



Food analysis

Accurate and robust long-term analysis of food and beverage samples using single quadrupole ICP-MS

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Keywords

iCAP RQplus ICP-MS, food analysis,
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Goal

To demonstrate the applicability of the Thermo Scientific™ iCAP™ RQplus ICP-MS for robust and reliable analysis of nutritional and toxic trace elements in a large variety of food and beverage samples.

Introduction

The daily nutrition of human beings is essential to ensure good health and long life expectancy. Elements such as calcium and magnesium are examples of essential macronutrients typically contained in fresh and packaged foods, food supplements, and beverages. The same foods can pose a serious threat to the consumer's health if toxic heavy metals such as lead, cadmium, and mercury find their way into these foods through bioaccumulation and production processes. Official regulations, such as EC No 1881/2006¹ in the EU, GB-2762-2017² in China, and others, specify maximum permissible limits for such elements in food, based on concentration levels in which these toxic elements become hazardous if introduced into the human body.

All foods must undergo regular screening for both nutritional and toxic elements and fulfill the criteria specified in the applicable regulatory methods. Inductively coupled plasma mass spectrometry (ICP-MS) is a highly sensitive technique well suited for such measurements. However, the high potential variability of the sample matrix created by the wide range of food sample types can cause challenges and complications in the analysis. This is particularly important for laboratories testing a large number of samples every day.

At the same time, the analysis of a large number of diverse sample types may induce a need for more frequent maintenance of the analytical system. When not appropriately planned for, this can lead to unexpected downtime and interruptions to the overall laboratory productivity. This application note describes how the Thermo Scientific™ iCAP™ RQplus ICP-MS instrument, equipped with argon gas dilution (AGD), helps streamline the multi-element analysis of typical foods and beverages, analyzed at a large scale. This note will demonstrate how integrated dilution using argon gas, provided directly from the instrument, can remove matrix effects and improve data quality and analytical stability, producing accurate and highly reliable results day after day.

Experimental

Instrument parameters

An iCAP RQplus ICP-MS instrument was used for the analysis of 25 different food and beverage materials, plus six reference materials (certified – CRM, non-certified – RM) in this study. After microwave assisted digestion, where applicable, the samples were placed directly on the autosampler without any further dilution, and AGD was used to dilute the samples inside the standard sample introduction system of the ICP-MS. All instrument parameters are listed in Table 1.

Table 1. Instrument parameters

| Instrument parameter | Setting |
|---------------------------------|--|
| Nebulizer | Borosilicate glass MicroMist™, 400 µL·min ⁻¹ |
| Peristaltic pump tubing | Orange-green, 0.38 mm i.d. |
| Peristaltic pump speed | 25 rpm |
| Spray chamber | Quartz cyclonic, cooled at 2.7 °C |
| Torch | Quartz torch |
| Injector | 2.5 mm I.D., Quartz |
| Interface | Nickel sampler and skimmer cone, high matrix insert |
| Plasma power | 1,550 W |
| AGD setting | Low |
| Nebulizer gas | 0.39 L·min ⁻¹ |
| Additional gas flow (Ar) | 0.55 L·min ⁻¹ |
| CRC gas | 100% He 4.3 mL·min ⁻¹ |
| KED | 3 V |
| Scan setting | Dwell times – 0.2 s for Hg, Pb, Se; 0.1 s for As, Cd, Ni, Cu, Fe, Zn; 0.05 s for others; 5 sweeps, 3 main runs |
| Autosampler | iSC-65 |
| Time per sample | 2 min 32 s (including uptake, 3 repeats, wash and rinse) |

The argon gas for AGD operation was supplied directly from the instrument, accurately regulated using a mass flow controller. For the measurements, AGD was combined with the kinetic energy discrimination (KED) mode using helium as a collision cell gas. Accurate and highly sensitive analysis free from excessive matrix suppression and/or enhancement is possible using this method. Simultaneously, accurate quantification of trace levels of critical analytes within the food and beverage samples of varied matrix types is also enabled. The automatic dilution greatly reduces the amount of sample reaching the plasma, and subsequently the interface region to the mass spectrometer. By reducing this, typical maintenance actions, such as inspection or cleaning of the sample introduction system components or the interface cones, can be significantly reduced. In fact, maintenance steps like cleaning the nebulizer, cones, etc. were found to be unnecessary throughout the entire duration of the study.

