

Accurate and reliable multielement analysis of alternative protein foods using ICP-MS

Authors

Sukanya Sengupta¹, Torben Stichel², Daniel Kutscher¹

¹Thermo Fisher Scientific, Bremen, Germany

²Thermo Fisher Scientific, Dreieich, Germany

Keywords

ICP-MS, AGD, He-KED, half-mass correction, accuracy, robustness, toxic elements, nutritional elements

Goal

To demonstrate the applicability of the Thermo Scientific[™] iCAP[™] RQplus ICP-MS instrument for fast and accurate, highly sensitive, and reliable determination of nutritional and toxic trace elements in alternative protein food samples

Introduction

Plant-based, protein-rich foods and other alternatives to conventional meat and meat-based food products have long been a part of the human diet—termed here as "alternative protein foods." Historically, foods like soy and other beans, lentils, non-dairy milks like almond milk, locally available edible insects, etc. have been consumed traditionally in many parts of the world and in smaller amounts as "alternative" options elsewhere. In recent times, an exponential increase of the plant-based alternatives has been seen globally due to increasing concerns of the impact of conventional and industrial meat production on the global climate and the environment, as well as ethical concerns and the effects of over-consumption of animal-derived products on human health.

The alternative protein foods industry is expected to continue to grow as more consumers worldwide include such foods in their daily diet. Therein lies the need to assess the nutritional content of these foods and to regulate any potential harmful components within them, as with conventional food. The Commission Regulation (EU) 2023/91512¹, for example, lays down guidelines on permitted levels of contaminants,

thermo scientific

including toxic metals, in many different foods including some alternative options. For example, the prescribed limits for Cd in pulses and proteins from pulses in this regulation are 0.040 mg·kg⁻¹ and 0.10 mg·kg⁻¹, respectively, and analytical techniques must be able to accurately determine such low concentration levels in relevant samples.

For a comprehensive multielement analysis of the wide variety of alternative foods, a highly sensitive technology with large linear dynamic range such as inductively coupled plasma mass spectrometry (ICP-MS) is required so low levels of toxic elements as well as high amounts of nutrients like K and Ca can be accurately quantified in the samples. In this study, an iCAP RQplus ICP-MS instrument and a Thermo Scientific[™] iSC-65 Autosampler have been used for the accurate, fast, and reliable multielement analysis of ten different alternative protein food samples.

Experimental

Instrument parameters

The iCAP RQplus ICP-MS instrument, equipped with argon gas dilution (AGD) for online dilution of samples inside the sample introduction system, was used in this study. All instrument parameters are listed in Table 1.

Table 1. Instrument parameters

Instrument parameter	Setting
Nebulizer	Borosilicate glass MicroMist [™] , 400 µL·min⁻¹
Spray chamber	2.5 mm i.d., quartz
Injector	Quartz cyclonic, cooled at 2.7 °C
Interface	Nickel sampler and skimmer cone, high matrix insert
Plasma power	1,550 W
Nebulizer gas	0.39 L·min ⁻¹
CRC conditions	4.3 mL·min ⁻¹ of He, 3 V KED
Tune setting	KED AGD Low
Additional gas	0.55 L·min ⁻¹
Scan setting	Dwell times: 0.2 s for As, Hg, Pb, Se 0.1 s for Ni 0.05 s for others 5 sweeps, 3 main runs
Lens setting	Optimized using autotune
Timer per sample	<2 min (including uptake, 3 repeats, and wash)

The analysis of 26 analytes—at major, minor, and trace level concentrations—was carried out in a single read out per sample, combining the lowest dilution level available with kinetic energy discrimination (KED) using He in the collision/reaction cell (CRC). The argon gas for the AGD was directly supplied from within the instrument to minimize manual dilutions of the samples. While the AGD Low dilution level helped minimize sample matrix effects, the He-KED mode provided comprehensive interference removal benefits. In addition, half mass correction was applied to correct for potentially occurring doubly charged interferences of lanthanide elements, often affecting the accurate determination of As and Se.

The iSC-65 Autosampler was used for sample delivery, and the Step Ahead feature was utilized for overlapping the wash of the autosampler probe and capillary with the acquisition of the previous sample, thereby shortening the overall run time per sample. The total run time per sample analysis in this study, including three repeat acquisitions, uptake, and wash times, was only 1 min 58 s without the use of an additional sampling valve.

