ThermoFisher
SCIENTIFIC

Food and beverage

Reliable and productive analysis of food samples using single quadrupole ICP-MS

Authors

Sukanya Sengupta[1], Rasheed Ishola[2],
Adrián Vicent Claramunt[2], Vytautas
Tamošiūnas[2], Daniel Kutscher[1]

[1]Thermo Fisher Scientific, Bremen, Germany

[2]Thermo Fisher Scientific, Vilnius, Lithuania

Keywords

ICP-MS, iCAP MSX ICP-MS, food
analysis, high sensitivity, accuracy,
robustness, toxic elements, nutritional
elements, argon gas dilution

Goal

To demonstrate robust and reliable analysis of nutritional and toxic trace elements for
more than 7,000 food samples in eight weeks, with consistent day-to-day instrument
performance

Introduction

Verification of the absence of toxic elements, such as arsenic, cadmium, mercury,
or lead, and conversely, the presence of essential nutrients in foods, is a key task for
laboratories in the Food Safety market. Food samples are subject to regular screening
for trace elements to ensure their safety for human consumption. Strict official
regulations such as EC No 2023/915[1] in the EU, GB-2762-2017[2] in China, and others
in different parts of the world specify maximum permissible limits for toxic elements in
food. These limits are based on concentration levels at which they become hazardous
if introduced into the human body. At the same time, determining the quantity of several
nutrient elements in certain foods, like milk powders and infant formula, is an important
focus area when it comes to food analysis. However, the associated task set for testing
laboratories includes not only the analysis of the composition of a given product, but
also extends to other aspects, such as verification of geographic origin using trace
elemental or isotopic fingerprints or investigation of the effect of contact of the product
with packaging materials under different conditions.

thermo scientific

Inductively coupled plasma mass spectrometry (ICP-MS) is the technique of choice for accurately measuring toxic elements, especially for analytical testing laboratories. At the same time, it also allows the analysis of major nutritional elements present in concentrations several orders of magnitude higher. However, sample matrix variability and the large sample load usually found in testing laboratories may lead to challenges like unstable internal standard recoveries outside acceptable ranges, quality control failures and drift, rapid degradation of instrument performance. If left unaddressed, these may cause unexpected downtime and interruptions to the overall laboratory productivity. This study describes how the Thermo Scientific™ iCAP™ MSX ICP-MS instrument, equipped with argon gas dilution (AGD), is able to reliably overcome these challenges and enables seamless multi-element analysis of different kinds of food samples with simplicity.

Experimental

Instrument parameters

The instrumental setup used in this study consisted of an iCAP MSX ICP-MS and a Thermo Scientific™ iSC-65 autosampler that was used for the analysis of a variety of food samples.

To demonstrate accuracy of the results, a range of certified reference materials were analyzed as part of this study. The samples and reference materials differed in food matrix compositions with varying amounts of carbohydrates, fats, proteins, and moisture contents. The content of elemental impurities in the matrices was also varied, for example, varying amounts of toxic elements and rare earth elements (REE) in the certified/standard reference materials (CRM or SRM). These were chosen to test the suitability of the iCAP MSX ICP-MS to handle different matrices with a single robust and reliable analytical method.

All samples as well as reference materials were prepared for analysis using closed vessel microwave digestion (≈0.5 g dry samples or ≈2 g wet samples, final volume of sample digest solution 50 mL), as described in detail elsewhere.[3] The digested samples were directly placed on the autosampler without any further dilution. Instead, AGD was employed to automatically dilute the samples inside the standard sample introduction system of the ICP-MS. Parameters typically used are listed in Table 1. To overcome typically occurring interferences, kinetic energy discrimination (KED) mode using helium as a collision cell gas was applied for all analytes. Combining the approach with the Step Ahead feature of the iSC-65 autosampler typically allows for a sample run time of under 2 minutes per sample, as found in a previous study.[3] The iCAP MSX ICP-MS enables the accurate and highly sensitive analysis of trace levels of critical analytes as well as high concentrations of nutrient elements contained within food samples, devoid of matrix suppression and/or enhancement.