The Thermo Scientific™ iSC-65 Autosampler was used for sample uptake, and the unique Step Ahead function was used for overlapping the data acquisition of a sample with the subsequent rinsing of the probe and washing of the sample introduction system, thereby shortening the overall runtime per sample. The analysis time per sample, including the analysis of a total of 26 elements with three repeats was 2 minutes 32 seconds.

List of samples and certified reference materials

A total of 20 different dry and moisture-containing food samples and five different beverage samples, purchased from local supermarkets, were analyzed in this study. The samples were separated into five groups depending on their characteristics, with each group containing five unknown samples (Table 2). Different food groups and food matrix compositions were distinguished from each other, e.g., carbohydrate rich, fat rich, etc. For accuracy checks, six different CRMs and RMs were also prepared and analyzed using the same method. Details for these materials are summarized in Table 2. The variety of sample types and reference materials was intended to test the analytical method over a broad range of typical samples analysed in analytical testing laboratories.

Table 2. Details of different sample groups, and the experiment days on which they were run, with five individual samples each and the reference materials (CRMs and RMs) analyzed in the study. The high moisture containing samples are marked in blue, the beverages were clear liquids, and the rest were dry samples.

| Experiment day | Group no. | Sample group details | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 |
|--|-----------|-------------------------------|-----------------|--------------------------------|--------------------------|-------------------------|----------------------|
| Day 1 | 1 | Carbohydrate and protein rich | Rice | Cereal | Couscous | Flour | Fish (sardines) |
| Day 2 | 2 | Vegetables and fruits | Puree-1 | Puree-2 | Tomato | Spinach | Carrot |
| Day 3 | 3 | Spices | Mustard sauce | Pepper | Coriander | Basil | Cinnamon |
| Day 4 | 4 | Milk and high fat | Infant formula | Milk powder | Biscuits | Cocoa powder | Drinking chocolate |
| Day 5 | 5 | Beverages | Apple juice | Orange drink | Cola | Flavoured water | Energy drink |
| Days 6 to 9 consisted of runs where samples from all different groups were randomly mixed together. | | | | | | | |
| CRMs and RMs | | Rice Flour SRM 1568b | Fish CRM DORM-3 | NIST 2383a Baby food composite | Infant Formula RM TYG082 | Milk powder CRM BCR-063 | Biscuit CRM LGC-7103 |

Sample preparation

The food samples, groups 1–4 (Table 2), were prepared by closed vessel microwave digestion in a microwave digestion system (Milestone ETHOS™, Milestone SRL, Italy). Homogenized aliquots of 0.50 ± 0.05 g (dry samples) or 2.0 ± 0.2 g (wet samples) of each sample were accurately weighed in and mixed with 4 mL HNO₃, 1.5 mL HCl (67% and 35–37% (m/v)) (Optima™ grade, Fisher Scientific™) and 0.5 mL of 18 MΩ-cm ultrapure water. The samples were digested and allowed to cool (30 min at 210 °C, 1,200 W, ramp up time 15 min). The solutions were quantitatively recovered and made up to a final volume of 50 mL with ultrapure water. All twenty food samples from Groups 1–4 and the six reference materials were digested using this method. The beverage samples belonging to Group 5 did not require a digestion step; these were simply degassed for 10 minutes in an ultrasonic bath and then diluted 10-fold using 2% (v/v) HNO₃ acid.

Standards

All calibration standards were prepared from single element standards (SPEX CertiPrep™, Metuchen, NJ, USA) in an acid matrix of 2% (v/v) HNO₃. The elements were grouped according to their expected concentration range in regular food matrices (Table 3). DORM-3 Fish CRM was diluted 10x further in order to bring the concentrations of arsenic and mercury within the calibration range without adding higher concentration standards of these elements, which are regulated strictly at extremely low limits in most of the studied foods.

An internal standard solution containing 1 mg·L⁻¹ of Sc and 20 µg·L⁻¹ of Y, Rh, Lu, and Tl each was added online via a T-piece. For sequences including the beverage samples, 4% (v/v) isopropyl alcohol (IPA) was added to the internal standard solution along with the other elements to compensate for the well-known carbon

enhancement effect on certain elements such as As and Se. Calibration standard (Std 4) was furthermore used as a quality control (QC) standard for regularly checking the data quality during uninterrupted analysis of a large number of samples.