Samples

A total of ten different alternative proteins food samples were analyzed in this study. Seven from categories 1 to 3 were proteinrich alternatives to conventional meat-based foods (alternative meat). The two samples in category 4 were alternatives to conventional dairy milk. The sample in category 5 was an edibleinsect-based, protein-rich alternative food that is increasingly being considered a part of sustainable food sources for human beings and is therefore also regulated in certain countries, for example regulated as "novel foods" in the EU region².

Table 2. Sample types included in the current study

Sample category	Samples
1. Plant-based unprocessed	Pulses, quinoa
2. Plant-based processed	Tofu, gram flour
3. Plant-based meat analogues	Meat replacement products: 1. Filet type, wheat protein based 2. Cold cut, wheat protein based 3. Cold cut, pea protein based
4. Milk alternatives	Oat milk, almond milk (liquid samples)
5. Novel foods	Insect energy bar

Reference materials

Non-traditional protein-rich foods, such as meat replacement products or plant-based meat analogs, are still new in the food market. Thus, suitable standard reference materials (SRM) are not available yet. Since plant components and extracts/concentrates are major constituents of such products, plant leaves standard reference materials, namely, NIST[™] SRM[™] 1515 (Apple Leaves) and SRM 1547 (Peach Leaves), were analyzed in this study. Additionally, SRM 1568b (Rice Flour) was included in the study to include a grain-based SRM.

Sample preparation

The food samples and SRMs were prepared by closed vessel microwave digestion in a Milestone Ethos[™] microwave digestion system (Milestone SK-10). For each sample, homogenized aliquots of 0.5 ± 0.05 g (dry samples) or 2 ± 0.2 g (wet samples) were accurately weighed and mixed with 3 mL HNO₃, 1 mL HCl (67 and 35-37 weight %, Optima[™] grade, Fisher Scientific[™]), 3 or 1 mL (for dry or wet sample, respectively) of 18 MΩ ultrapure water, and 200 µg·L¹ Au (prepared from 1,000 mg·L¹ single element solution, SPEX CertiPrep[™], Metuchen, NJ, USA). After digestion and cooling, the sample solutions were quantitatively recovered and made up to a final volume of 50 mL with ultra-pure water.

Standards

All calibration standards were prepared using single element standards (SPEX CertiPrep, Metuchen, NJ, USA) in an acid matrix of 2% HNO₃ (v/v) and 1% HCl (v/v). The elements were grouped according to the expected concentrations in regular food matrices (Table 3). 200 μ g·L⁻¹ Au was included in all standards as a mercury stabilizer. As an internal standard solution, 1 mg·L⁻¹ Sc, 20 μ g·L⁻¹ In, TI in 2% HNO₃ (v/v), and 1% HCl (v/v) were added online via a T-piece. The calibration standard Std 5 was used as a quality control standard (QC) for regularly checking the data quality during an uninterrupted analysis of a large number of samples.

Software

The Thermo Scientific[™] Qtegra[™] Intelligent Scientific Data Solution[™] (ISDS) Software was used for all steps in the workflow, starting from instrument tuning using the readily available AGD tune sets, to setting up measurement sequences, data acquisition, and evaluation. The Instrument Health Monitoring tool within the software provides an overview of the current instrument condition and tracking of the daily performance of the instrument, thus ensuring that every measurement sequence is successful and there is no need for sample re-runs. The Thermo Scientific[™] Hawk[™] Consumables and Maintenance Assistant, also fully integrated within the main software, can be used for consumables and spare parts management to minimize instrument downtime—a tool especially useful for laboratories facing a high sample volume.

Results and discussion

Linearity and sensitivity

A wide analytical range was required to cover relevant concentration levels for both toxic as well as nutritional elements, starting from 0.005 µg·kg⁻¹ for Hg up to 600,000 µg·kg⁻¹ for Na. All elements were calibrated at different levels within these ranges as shown in Table 3. The difference between the minimum and maximum concentrations covered for each element was 200x. The selected concentration ranges were set to accommodate the expected concentrations of the elements in different food samples and in the chosen reference materials.

Excellent sensitivity was obtained using the developed method and AGD-KED settings. The instrument detection limit (IDL) based on repeat measurements of the blank, background equivalent concentration (BEC), and coefficient of determination (R²) are readily available in the Qtegra ISDS Software. The IDL and method limit of quantification (MLOQ) values are all well below the limits typically required by official regulations for different food matrices and are listed in Table 4.