The use of AGD allows for a series of advantages. First and foremost, signal suppression and drift during long sequences are significantly reduced, leading to immediate reporting of results and avoiding re-runs following QC failures. Secondly, AGD also leads to more consistent uptime of the instrument and can help to increase maintenance intervals. By reducing the amount of sample reaching the plasma, and subsequently the interface region to the mass spectrometer, the sample introduction system components require less cleaning and continue to deliver stable results over long time spans. For more plannable downtime, the Thermo Scientific™ HAWK™ Consumables and Maintenance Assistant, included in Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution Software, supports laboratory managers and technicians by tracking the system´s need for maintenance to be performed.

Table 1. Instrument parameters typically used in this study

Parameter	Value
Nebulizer	iCAP MX Series Nebulizer
Interface cones	Ni – tipped sample and skimmer
Spray chamber	Cyclonic quartz
Spray chamber temp. (°C)	2.7
Injector	Quartz, 2.5 mm ID
Torch	Quartz torch
Auxiliary flow (L·min^{-1})	0.8
Cool gas flow (L·min^{-1})	14
Nebulizer flow (L·min^{-1})	0.446
CRC gas	Pure helium, 4.4 mL·min^{-1}
KED	3 V
AGD setting	AGD Level 5
RF power (W)	1,550
Sampling depth (mm)	8
Scan settings	Dwell times: 0.2 s for As, Se, Pb, Hg; 0.1 s for Cd, Ni, Cu, Fe, Zn; 0.05 s for others; 5 sweeps, 3 main runs

Software

All required steps in operating the instrument were performed in the Qtegra ISDS software. This includes starting the plasma and verification of the system´s performance using the Get Ready function. The instrument can be automatically tuned using the appropriate autotune procedure whenever desired and is typically recommended in case the required performance specifications are not achieved. Qtegra ISDS software allows to interface agnostically with a wide variety of LIMS systems so that sample details can be easily imported and combined with the analytical method in Qtegra LabBooks.

Results and discussion

The method used in this application note has been previously tested with similar sample types[3] and is now applied to the analysis of food samples on a large scale, as this is a key task set in an analytical testing laboratory. The performance of the method was again briefly verified using the analysis of a series of certified reference materials, corresponding to a range of sample types commonly analyzed in food testing.

Major elements analysis

Even though microwave assisted acid digestion commonly removes the largest part of the (mostly carbon containing) sample matrix by evaporation as CO_2, challenges caused by matrix effects remain. Major nutrient elements like Ca, Mg, Na, etc. can be present even at percent levels (i.e., in milk and other dairy products), and a wide range of concentrations may be observed in a variety of different foods for the same nutrient. Figure 1 provides an overview on the content of the most abundant nutrients in the CRMs analyzed as part of this study, as well as the results of the measurement. Despite the variability and the wide range of concentrations found in the reference materials, the iCAP MSX ICP-MS performed exceptionally well in handling the different matrices and resulted in not only excellent data accuracy but also a wide linear dynamic range of measurement for different elements. The recoveries of concentrations of reference material

analytes, against the certificate values, were in the range of 90–105% for Na, Mg, Al, P, K, Ca, Mn, Cu, and Zn and 85–120% for P and Fe (Figure 1).

When considering the nutrient elements Na, Mg, Al, P, K, Ca, Mn, Fe, Cu, Zn, and Se together, the variability of their concentrations added up ranges from ≈35 to 462 mg·L^{-1} in the different reference materials digests. This means that even though similar amounts of material were used for digestion (meaning an equal amount of total dissolved solids, TDS), the overall matrix remaining in the digested samples was highly different. Despite these variations, the iCAP MSX ICP-MS was able to run all reference materials day after day without any compromise in data quality and consistently delivered accurate results.