Table 3. List of calibration standards and their concentrations (in µg·L⁻¹ for Groups 1–3, and in mg·L⁻¹ for Groups 4–8)

| Group | Elements | Std 1 | Std 2 | Std 3 | Std 4 | Std 5 | Std 6 |
|-------|--|-------|-------|-------|-------|-------|-------|
| 1 | Hg | 0.005 | 0.01 | 0.05 | 0.1 | 0.5 | 1 |
| 2 | Ag, As, Cd, Ce, Co, Cr, Cu, Li, Mo, Ni, Pb, Sb, Se, Sn | 0.05 | 0.1 | 0.5 | 1 | 5 | 10 |
| 3 | Mn | 0.5 | 1 | 5 | 10 | 50 | 200 |
| 4 | B, Be, Fe, Zn | 0.005 | 0.01 | 0.05 | 0.1 | 0.5 | 1 |
| 5 | Al | 0.025 | 0.05 | 0.25 | 0.5 | 2.5 | 5 |
| 6 | P, Mg | 1 | 2 | 10 | 20 | 100 | 200 |
| 7 | Ca, K | 1.5 | 3 | 15 | 30 | 150 | 300 |
| 8 | Na | 3 | 6 | 30 | 60 | 300 | 600 |

Experimental design

To demonstrate the suitability of this method for analyzing the varied food matrices as well as a high volume of samples overall daily, as in a typical food testing laboratory, several long experiments were run on nine working days over two weeks. On days 1 to 5, samples belonging to the respective sample groups shown in Table 2 were run in a single sequence. From day six onwards, all samples were run together for three more days. The beverage samples (Group 5) were included in the mix on days 5 and 9 so that 4% (v/v) IPA was included in the internal standard solution on both days.

The measurement sequences consisted of a calibration block with blanks and standards, followed by the QC standard (Std 4) run first as an initial calibration verification (ICV) solution and then as a continuing calibration verification (CCV) solution once every ten unknown samples. The CRMs and RMs were distributed throughout the sequences for regular accuracy checks. The daily experiments consisted of about 8 to 10 hours of uninterrupted measurements to check signal stability and robustness of the developed method. The results are discussed in the following sections.

Software

The Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software was used for instrument tuning, using one of the three readily available AGD tune sets, as well as for setting up measurement sequences and data acquisition. Qtegra ISDS Software for the iCAP RQplus ICP-MS includes the Thermo Scientific™ Hawk™ Consumables and Maintenance Assistant, which allows dedicated alerts for regular maintenance requirements to be set. Tracking of instrument performance over longer periods based on the data acquired during the regular performance checks is also possible, enabling a comprehensive record of the instrument's status over its lifetime. The AGD level was optimized using the available autotune routine within the instrument software. Over the entire duration of this study, the instrument was tuned only once at the very beginning, and no significant drop in sensitivity was observed throughout, as demonstrated in Figure 1.

Results and discussion

Linearity and sensitivity

A wide analytical range was covered to accommodate both toxic and nutritional elements in a single read out of a sample, ranging from 0.005 µg·L⁻¹ for mercury to 600,000 µg·L⁻¹ for sodium, according to expected concentrations in different food samples (Table 2 and Table 3).

The instrument detection limit (IDL), based on ten repeats of the blank, the background equivalent concentration (BEC), and coefficient of determination (R²) are readily available in the Qtegra ISDS Software. The method limit of quantification (MLOQ) for a particular element in the unprocessed samples is calculated by multiplying the relevant dilution factor (DF) with the limit of quantification in solution (LOQ) where LOQ = 3 x IDL. The IDL, R² and MLOQ values determined in this study are listed in Table 4. All MLOQs are well below the limits typically required by official regulations for different food matrices.

Since the beverage samples were not digested but simply diluted, the amount of carbon therein (i.e., sugars, sweeteners, etc.) may lead to an artificial signal enhancement of elements with higher ionization potential, in particular arsenic and selenium (As, Se), in the plasma. The addition of 4% (v/v) IPA in the internal standard solution leads to similar carbon content across all samples and standard solutions irrespective of their native matrix compositions. This ensures accurate measurements of arsenic and selenium and provides better IDLs for these elements too (Table 3). This procedure also allows both digested as well as undigested samples (but free of particulate matter) to be analyzed within the same measurement sequence using the same method for all samples.