	Concentrations (blue in mg·L ⁻¹ , the rest in μ g·L ⁻¹)						
Elements	Std 1	Std 2	Std 3	Std 4	Std 5	Std 6	
Hg	0.005	0.01	0.05	0.1	0.5	1	
As, Cd, Ce, Co, Cr, Li, Mo, Ni, Pb, Sb, Se, Sn	0.05	0.1	0.5	1	5	10	
B, Be, Cu, Fe, Zn, Sr	5	10	50	100	500	1,000	
AI	25	50	250	500	2500	5,000	
P, Mg, Mn	1	2	10	20	100	200	
Ca, K	1.5	3	15	30	150	300	
Na	3	6	30	60	300	600	

Table 3. List of calibration standards (IS) and their concentrations

Table 4. List of target analytes and sensitivity expressed as limits of detections (IDL) and method limits of quantification (MLOQ) for individual elements in unprocessed samples. MLOQ is calculated as dilution factor (DF) $\times 3 \times IDL$. The values in blue are in mg·L¹ and other values are in μ g·L¹.

Analyte	R ²	BEC (µg·L⁻¹)	IDL (μg·L⁻¹)	MLOQ for liquid samples (µg·L⁻¹) DF = 25	MLOQ for solid samples (μg·kg⁻¹) DF = 100
⁷ Li	0.9996	0.056	0.398	29.88	119.52
⁰Be	0.9997	<0.00001	0.0001	0.01	0.03
¹¹ B	>0.9999	0.017	0.0003	0.02	0.09
²³ Na	0.9998	0.039	0.004	0.31	1.25
²⁴ Mg	0.9998	0.001	0.0007	0.05	0.21
²⁷ AI	0.9998	0.0003	0.0005	0.04	0.15
³¹ P	0.9998	0.011	0.003	0.22	0.87
³⁹ K	0.9999	0.055	0.005	0.39	1.56
⁴⁴ Ca	0.9999	0.011	0.004	0.31	1.23
⁵² Cr	0.9999	0.127	0.009	0.68	2.70
⁵⁵ Mn	0.9997	0.003	0.0002	0.01	0.05
⁵⁷ Fe	>0.9999	0.0002	0.0004	0.03	0.12
⁵⁹ Co	0.9997	0.0008	0.002	0.15	0.60
⁶⁰ Ni	0.9998	0.014	0.015	1.13	4.50
⁶⁵ Cu	0.9999	0.008	0.003	0.20	0.78
⁶⁶ Zn	0.9993	0.001	0.0003	0.02	0.09
⁷⁵ As	0.9999	0.024	0.006	0.45	1.80
⁷⁸ Se	0.9992	0.162	0.062	4.65	18.60
⁸⁸ Sr	>0.9999	0.025	0.0003	0.02	0.09
⁹⁵ Mo	0.9999	0.003	0.005	0.38	1.50
¹¹¹ Cd	0.9997	0.001	0.003	0.23	0.90
¹¹⁸ Sn	>0.9999	0.006	0.009	0.68	2.70
¹²¹ Sb	0.9996	0.007	0.003	0.23	0.90
¹⁴⁰ Ce	>0.9999	0.0002	0.001	0.11	0.42
²⁰² Hg	0.9993	0.013	0.001	0.06	0.25
²⁰⁸ Pb	0.9996	0.002	0.001	0.08	0.30

Accuracy

The three SRMs analyzed in this study serve as an accuracy check for the different elements that are present at certified concentrations in these reference materials. The recoveries for the certified elements within the Rice Flour SRM were between 82.3% and 109.0% (Table 5). For Apple Leaves SRM 1515 and Peach Leaves SRM 1547, the recoveries were tightly constrained between 87% and 103% for the different analytes, which validates the method accuracy for these elements. However, the concentrations recoveries for As and Se, after applying a mathematical correction for interferences, were slightly elevated at 112-113% in the Peach Leaves SRM 1547 (Table 5). For the Apple Leaves SRM 1515, based on previously published concentration values in the SRM certificate, the recoveries for As are around 115–120%, whereas for Se the recoveries are higher/ more varied and outside of the typically acceptable accuracy range of 75–125%³ (marked with * in Table 5).

