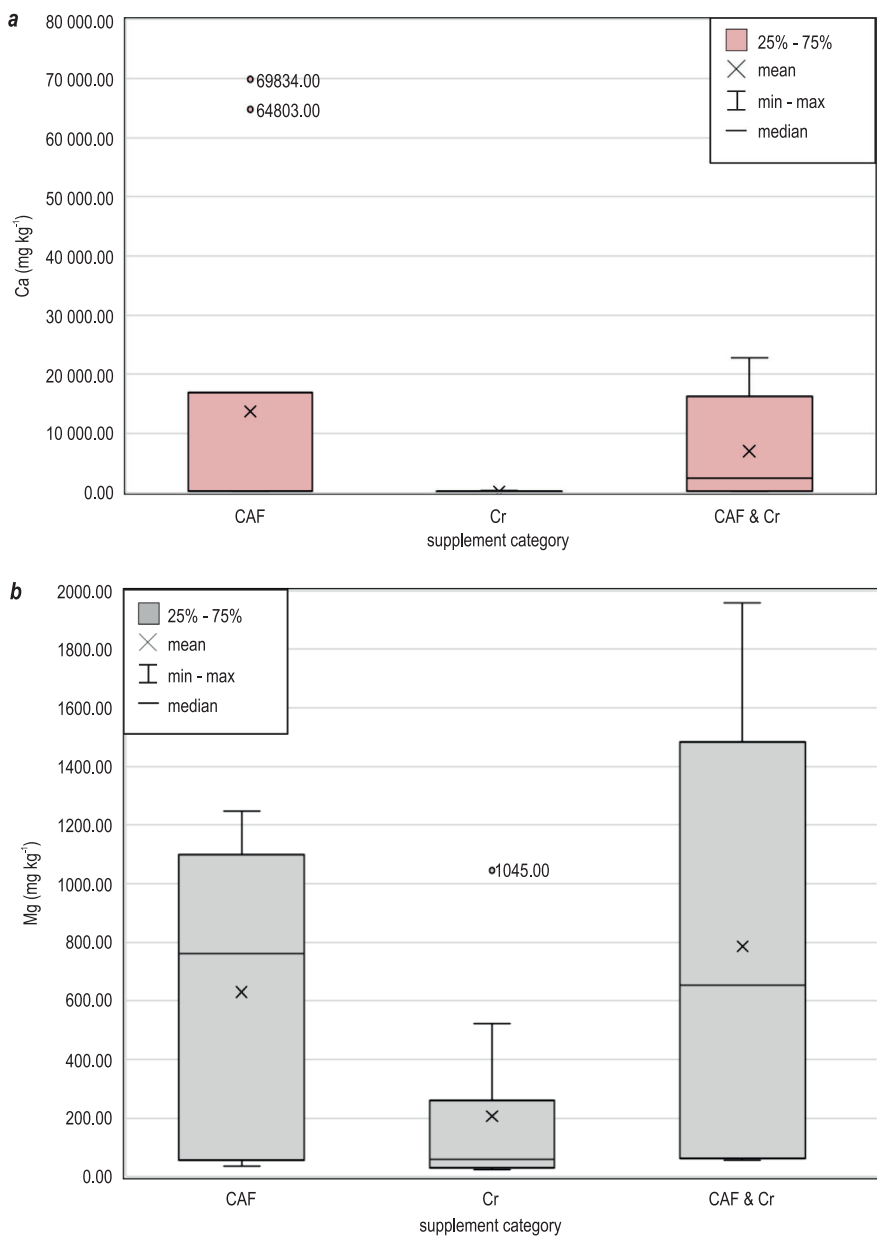


Fig. 1a, b. Graph showing the content of calcium (a) and magnesium (b) in supplement types (CAF (caffeine-based supplements), Cr (creatine-based), and CAF & Cr (containing both substances simultaneously))

The distribution of calcium concentrations (Figure 1a) showed significant variation both between and within groups. The highest median calcium concentrations were observed in the CAF and CAF & Cr groups, with the CAF