Analysis of toxic elements

The amounts of toxic elements typically found in food samples are again variable depending on the food type but must be commonly quantified at concentrations typically in the low to sub µg·kg^{-1} range, in compliance with local regulatory requirements. For example, according to the Commission Regulation (EU) 2023/915,[1] the lowest limit specified for Cd in a dry food is 40 µg·kg^{-1}. During sample preparation, a dilution factor of 100 was applied, which means that the maximum value to be measured in the final sample would be 0.4 µg·L^{-1}.
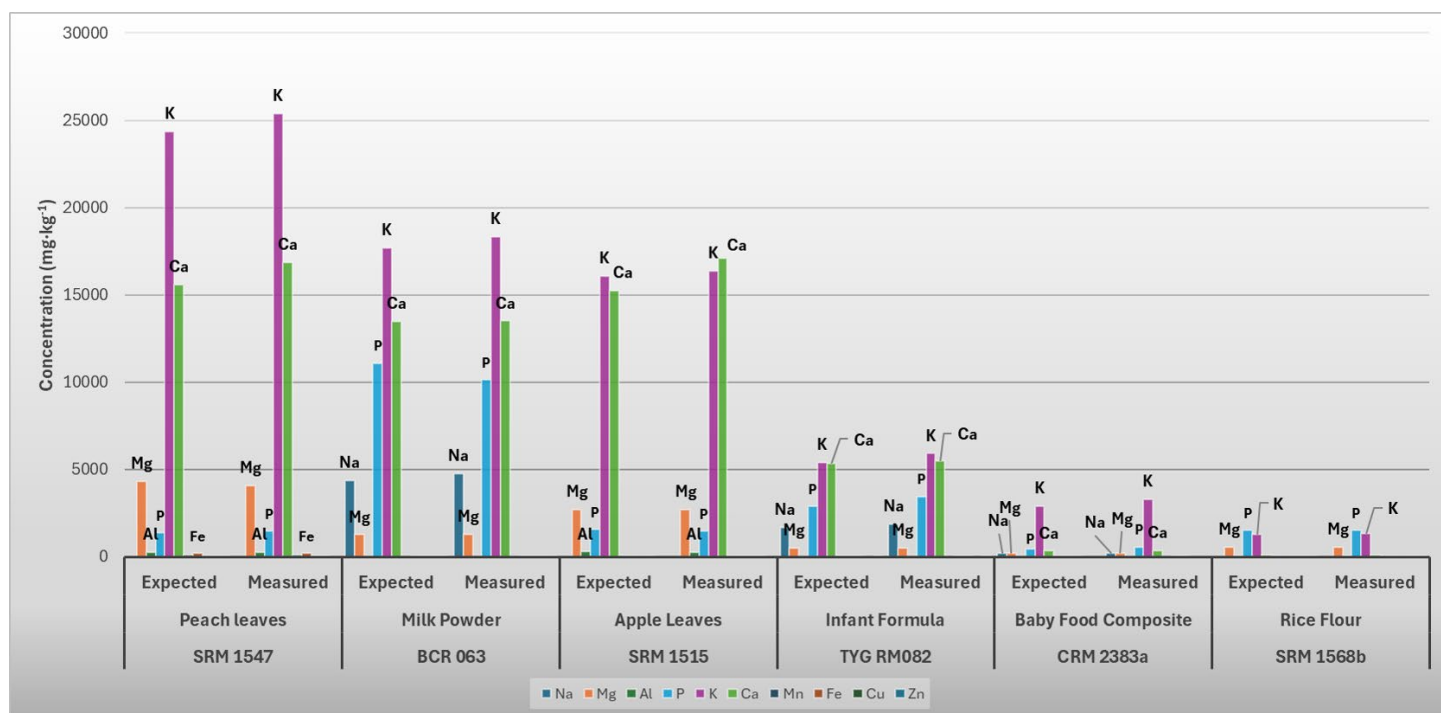

Figure 1. Major element concentrations in plant leaf and food reference materials, arranged from left to right in the order of decreasing total dissolved solids (TDS) content in the as such samples. The recoveries are between 90% and 105% for all elements depicted here except for P and Fe, which have recoveries in the 85–120% range.

Thus, measurement techniques must be able to accurately quantify in concentration ranges below these limits and handle the matrix effects from the bulk components of the sample. As an example, the measured concentrations for selected critical analytes in Rice Flour 1568b SRM have been compared to the certificate values in Table 2. The data shows excellent recoveries of the selected analytes As, Se, Mo, Cd, and Sn within 91–101% and slightly lower but still well within acceptable range[4] recovery of Pb at 83%.