Both these SRMs contain rare earth elements (REE) in the mg·L⁻¹ range. Upon ionization inside the ICP-MS, these REEs produce doubly charged species that can interfere with certain target analytes, depending on the rate of formation of such species, the isotopic abundances of the particular REEs, and their mass/ charge ratios (m/z). For example, the doubly charged species $^{\rm 150++}{\rm Nd}$ and $^{\rm 150++}{\rm Sm}$ have the same m/z as $^{\rm 75}{\rm As},$ and their presence leads to inaccurately high values for arsenic. Similarly, ¹⁵⁶⁺⁺Gd interferes with and yields inaccurate data for ⁷⁸Se. Such interferences can be resolved and corrected, to a certain extent, in a single quadrupole ICP-MS by using mathematical corrections based on the natural abundances of these REE isotopes. For the correction, an interference-free doubly charged species of a stable isotope of the REE, for example, ¹⁴⁵⁺⁺Nd, i.e., ^{72.5}Nd, is measured and used to estimate the contribution of the interfering doubly charged species on the target m/z. However, the accuracy of such mathematical corrections is limited, suffering from a significant mass-bias, and decreasing with increasing concentration levels of those particular REEs in the samples.

Table 5. Certified values, mean recoveries, and associated standard deviations for the different analytes in the standard reference materials (SRM)

	Apple Leaves	SRM 1515	Peach Leaves	SRM 1547	Rice Flour SRM 1568b		
Element	Certified value (mg·L ⁻¹)	Recovery (%)	Certified value (mg·L ⁻¹)	Recovery (%)	Certified value (mg·L⁻¹)	Recovery (%)	
¹¹ B	27.6 ± 2.8	97.6 ± 3	28.73 ± 0.81	103.5 ± 4	-	-	
²⁴ Mg	2710 ± 120	94.5 ± 3	4320 ± 150	102.7 ± 3	559 ± 10	98.3 ± 3	
²⁷ AI	284.5 ± 5.8	92.0 ± 4	248.9 ± 6.5	98.0 ± 6	4.21 ± 0.34	96.0 ± 8	
³¹ P	1593 ± 68	91.9 ± 3	1371 ± 82	103.3 ± 4	1530 ± 40	84.0 ± 5	
³⁹ K	16080 ± 210	94.1 ± 1	24330 ± 380	101.8 ± 6	1282 ± 11	87.9 ± 5	
⁴⁴ Ca	15250 ± 100	93.7 ± 6	15590 ± 160	100.0 ± 6	118.4 ± 3.1	101.6 ± 1	
⁵⁵ Mn	54.1 ± 1.1	89.7 ± 3	97.8 ± 1.8	95.6 ± 7	19.2 ± 1.8	84.7 ± 3	
⁵⁷ Fe	82.7 ± 2.6	86.9 ± 1	219.8 ± 6.8	94.9 ± 4	7.42 ± 0.44	109.0 ± 4	
⁶⁰ Ni	0.936 ± 0.094	90.6 ± 3	0.689 ± 0.095	97.9 ± 8	-	-	
⁶⁵ Cu	5.69 ± 0.13	99.8 ± 3	3.75 ± 0.37	95.7 ± 6	2.35 ± 0.16	86.9 ± 3	
⁶⁶ Zn	12.45 ± 0.43	100.6 ± 4	17.97 ± 0.53	95.9 ± 5	19.42 ± 0.26		
⁷⁵ As	-	-	0.062 ± 0.014	112.8 ± 7*	0.285 ± 0.014	94.8 ± 7	
⁷⁸ Se	-	-	0.120 ± 0.017	111.7 ± 6*	0.365 ± 0.029	99.2 ± 3	
⁸⁸ Sr	25.1 ± 1.1	92.0 ± 5	53.0 ± 5.0	93.9 ± 4	-	-	
⁹⁵ Mo	0.095 ± 0.011	95.1 ± 8	0.0603 ± 0.0068	87.0 ± 5	1.451 ± 0.048	89.0 ± 1	
¹¹¹ Cd	0.0132 ± 0.0015	94.6 ± 4	0.0261 ± 0.0022	94.2 ± 3	0.0224 ± 0.0013	82.3 ± 2	
118 Sn	-	-	-	-	107.8 ±	107.8 ± 5	
²⁰² Hg	0.0432 ± 0.0023	91.9 ± 3	0.0317 ± 0.0043	96.8 ± 5	0.00591 ± 0.00036	88.5 ± 5	
²⁰⁸ Pb	0.470 ± 0.024	87.1 ± 3	0.869 ± 0.018	97.7 ± 2	-	-	