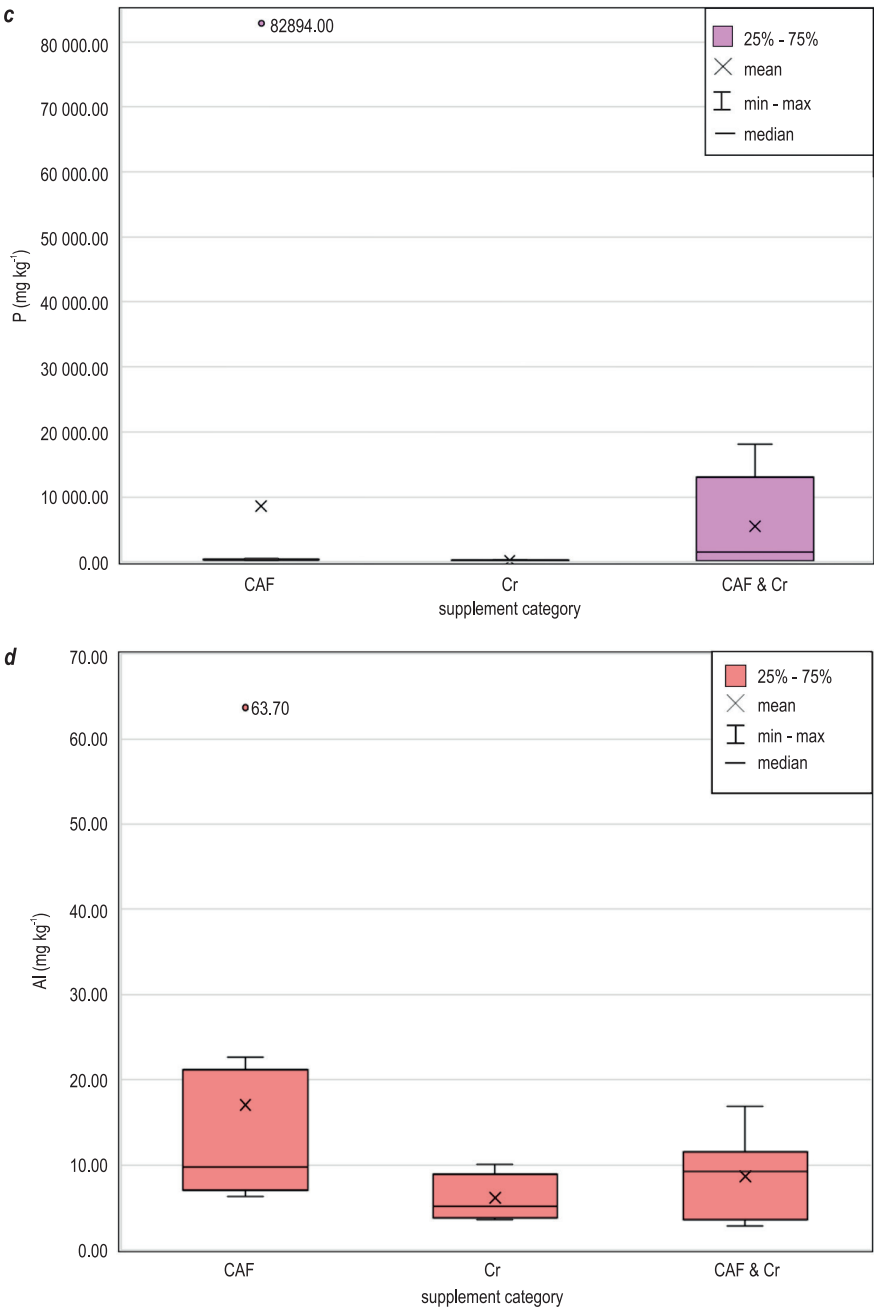


Fig. 1c, d. Graph showing the content of phosphorus (c) and aluminum (d) in supplement types (CAF (caffeine-based supplements), Cr (creatine-based), and CAF & Cr (containing both substances simultaneously))