Table 2. Certified/reference (latter marked with *) concentrations and recoveries of certain elements in the Rice Flour 1568b SRM

|	Certified/reference* value in solid sample (mg·L^{-1})	Expected concentration in digested solution (µg·L^{-1})	Recovery for n=3 (%)
^{75}As	0.285 ± 0.014	2.85	95.8 ± 3
^{78}Se	0.365 ± 0.029	3.65	98.4 ± 9
^{95}Mo	1.451± 0.048	14.51	101.2 ± 2
^{111}Cd	0.0224 ± 0.0013	0.224	90.6 ± 5
^{118}Sn*	0.005 ± 0.001	0.05	93.8 ± 6
^{208}Pb*	0.008 ± 0.003	0.08	83.2 ± 2

Interference removal

Interferences that have the same mass to charge ratio (*m/z*) as the analyte are a common challenge in mass spectrometry and can lead to false positives and inaccurate results. Whereas so-called polyatomic interferences can be well resolved in the He-KED mode, resolving interferences from doubly charged species, such as from rare earth elements, like ^{150}Nd^{++} and ^{150}Sm^{++} on ^{75}As, is more difficult. Using a mathematical correction based on the measurement of another isotope of the interfering element (i.e., ^{145}Nd^{++} to correct for ^{150}Nd^{++}), it is possible to account for their unwanted contribution, but the achievable accuracy depends on the concentration levels of the interferants as well as the target analytes. These effects are described in further details elsewhere.[3,5]

Robustness

The iCAP MSX ICP-MS was operated for eight weeks for the analysis of food samples. Actual analysis occurred on 25 days, with sequences lasting >12 hours on most days. After initial tuning of the instrument, performance checks available within the Qtegra ISDS Software were run daily in order to ensure instrument performance. An autotune was performed only in case the performance check criteria were not fulfilled. Daily plasma ignition, warm-up, autotunes (as required), and performance checks in desired modes can be scheduled through the Get Ready function of the software, allowing for unattended and automatic startup and preparation of the instrument for analysis. This process can also be scheduled to start at a user specified time.