*See text for explanation

The concentrations of the REEs that caused interferences on As and Se in the SRM 1547 (Peach Leaves) are within the range where the mathematical corrections can be applied to achieve reasonable accuracy (1 mg·L⁻¹ each of Gd and Sm; 7 mg·L⁻¹ of Nd). For the SRM 1515 (Apple Leaves), the higher REE concentrations make it more challenging to obtain accurate data. While it is possible to correct for the interference on As (from 17 mg·L⁻¹ Nd and 3 mg·L⁻¹ Sm), the accuracy decreases yielding recoveries in the range of 115–120% (for a target concentration of 0.038 mg·L⁻¹ of As). For Se in the same SRM, the interferant concentration (3 mg·L⁻¹ of Gd) relative to analyte concentration (0.05 mg·L⁻¹) is too high to successfully correct and obtain accurate data. For further details and proof data, the reader is referred to a previously published technical note.⁴

One of the major elements commonly found in high amounts in conventional meat cold cuts is Na. The meat replacement products in the market also tend to have high Na contents.^{5,6} The measured CRMs do not contain certified values of the element, so a pooled sample was spiked with 10 mg·L⁻¹ of Na and the recovery obtained was 103.1 \pm 1.0% on replicate analysis of the sample.

Robustness

Long measurement sequences were run on two different days including more than 250 unknown samples, reference materials, and quality control (QC) standards each day. A single calibration block was run at the beginning of each sequence; this was sufficient for accurate data acquisition throughout the run and all sequences showed excellent stability. The calibration block was followed by the unknown samples and reference materials and QC standards (Std 5) that were regularly run after every ten unknown samples. Stable internal standard recovery was seen throughout the sequences as seen in Figure 1, demonstrating the efficiency of AGD in minimizing matrix effects on long term data quality. The internal standard recovery values remained between 85% and 116% for all samples. The QC recoveries also remained well within the 80–120% envelope throughout the sequences (Figure 2), demonstrating excellent data accuracy.



Figure 1. Stable response of the internal standards Sc, Rh, In, and TI of 85-116% during >8 h robustness test



Figure 2. Accuracy of quality control standards (Continuing Calibration Verification, CCV) spanning >8 h of continuous measurements

Sample analysis

The ten samples analyzed in this study were found to contain variable levels of nutrient elements and relatively low levels of toxic elements (Table 6, Figures 3 and 4). The meat replacement products showed elevated amounts of Na, 6,209–8,802 mg·kg⁻¹, similar to the trend shown by results in other studies on alternative protein foods as well as conventional meat cold cuts.^{5,6}

Other nutrients found in high amounts in the different food samples were K and P in pulses, gram flour, quinoa, and insect bar, and Ca in tofu and insect bar. The toxic metals were generally at low levels in the final products with As and Hg being <0.006 mg·kg⁻¹, Pb <0.015 mg·kg⁻¹, and only Cd being detected in higher amounts in some samples, for example, 0.067 mg·kg⁻¹ in quinoa.

Table 6. Multielement composition of the samples measured in this study. The concentrations in black are in µg·kg⁻¹ and those in blue are in mg·kg⁻¹.