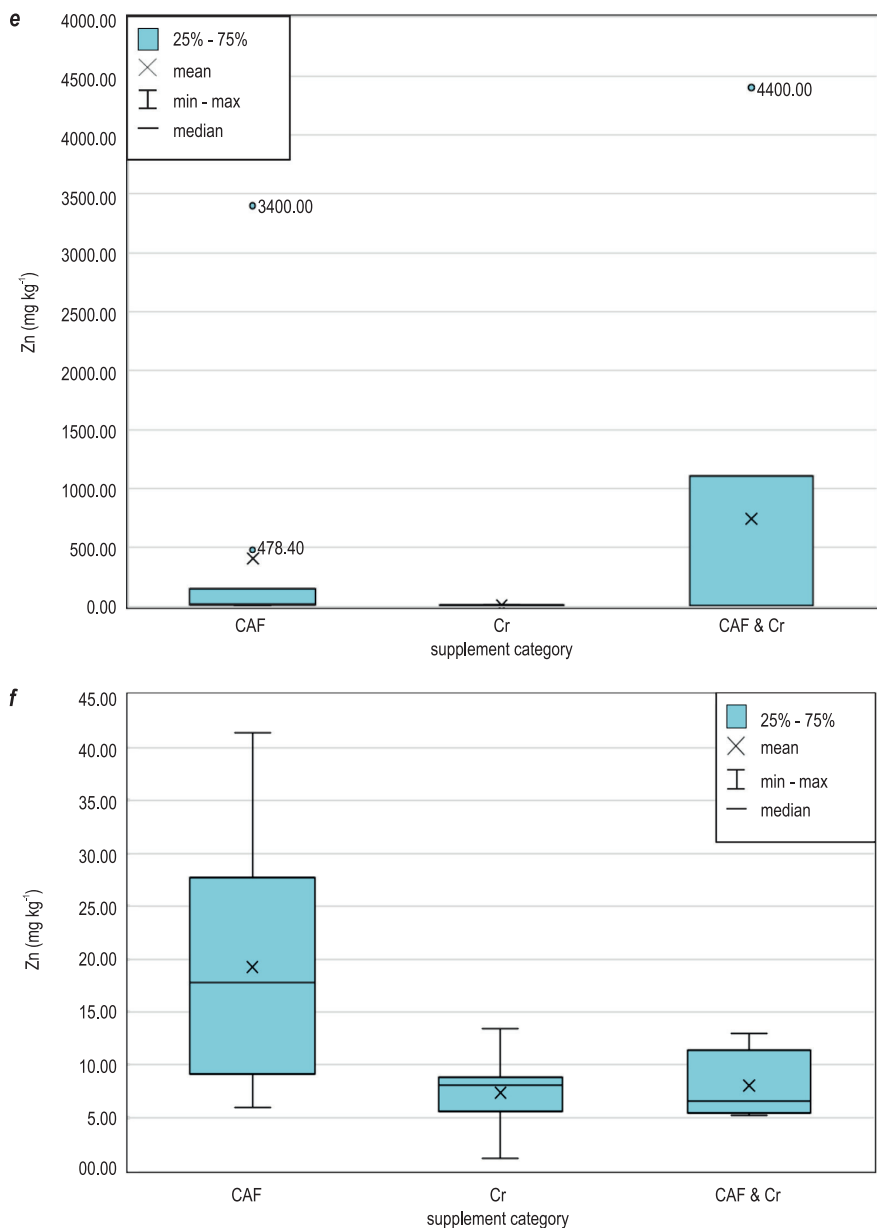


Fig. 1e, f. Graph showing the content of zinc before (e) and after removing outliers from the graph (f) in supplement types (CAF (caffeine-based supplements), Cr (creatine-based), and CAF & Cr (containing both substances simultaneously))

group also exhibiting the greatest data dispersion, indicating a marked heterogeneity in composition. Two outliers were identified in the CAF group, with calcium concentrations of approximately 69 834 mg kg⁻¹ (DS6) and

64 803 mg kg⁻¹ (DS8), which exceeded the values of the other samples in this group. The high calcium content, coupled with the absence of information about this element on the labels of both products, indicates the use of calcium-based fillers, most likely calcium carbonate or calcium phosphate. The Cr group had the lowest calcium concentrations of all groups; their median did not exceed 300 mg kg⁻¹, and the range of values was significantly narrower, indicating a more uniform and simplified composition of excipients in this product group. The literature confirms that calcium concentrations in dietary supplements vary widely depending on the market and the type of product. Udousoro et al. (2017) found that calcium concentrations in supplements available on the Nigerian market varied widely, from 12 to 82 600 mg kg⁻¹, a range comparable to the values reported in this study. Filipiak-Szok et al. (2015), when determining the elemental composition of dietary supplements using ICP-MS and ion chromatography (IC), reported calcium concentrations ranging from 4 890 to 25 140 mg kg⁻¹ (ICP-MS) and from 2 620 to 7 130 mg kg⁻¹ (IC), respectively. In the study by Avula et al. (2010), the calcium concentrations in the supplements examined ranged from 1 170 to 22 700 mg kg⁻¹; one supplement had a calcium concentration below 1 000 mg kg⁻¹. Oško et al. (2022) reported relatively low calcium concentrations, ranging from 0.02 to 89.01 mg kg⁻¹, with two samples analyzed in the study falling below the limit of detection (LOD).

The distribution of magnesium concentrations (Figure 1b) showed clear differences between the groups. The widest range was observed in the CAF & Cr group, where magnesium concentrations varied from approximately 57 to over 1 950 mg kg⁻¹, indicating significant differences in the composition of excipients used by individual manufacturers in this category. The CAF group also exhibited a wide range, with four out of ten samples exhibiting concentrations below 150 mg kg⁻¹, while the remaining samples had concentrations of this metal exceeding 700 mg kg⁻¹. The bimodal distribution suggests that some manufacturers do not use magnesium salts as excipients at all, while others use them in significant quantities. The narrowest range was found in the Cr group, where most data points were within approximately 30 to 500 mg kg⁻¹ with one outlier at 1045 mg kg⁻¹ (DS16). The lower and more uniform magnesium concentrations in the Cr group confirm that the matrix of these products is less complex compared to the other supplements. Filipiak-Szok et al. (2015) reported magnesium levels in dietary supplements ranging from 3 030 to 7 480 mg kg⁻¹ when analyzed by ICP-MS, and from 820 to 3 020 mg kg⁻¹ when analyzed by ion chromatography. These values are higher than those reported in this study, which may reflect the different category of products examined. The magnesium concentration in dietary supplements in the study by Udousoro et al. (2017) ranged from 1.85 to 2 440 mg kg⁻¹, whereas in the study by Oško et al. (2022), it was within a narrow range of 0.01 to 4.05 mg kg⁻¹. Avula et al. (2010) reported magnesium concentrations ranging from 1 830 to 11 200 mg kg⁻¹, with most samples between 3 000 and 6 000 mg kg⁻¹.

The distribution of phosphorus concentrations (Figure 1c) showed little intra-group variation in most samples. In the CAF group, most phosphorus concentrations ranged from 189.8 to 488 mg kg⁻¹; in the Cr group, the range was narrower, from 195 to 246 mg kg⁻¹. The only outlier was sample (DS8) from the CAF group, which had a phosphorus concentration of 82 894 mg kg⁻¹, several dozen times higher than the others. This sample also had a high calcium concentration, indicating that calcium phosphate was used as a filler in this product. Udousoro et al. (2017) found phosphorus concentrations in the tested supplements ranging from 4.44 to 27 800 mg kg⁻¹, confirming that phosphorus is an element with highly variable concentrations in dietary supplements, depending primarily on the type of excipients used.