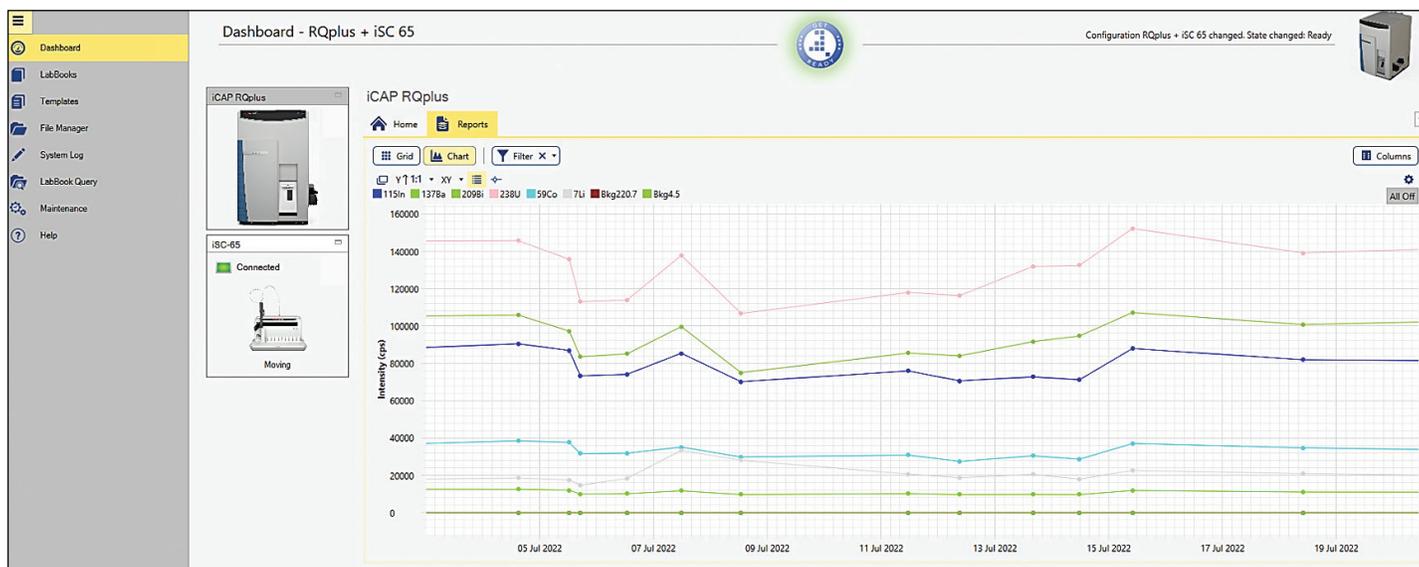


Figure 1. Sensitivity performance chart of the iCAP RQplus ICP-MS instrument over the entire duration of this study

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) x 3 x IDL. The values in blue are in mg·L⁻¹ and other values are in µg·L⁻¹.

| Analyte | R ² | LOD | MLOQ in beverage samples, DF = 10 | MLOQ in wet food samples, DF = 25 | MLOQ in dry food samples, DF = 100 |
|-------------------|----------------|--------|-----------------------------------|-----------------------------------|------------------------------------|
| ⁷ Li | 0.9994 | 0.193 | 5.8 | 14.5 | 57.9 |
| ⁹ Be | 0.9997 | 0.002 | 0.05 | 0.11 | 0.45 |
| ²³ Na | 0.9995 | 0.002 | 0.1 | 0.1 | 0.5 |
| ²⁴ Mg | 0.9999 | 0.001 | 0.02 | 0.05 | 0.18 |
| ²⁷ Al | 0.9994 | 0.001 | 0.02 | 0.05 | 0.19 |
| ³¹ P | 0.9999 | 0.012 | 0.4 | 0.9 | 3.6 |
| ³⁹ K | 0.9995 | 0.006 | 0.2 | 0.4 | 1.7 |
| ⁴⁴ Ca | 0.9998 | 0.006 | 0.2 | 0.4 | 1.7 |
| ⁵² Cr | 0.9994 | 0.015 | 0.5 | 1.1 | 4.5 |
| ⁵⁵ Mn | 0.9999 | 0.0004 | 0.01 | 0.03 | 0.11 |
| ⁵⁷ Fe | 0.9994 | 0.0005 | 0.01 | 0.04 | 0.15 |
| ⁵⁹ Co | 0.9999 | 0.004 | 0.1 | 0.3 | 1.2 |
| ⁶⁰ Ni | 0.9998 | 0.013 | 0.4 | 1.0 | 4.0 |
| ⁶⁵ Cu | 0.9994 | 0.032 | 1.0 | 2.4 | 9.6 |
| ⁶⁶ Zn | 0.9998 | 0.133 | 4.0 | 9.9 | 39.8 |
| ⁷⁵ As* | 0.9996 | 0.004 | 0.1 | 0.3 | 1.3 |
| ⁷⁷ Se* | 0.9997 | 0.015 | 0.5 | 1.2 | 4.6 |
| ¹¹¹ Cd | 0.9997 | 0.005 | 0.1 | 0.4 | 1.5 |
| ¹¹⁸ Sn | 0.9993 | 0.013 | 0.4 | 0.9 | 3.8 |
| ¹²¹ Sb | 0.9999 | 0.013 | 0.4 | 1.0 | 3.9 |
| ¹⁴⁰ Ce | 0.9999 | 0.001 | 0.02 | 0.06 | 0.23 |
| ²⁰² Hg | 0.9998 | 0.003 | 0.1 | 0.2 | 0.9 |
| ²⁰⁸ Pb | 0.9997 | 0.001 | 0.03 | 0.08 | 0.30 |