	Concentrations (µg·kg ⁻¹ or mg·kg ⁻¹)									
	Plant-	Plant-based, Plant-based			Milk		Novel			
	unpro	cessed	pro	cessed	meat replacement products			alternatives foods		
Elements	Pulses	Quinoa	Tofu	Gram flour	1 (Wheat protein)	2 (Wheat protein)	3 (Pea protein)	Oat milk	Almond milk	Insect bar
⁷ Li	<dl< th=""><th><dl< th=""><th>64.5</th><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	<dl< th=""><th>64.5</th><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	64.5	<dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""></dl<></th></dl<>	<dl< th=""></dl<>
⁰Be	<dl< th=""><th><dl< th=""><th>0.0</th><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	<dl< th=""><th>0.0</th><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	0.0	<dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""></dl<></th></dl<>	<dl< th=""></dl<>
¹¹ B	6.7	10.1	2.4	8.1	0.7	1.3	0.7	0.6	0.5	8.4
²³ Na	35.2	228	81	126	6682	8802	6209	407	432	266
²⁴ Mg	845.0	1851	487	1140	118	192	144	76	94	1615
²⁷ AI	15.2	4.5	13.3	2.8	2.8	6.4	6.6	0.2	0.8	36.0
³¹ P	2889	3775	1787	2787	437	559	574	265	176	2980
³⁹ K	15318	6287	1724	9061	1378	2191	2473	692	438	3431
⁴⁴ Ca	470	486	3303	451	71	277	275	72	169	4442
⁵² Cr	239.4	26.6	340.8	89.1	31.0	40.4	53.7	<dl< th=""><th>5.2</th><th>132</th></dl<>	5.2	132
⁵⁵ Mn	9.3	47.4	18.4	28.6	1.5	12.6	1.3	1.2	0.5	14.4
⁵⁷ Fe	38.3	33.0	21.9	38.9	4.1	5.6	11.4	1.7	1.3	39.2
⁵⁹ Co	380.4	93.7	369.2	477.4	<dl< th=""><th>7.2</th><th>30.9</th><th>3.8</th><th>18.2</th><th>95.9</th></dl<>	7.2	30.9	3.8	18.2	95.9
⁶⁰ Ni	1028	987	173	5698	277	359	86	187	106	580
⁶⁵ Cu	6337	5096	3619	6439	1015	907	551	549	328	9640
⁶⁶ Zn	34.7	34.4	16.0	28.8	3.4	4.5	5.2	1.9	1.3	46.8
⁷⁵ As	<dl< th=""><th>6.2</th><th>1.5</th><th>5.0</th><th>2.5</th><th>2.6</th><th>4.0</th><th><dl< th=""><th><dl< th=""><th>4.1</th></dl<></th></dl<></th></dl<>	6.2	1.5	5.0	2.5	2.6	4.0	<dl< th=""><th><dl< th=""><th>4.1</th></dl<></th></dl<>	<dl< th=""><th>4.1</th></dl<>	4.1
⁷⁸ Se	15.8	11.7	8.7	264.2	<dl< th=""><th>1.9</th><th>9.8</th><th><dl< th=""><th><dl< th=""><th>85.0</th></dl<></th></dl<></th></dl<>	1.9	9.8	<dl< th=""><th><dl< th=""><th>85.0</th></dl<></th></dl<>	<dl< th=""><th>85.0</th></dl<>	85.0
⁸⁸ Sr	1.2	2.8	11.4	6.5	0.5	1.0	1.1	0.1	1.4	21.7
⁹⁵ Mo	195.2	136.5	108.7	1242.9	161.3	<dl< th=""><th>52.3</th><th>215.4</th><th>4.9</th><th>458.5</th></dl<>	52.3	215.4	4.9	458.5
¹¹¹ Cd	12.1	67.4	4.1	<dl< th=""><th>19.3</th><th>21.1</th><th>7.0</th><th></th><th><dl< th=""><th>9.0</th></dl<></th></dl<>	19.3	21.1	7.0		<dl< th=""><th>9.0</th></dl<>	9.0
¹¹⁸ Sn	37.2	3.7	38.0	3.1	27.0	14.2	7.7	9.4	<dl< th=""><th>5.3</th></dl<>	5.3
¹²¹ Sb	1.2	<dl< th=""><th>2.0</th><th><dl< th=""><th>1.2</th><th>17.5</th><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	2.0	<dl< th=""><th>1.2</th><th>17.5</th><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	1.2	17.5	<dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""></dl<></th></dl<>	<dl< th=""></dl<>
¹⁴⁰ Ce	3.3	<dl< th=""><th>4.2</th><th><dl< th=""><th><dl< th=""><th>6.5</th><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	4.2	<dl< th=""><th><dl< th=""><th>6.5</th><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	<dl< th=""><th>6.5</th><th><dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	6.5	<dl< th=""><th><dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""><th><dl< th=""></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""></dl<></th></dl<>	<dl< th=""></dl<>
²⁰² Hg	<dl< th=""><th>0.6</th><th>0.2</th><th>0.3</th><th><dl< th=""><th>0.1</th><th><dl< th=""><th><dl< th=""><th><dl< th=""><th>0.4</th></dl<></th></dl<></th></dl<></th></dl<></th></dl<>	0.6	0.2	0.3	<dl< th=""><th>0.1</th><th><dl< th=""><th><dl< th=""><th><dl< th=""><th>0.4</th></dl<></th></dl<></th></dl<></th></dl<>	0.1	<dl< th=""><th><dl< th=""><th><dl< th=""><th>0.4</th></dl<></th></dl<></th></dl<>	<dl< th=""><th><dl< th=""><th>0.4</th></dl<></th></dl<>	<dl< th=""><th>0.4</th></dl<>	0.4
²⁰⁸ Pb	3.4	13.1	14.7	0.7	5.0	12.4	4.1	<dl< th=""><th><dl< th=""><th>10.1</th></dl<></th></dl<>	<dl< th=""><th>10.1</th></dl<>	10.1