Most samples showed low aluminum concentrations (Figure 1d), except for one outlier in the CAF group. The outlier DS3 had a concentration of 63.7 mg kg⁻¹, more than five times the group average of 10.9 mg kg⁻¹. Supplements containing only creatine as the active ingredient exhibited the narrowest range of aluminum concentrations and the lowest aluminum levels.

The distribution of zinc concentrations varied considerably across the study groups, which justified presenting it in two separate graphs, one including outliers (Figure 1e) and one with outliers removed (Figure 1f), allowing for a clearer interpretation of typical values. In this study, the zinc content in the diet ranged from 1.18 to 4 400 mg kg⁻¹. Outliers were observed in the CAF (DS3, DS4) and CAF & Cr (DS22) groups, while creatine-based supplements had the lowest and most consistent zinc concentrations among all groups. After removing outliers (Figure 1f), zinc concentrations in the remaining samples fell within a narrow range of approximately 1 to 42 mg kg⁻¹, with the Cr group exhibiting the narrowest range in the middle half of the data, indicating a less complex matrix in the creatine supplements. The literature indicates that zinc concentrations in dietary supplements vary. Filipiak-Szok et al. (2015) reported zinc concentrations ranging from 18.65 to 66.57 mg kg⁻¹, which is a narrower range than found in the present study. Marrero et al. (2013) reported the highest zinc concentrations in the group of multivitamin supplements stating zinc was present as gluconate or oxide, ranging from 5 133 to 9 307 mg kg⁻¹, while other product groups contained between 1 and 104 mg kg⁻¹. Salles et al. (2017) found the zinc concentration was as high as approximately 5 000 mg kg⁻¹ for the selected multiminerall supplement, whereas for the other groups, the zinc concentration did not exceed 160 mg kg⁻¹. Even lower zinc concentrations were observed in marine-based supplements derived from krill, shark cartilage, algae, and corals, with zinc levels of 50 mg kg⁻¹, 37 mg kg⁻¹, 10 mg kg⁻¹, and 1 mg kg⁻¹ respectively (Leblond et al. 2008). An analysis of herbal weight-loss products revealed zinc concentrations ranging from 0 to 119.1 mg kg⁻¹, with the lowest non-zero value recorded at 0.9 mg kg⁻¹. The zinc content for the individual plants comprising the DS was as follows: *Ilex paraguariensis* 3.76-33.26 mg kg⁻¹, *Cordia ecalyculata* 2.08-12.43 mg kg⁻¹, *Senna alexandrina*

1.88-16.13 mg kg⁻¹, *Citrus aurantium* 0-119.1 mg kg⁻¹, *Cissus quadrangularis* 0.9-16.23 mg kg⁻¹, *Camellia sinensis* 4.28-34.66 mg kg⁻¹ (Barrella et al. 2016). In a study of supplements available in Romania, Amarei et al. (2017) found zinc levels ranging from 5 to 573.63 mg kg⁻¹, with none of the samples exceeding the maximum allowable level. The zinc concentrations reported by Avula et al. (2010) ranged from 4.89 to 62 mg kg⁻¹, except for one sample, which had a concentration of 113 mg kg⁻¹. In a study of supplements containing green tea extract, Oško et al. (2022) reported zinc concentrations ranging from 0.48 to 31.5 mg kg⁻¹ for most samples, with one supplement showing a concentration of 4 465 mg kg⁻¹, which is similar to the outliers observed in this study. Jasińska-Balwierz et al. (2025) in a study of dietary supplements containing adaptogenic plants found zinc concentrations ranging from 2.388 to 31.192 mg kg⁻¹, and the maximum single-dose intake for zinc was not exceeded in any of the supplements tested. Both in this study and in the literature, variations in zinc content in supplements are common. The highest levels were found in products that listing zinc as an active ingredient or containing complex formulations, whereas supplements based on a single active ingredient, such as creatine, contain significantly lower concentrations of this element.

Compliance of the experimental formulation with the manufacturer's declaration

The consistency of the experimental results with the values declared by manufacturers on the labels was assessed. Of the 26 supplements examined, only some manufacturers included quantitative information on the content of selected minerals on the label. For most elements (Ca, Mg, P, Zn), the quantitative content was declared by two or three manufacturers. Aluminum was not listed on the label of any of the products tested. Such a limited scope of quantitative declarations makes it difficult to fully assess the accuracy of the ingredient list. The data are presented as bar charts, showing the average value for all samples and the values listed on the product label, if provided by the manufacturer.

Calcium had the highest concentration levels among all the elements tested. Salts of this metal are most commonly used as fillers in the food products examined.