Figure 2 shows a cumulative plot of all samples run over the entire duration of the study. Despite running food samples extensively for two months, amounting to almost 7,000 food samples, the performance of the instrument remained stable throughout without any maintenance actions (for example, cone cleaning) being required. The data acquired in the individual LabBooks also reflected stability with excellent internal standard response within 80–120% overall and within 90–115% on many days (Figure 3) even though the sequences had a wide range of food samples (rice, tomato, yogurt, meat, etc.) being analyzed for more than 12 hours per day without interruption.
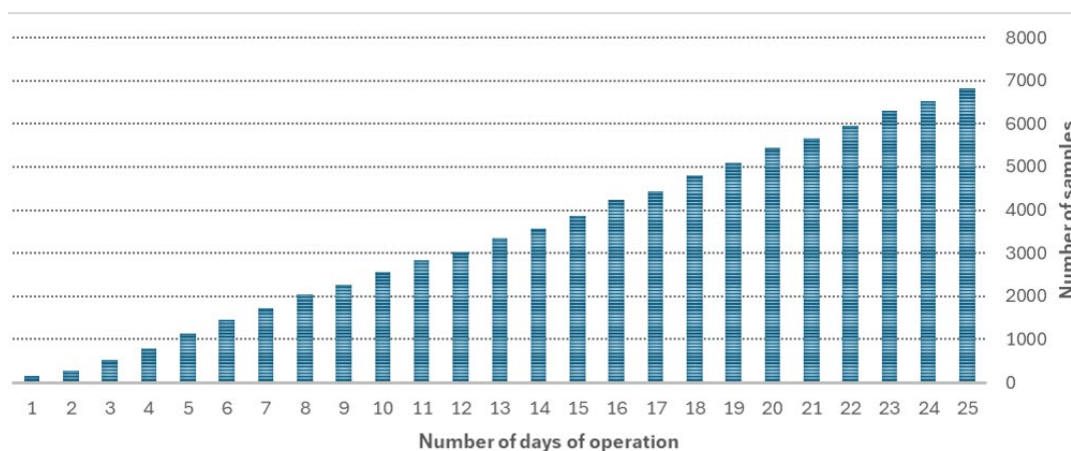
Figure 2. Cumulative plot of food samples analyzed over the entire duration of the study
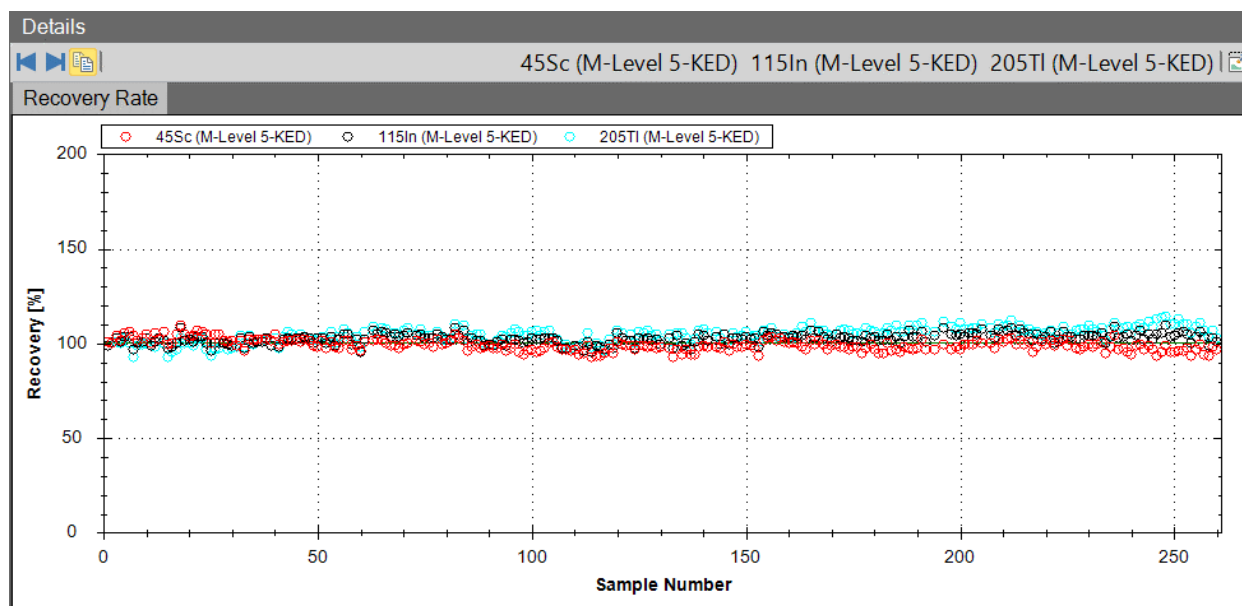
4

Figure 3. Internal standard response when running a variety of food samples in a >12 hour sequence

Conclusions

This study demonstrates how the iCAP MSX ICP-MS fulfils the requirements of food testing laboratories and resolves the typical challenges associated with this sample type. Close to 7,000 food samples were measured in this study over eight weeks where daily sequences ran for more than 12 hours with stable data, negligible analytical drift, excellent internal standard response, and without the need for any maintenance actions.

- The use of AGD allowed effective handling of matrix and compositional variability introduced by widely different food samples, commonly analyzed together on the same day in analytical testing laboratories.

- Accuracy and precision were demonstrated using applicable certified reference materials, which were successfully analyzed and quantified with excellent recoveries.

- High concentrations of nutritional elements were accurately measured alongside accurate quantification of low-level toxic elements in the same samples.

- Specially challenging elements like arsenic, typically expected in the range of a few µg·kg^{-1} or lower in food samples were measured and quantified using He-KED mode. Mathematical correction using half mass signals was further used to reduce the impact of rare earth elements, potentially present in some samples.

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.
2. GB-2762-2017 National Food Safety Standard for Maximum Levels of Contaminants in Foods, China.
3. Accurate and reliable multielement analysis of alternative protein foods using ICP-MS. Thermo Scientific Application Note 002282, https://assets.thermofisher.com/TFS-Assets/CMD/Application-Notes/an-002822-tea-icap-rqplus-food-an002822-em-en.pdf
4. AOAC Official Method 2015.01 Heavy Metals in Food Inductively Coupled Plasma–Mass Spectrometry First Action 2015.
5. 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-interference-removal-sn000214-na-en.pdf

Learn more at **thermofisher.com/icp-ms**