Figure 3. Major nutrient elements concentrations in representative alternative protein food samples from this study



Figure 4. Toxic elements within the analyzed samples

Conclusions

The application note presents a method for the multielement analysis of alternative protein foods with sensitivity, accuracy, robustness, and speed using the iCAP RQplus ICP-MS and iSC-65 Autosampler.

- This study proposes an ICP-MS method with integrated AGD for the accurate quantitation of 26 different nutritional and toxic elements in a variety of alternative protein food samples including plant-based, protein-rich foods, meat analogs, non-dairy milks, and edible insect bars.
- Excellent sensitivity and a wide analytical range were achieved for both high concentration as well as trace level analytes within the same method and using a single dilution level.
- The method accuracy was validated using SRMs that yielded very good recoveries on the certified concentrations of the different analytes, and the capability of the He-KED mode in removing some challenging interferences was demonstrated.
- The approach of half mass correction was evaluated and allowed reasonable correction results for As in both SRMs evaluated, whereas correction for Se was found to be more critical and matrix dependent.
- The use of the AGD feature resulted in stable data over two uninterrupted sequences of over 8 hours each, including

more than 250 samples with stable internal standard recovery 85–116% and quality control standards concentration recoveries well within 80–120%.

• The method developed is not only robust and highly sensitive but also simple and fast with a sample run time of less than two minutes per sample using a single analysis mode of the iCAP RQplus ICP-MS and the Step Ahead feature of the iSC-65 Autosampler, making this ideal for an analytical laboratory setting.

References

- 1. COMMISSION REGULATION (EU) 2023/915 of 25 April 2023 on maximum levels for certain contaminants in food and repealing Regulation (EC) No 1881/2006.
- REGULATION (EU) 2015/2283 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 25 November 2015 on novel foods, amending Regulation (EU) No 1169/2011 of the European Parliament and of the Council and repealing Regulation (EC) No 258/97 of the European Parliament and of the Council and Commission Regulation (EC) No 1852/2001.
- AOAC Official Method 2015.01 Heavy Metals in Food Inductively Coupled Plasma–Mass Spectrometry First Action 2015.
- Complete and comprehensive interference removal in ICP-MS: Can this be achieved? Thermo Scientific Smart Note 000214. https://assets.thermofisher.com/TFS-Assets/ CMD/Specification-Sheets/sn-000214-tea-iCAP-RQ-ICP-MS-food-interferenceremoval-sn000214-na-en.pdf
- Ložnjak Švarc, P.; et al. Nutrient content in plant-based protein products intended for food composition databases. *Journal of Food Composition and Analysis* 2022, *106*, 10433210.
- FRIDA, 2019. Danish Food Composition Database. Natl. Food Institute, Tech. Univ. Denmark. https://frida.fooddata.dk (accessed 6.16.21).



Learn more at thermofisher.com/icp-ms

General Laboratory Equipment – Not For Diagnostic Procedures. © 2024 Thermo Fisher Scientific Inc. All rights reserved. All trademarks are the property of Thermo Fisher Scientific and its subsidiaries unless otherwise specified. MicroMist is a trademark of Glass Expansion. Ethos is a trademark of Milestone. SPEX CertiPrep is a trademark of the SPEX CertiPrep Group LLC. NIST and SRM are trademarks of National Institute of Standards and Technology. This information is presented as an example of the capabilities of Thermo Fisher Scientific products. It is not intended to encourage use of these products in any manner that might infringe the intellectual property rights of others. Specifications, terms and pricing are subject to change. Not all products are available in all countries. Please consult your local sales representative for details. **AN002822-EN 0324S**

thermo scientific