The presence of calcium in DS6, DS8, DS21, DS22, and DS24 was consistent with the manufacturers' declaration on the label (Figure 2). For DS21 and DS22, where the manufacturer provided information on the calcium content, the experimentally determined value did not differ significantly from the content stated on the label; for DS21, the experimental calcium value was 90.51% of the label value, while for DS22, the experimental value exceeded the declared value by approximately 20.07%. A relatively high calcium content was also found in DS24, whose label does not include any information regarding the presence of this element. On the DS23 label, the manu-

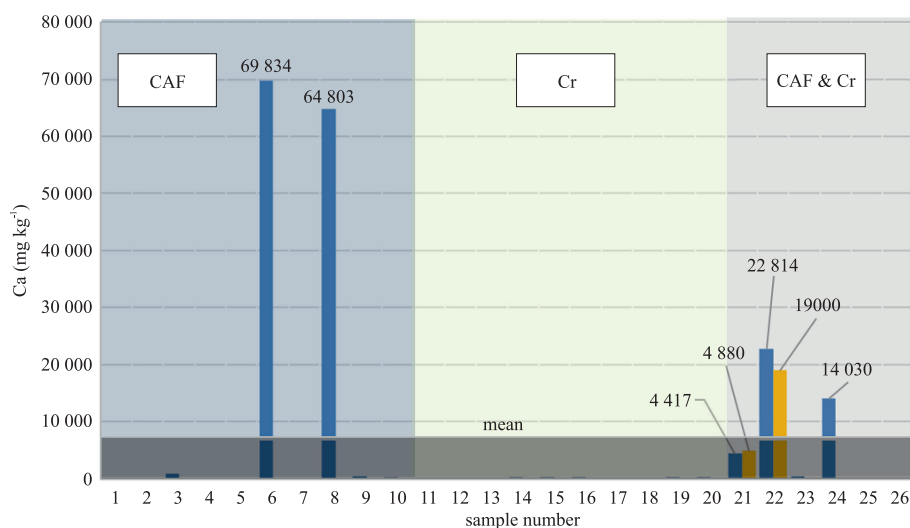


Fig. 2. Calcium concentration (mg kg^{-1}) in the dietary supplements tested ($n=26$): experimental values (ICP-OES, blue bars) and values declared by the manufacturer (yellow bars). Declared values are provided only for samples with a quantitative declaration available

facturer indicated the presence of calcium in the form of calcium D-pantothenate; analysis of the experimental results confirmed its presence in small amounts. A study by Maćkiewicz et al. (2025) showed that all samples analyzed contained higher calcium concentrations than indicated on the label. The measured values ranged from 148.8% to 353.3% of the declared amount. Magnesium was the element for which the greatest discrepancies were observed between experimental and declared values. In all three cases where the manufacturer provided the quantitative magnesium content, the value measured by the ICP-OES method was lower than the declared value (Figure 3).

The experimentally determined magnesium concentration for sample DS22 was 65.27% of the declared value, while for supplement DS21, the measured magnesium concentration was only 16.33% of the declared value. DS3 showed the greatest discrepancy between the experimental concentration and the label value, with the measured value amounting to only 7.9% of the value stated on the label. In the study by Udousoro et al. (2017), out of six samples with a quantitative declaration, the experimental magnesium content was less than 10% of the declared value in four samples, while one sample exceeded the declared value by more than double. Maćkiewicz et al. (2025) demonstrated that in dietary supplements intended for pregnant women, the actual magnesium content ranged from 247.6% to 360.8% of the declared value. It is worth noting that 12 out of 26 manufacturers listed only the presence of magnesium on the label, without specifying the amount, which is permissible under current regulations but may be insufficient from the consumer's perspective.

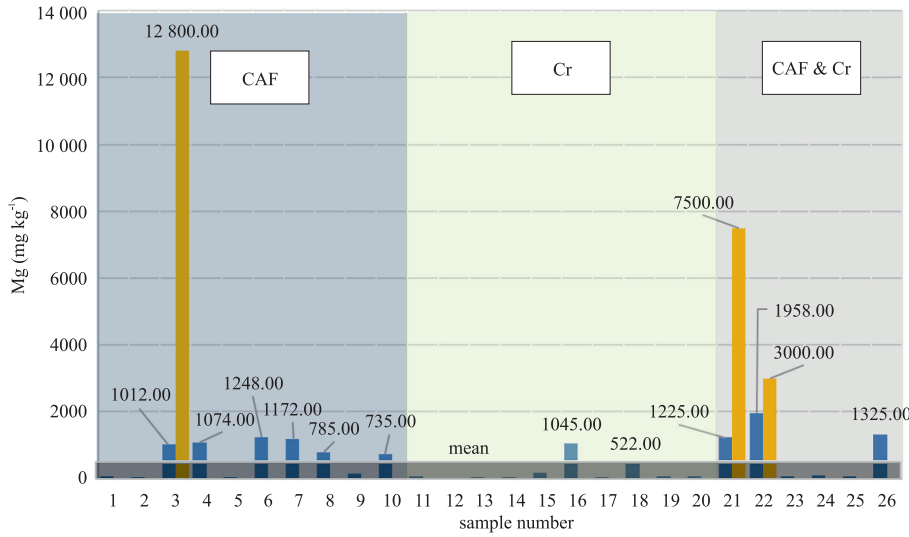


Fig. 3. Magnesium concentration (mg kg^{-1}) in the dietary supplements tested ($n=26$): experimental values (ICP-OES, blue bars) and values declared by the manufacturer (yellow bars). Declared values are provided only for samples with a quantitative declaration available

Like calcium, phosphorus was not declared as an active ingredient, but its high concentration in selected samples indicates the use of phosphates as fillers (Figure 4).

Three manufacturers (supplements DS21, DS22, and DS24) provided information on the presence of this element as phosphate on their labels.

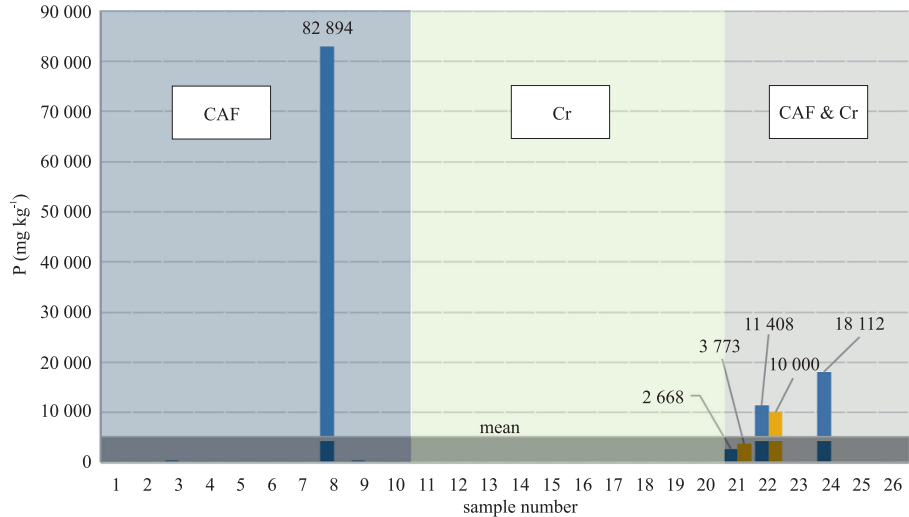


Fig. 4. Phosphorus concentration (mg kg^{-1}) in the dietary supplements tested ($n=26$): experimental values (ICP-OES, blue bars) and values declared by the manufacturer (yellow bars). Declared values are provided only for samples with a quantitative declaration available

Only DS22 explicitly listed the calcium content on its product label. The declared total phosphorus content in the DS21 supplement was calculated from the label information regarding calcium content in the form of calcium hydrogen phosphate (CaHPO_4). DS24 only qualitatively confirmed the presence of phosphate without specifying its amount. For DS22, the experimental value exceeded the declared value by 14.08%, while for DS21, the phosphorus content was only 70.71% of the value calculated from the label. The phosphorus content in sample DS8 was significant; it was the highest among the experimentally obtained values, yet the label gave no information about the presence of this element in the supplement tested. In another study, for three supplements with a quantitative declaration, the measured values ranged from 21% to 62% of the declared value, indicating that the actual phosphorus content was lower than stated on the label (Udousoro et al. 2017).

The consistency of the values for DS8, DS21, DS22, and DS24 regarding calcium and phosphorus concentrations (Figures 2 and 4) suggests the use of a filler in the form of calcium phosphate in these samples.

None of the 26 supplements tested included information about the presence of aluminum on their labels, however, this element was detectable in all samples (Figure 5).

The average aluminum concentration was 10.90 mg kg^{-1} ; however, sample DS3 stood out with a concentration of 63.7 mg kg^{-1} , which is nearly 6-fold higher than the overall average. Most common form of aluminum in food is a dye called carmine, with the food additive code E120 (Ganhör et al. 2024). None of the manufacturers provided information about the use of this

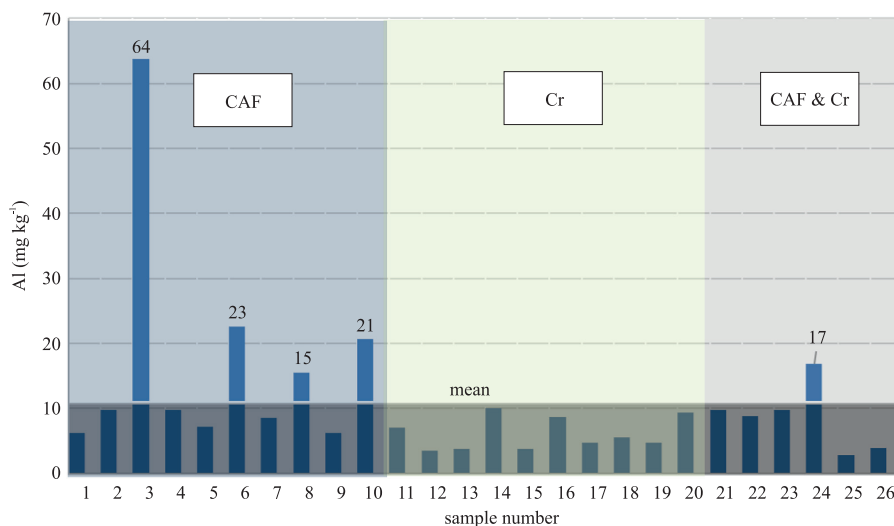


Fig. 5. Aluminum concentration (mg kg^{-1}) in the dietary supplements ($n=26$), determined by ICP-OES

additive, which can lead to accumulation of this metal without knowledge of the consumer. According to the EFSA, the tolerable weekly intake (TWI) of aluminum is 1 mg kg⁻¹ of body weight, and according to the WHO, it is 2 mg kg⁻¹ of body weight (Filippinia et al. 2019, Ganhör et al. 2024); the samples tested do not exceed these values.

Aluminum toxicity primarily depends of its physical behaviour and relative solubility in water. The toxicity of water soluble form such as Al³⁺ comes from formation of radicals capable of depleting mitochondrial Fe and promoting generation of H₂O₂, and oxygen radical species. This may cause oxidative damage leading to intrinsic apoptosis, and shift from oxidative ATP production to anaerobic glycolysis, resulting in reduced β -oxidation and increased peroxidation. Toxicity of insoluble Al depends on their behavior as particles. Trivalent Aluminum Al³⁺ as well as other metal ions, can bind to proteins leading to conformation changes, reduced solubility, spontaneous oligomerization, aggregation and precipitation (Willhite et al. 2014).

The zinc content, a trace mineral, was notably elevated in only three of the 26 supplements tested (Figure 6).

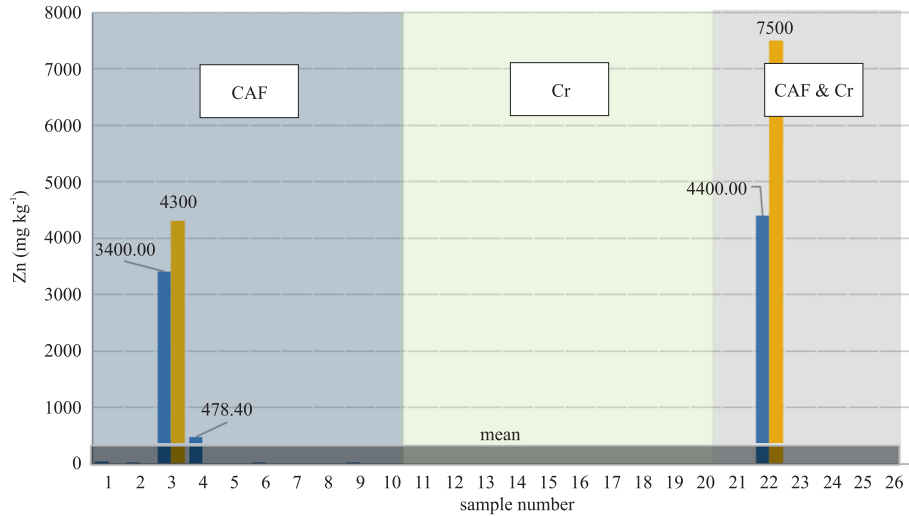


Fig. 6. Zinc concentration (mg kg⁻¹) in the dietary supplements tested ($n=26$): experimental values (ICP-OES, blue bars) and values declared by the manufacturer (yellow bars). Declared values are provided only for samples with a quantitative declaration available

Information regarding zinc content was provided on the label of 2 out of the 26 supplements tested. These samples contained significant amounts of this element. DS3 declared 4 300 mg kg⁻¹ of zinc, while DS22 declared 7 500 mg kg⁻¹. In both cases, the experimental values were lower than the declared values; for DS3, the detected concentration was 79.07% of the label value, while for DS22 it was 58.66% of the declared value. Although both products contain less zinc than stated on the label, their concentrations are

high enough that daily exposure to zinc when these supplements are used as directed by the manufacturer exceeds current safety standards. It is worth noting that DS4 had a higher zinc content than the others, yet its label did not include any information about the presence of this element. Consumers are therefore unaware that they are consuming zinc, and the daily intake, in this case 478 mg kg⁻¹ of product, may significantly supplement zinc intake from other sources. The findings of this study contribute to the broader picture of inconsistencies in zinc declarations documented in the literature. Van der Voet et al. (2008) reported high zinc concentrations in a product without any label declaration in a study of supplements using the ICP-OES method, noting zinc concentrations as high as 2 000 mg kg⁻¹ with no information on the label, while another sample contained as much as 2 400 mg kg⁻¹.

In the study by Maćkiewicz et al. (2025), only two of the samples analyzed contained less zinc than indicated by the manufacturer on the label, while the remaining samples showed zinc levels ranging from 101.9% to 299.1% of the declared value. Additionally, one sample tested, which did not declare the presence of zinc in its product, was found to contain a significant concentration of this element. Zinc supplements analyzed by Puścion-Jakubik et al. (2026) using AAS showed that the zinc content was within a tolerable range of -20% to +45% of the declared value for 70% of the supplements tested. Only 6.25% of the remaining samples contained less than 20% of the declared zinc content, while 23.75% contained more than 45% of that value. One sample tested exceeded the upper limit (UL) of 25 mg per day. The results of the study by Soriano et al. (2007) differed; the concentrations of zinc, copper, iron, and manganese in multivitamins/multimineral, as determined by F-AAS, matched the values declared by the manufacturers, indicating that label compliance is achievable. Zinc concentrations in the study by Salles et al. (2017) were similar in the group of multimineral-based supplements, showing a high degree of compliance with the declared values. In a study that also used the ICP-OES method, the zinc content in the tested supplements was determined. The highest values were found in the multivitamin group, where this element was present as gluconate or oxide. These values ranged from 5 133 mg kg⁻¹ to 9 307 mg kg⁻¹. The other groups studied showed low concentrations of this element, ranging from 1 to 104 mg kg⁻¹ (Marrero et al. 2013). The other supplements analyzed did not exceed the permissible limits set by the aforementioned regulatory authorities for any of the elements tested.

Assessment of daily zinc exposure and health implications

The experimental results were analyzed in terms of daily consumer exposure to the elements being measured, using the manufacturer's recommended daily dose of each supplement as the basis for calculation. This approach enabled determination of actual daily metal exposure during regular use of the product and comparison with applicable safety standards. Of all the

elements tested, only zinc exceeded the recommended daily intake (RDI) of 15 mg in two of the 26 products examined, as established by the Dietary Supplements Working Group of the Sanitary and Epidemiological Council of the Chief Sanitary Inspectorate (Resolution No. 10/2019). The other supplements analyzed did not exceed the permissible limits set by the aforementioned regulatory authorities for any of the elements tested. DS3 showed a daily zinc exposure equivalent to 3.4 times the RDI, while DS22 showed exposure equivalent to 5.9 times the RDI. Both products are powdered supplements, which may be relevant when assessing the risk of unintentionally taking too large a dose, as the powdered form tends to result in greater variability in the measured dose compared to capsules or tablets with a strictly defined unit weight. Importantly, both samples belong to different groups (CAF or CAF & Cr), indicating that the risk of exceeding the RDI for zinc is not limited to a single category of sports supplements, but may apply to products with varying active ingredients. One of the supplement samples in the aforementioned study by Udousoro et al. (2017) also showed a concentration above the acceptable limit.

Zinc is an essential trace element that plays a crucial role in the proper functioning of the body; it acts as a cofactor in numerous enzymatic reactions, regulates gene expression, and influences metabolic and immune processes. However, when safe intake levels are consistently exceeded, it becomes toxic. Excessive zinc intake can cause neutropenia, and secondary copper deficiency, and clinical side effects have been documented in both experimental studies and clinical observations (Schoofs et al. 2024). High zinc levels are rare, but they can cause anaemia (Jeng, Chen 2022). Studies confirm that zinc has an antagonistic effect on calcium, copper, and iron levels in the body (Sandstead 1982, Spencer et al. 1982, Agget 1983, Festa et al. 1985). Copper plays a role in the production of elastin and collagen in the human body, suggesting that its low levels may contribute to vascular disorders. Excessive zinc intake may also lead to ischemic heart disease (Klevay 1984). Copper absorption in the body can decrease by approximately 27% when taking zinc supplements at a dose of about 23.3 mg per day (Burke et al. 1981). These values are significantly lower than the recommended daily intake for DS22, which is nearly 88 mg. When the DS22 supplement is used regularly in accordance with the manufacturer's instructions, the risk of these complications should therefore be considered a real concern, particularly among athletes, whose intense physical activity already significantly increases their metabolic demand for trace elements.

Exceeding the permissible levels of metals in dietary supplements is a global phenomenon. In Lebanon, a study of 33 supplements found that 21% of samples in the vitamins and minerals category contained levels of iron, zinc, and manganese that exceeded the maximum allowable level (Korfali et al. 2013). In Croatia, five of the 30 supplements tested contained metal concentrations above the permissible limit, with lead concentrations exceeding this limit by 120-386% (Tumir et al. 2010). In Mexico, one of the samples tested

showed a zinc concentration of $4\,785\text{ mg kg}^{-1}$, corresponding to 17.9% of the recommended daily intake as specified by the Institute of Medicine (García-Rico et al. 2007).

CONCLUSIONS

The analysis revealed discrepancies between the declared composition and the experimentally determined concentrations of elements among the analyzed supplements. Caffeine-based products (CAF and CAF & Cr) show greater heterogeneity and a higher frequency of undeclared or excessive element concentrations compared to the more uniform creatine (Cr) group. In many cases, the magnesium and zinc content was lower than stated, which may be due to inaccuracies in the manufacturing process or inconsistencies in the product's ingredients. At the same time, elevated or undeclared levels of elements typical of excipients (calcium, phosphorus, and aluminum) were detected, indicating insufficient quality control of the formulation prior to market release. Two of the tested supplements exceeded the maximum intake level for zinc, indicating that their regular use may pose a real risk of metabolic disorders, underscoring the need for stricter control of micronutrient levels in dietary supplements.

The present study was limited to the analysis of selected mineral elements and did not include quantification of the active ingredients (caffeine and creatine) themselves. Future research should combine both analyses within a single study, enabling assessment of the ratio between active ingredient content and filler-derived elements, which would provide a more complete picture of product quality and formulation transparency.

The risk of heavy metal contamination is attributed to several factors, including bioaccumulation, environmental pollution, manufacturing and traditional practices, and regulatory gaps. The study was limited to highly processed conventional products relating exclusively to the factor associated with regulatory gaps, suggesting higher concentrations of heavy metals and other contaminants in analogous plant-based products. A rigorous analysis of conventional and plant-based dietary supplements for athletes is required in order to ascertain the relationship between the levels of heavy metals and contaminants in these products, depending on their production methods.

The results of this study indicate that athletes who regularly use dietary supplements should not base their assessment of a product's safety only on the composition provided by the manufacturer, given the discrepancies found between the mineral content listed on the label and the actual content. Preference should be given to products from manufacturers who fully disclose the mineral composition and hold an independent quality certificate issued by an independent institution.

Author contributions

K.S. – writing – original draft preparation, I.M. – supervision, review & editing, A.D. – review & editing, methodology.

Conflicts of interest

The authors declare no conflicts of interest.

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