*with IPA

Accuracy

Every long sequence in this study included several CRM and RM samples, representative of the wide sample range. The recoveries of different elements relative to the certified concentrations of the CRMs were well within the generally acceptable range of 80–120%, with most recoveries within 90–105% (Table 5). Some regulated methods applicable for food analysis even accept ranges of 75–125% for CRM analyte concentration recoveries³, thus the recoveries obtained here strongly validate the method accuracy.

For accuracy checks for beverages, where none of the reference materials matched the sample type, a spike recovery test at a concentration level of 5 µg·L⁻¹ was performed. The recoveries were within 100 ± 10% with the results for arsenic and selenium being in the same range as the other analytes, thus confirming that the results were not positively biased by carbon enhancement, and hence addition of 4% (v/v) IPA to the internal standard solution worked effectively to overcome this effect.

Table 5. Certified values, mean recoveries, and associated standard deviations for “n” number of individual measurements of CRMs and RMs in the long experiments. Values marked with * are known reference values and not certified values.

| Element | Biscuit CRM LGC-7103 | | TYG RM082 infant formula | | NIST CRM 2383a baby food composite | |
|-------------------|---------------------------------------|-----------------------|---------------------------------------|-----------------------|---------------------------------------|-----------------------|
| | n = 40 | Dilution factor = 100 | n = 50 | Dilution factor = 100 | n = 16 | Dilution factor = 25 |
| | Certified value (mg·L ⁻¹) | Average recovery (%) | Certified value (mg·L ⁻¹) | Average recovery (%) | Certified value (mg·L ⁻¹) | Average recovery (%) |
| ²³ Na | 5010 ± 400 | 96 ± 7 | 1698 ± 29 | 93 ± 6 | 195 ± 29 | 94 ± 9 |
| ²⁴ Mg | 254 ± 59 | 92 ± 6 | 505 ± 7 | 92 ± 6 | 212.2 ± 4.0 | 106 ± 7 |
| ³¹ P | 900 ± 140 | 91 ± 5 | 2898 ± 60 | 90 ± 11 | 453 ± 11 | 90 ± 9 |
| ³⁹ K | 1580 ± 170 | 97 ± 6 | 5408 ± 90 | 91 ± 6 | 2910 ± 220 | 102 ± 6 |
| ⁴⁴ Ca | – | – | 5355 ± 77 | 95 ± 4 | 342.6 ± 5.0 | 102 ± 11 |
| ⁵⁵ Mn | 5.49 ± 0.6 | 100 ± 8 | 0.623 ± 0.022 | 87 ± 11 | 0.963 ± 0.064 | 93 ± 5 |
| ⁵⁷ Fe | – | – | 74.8 ± 1.4 | 94 ± 5 | 4.420 ± 0.51 | 104 ± 9 |
| ⁵⁹ Co | – | – | – | – | 0.048 ± 0.005 | 100 ± 7 |
| ⁶⁵ Cu | – | – | 3.69 ± 0.08 | 101 ± 5 | 0.758 ± 0.082 | 94 ± 6 |
| ⁶⁶ Zn | 6.41 ± 0.99 | 113 ± 5 | 52.1 ± 0.9 | 115 ± 5 | 2.22 ± 0.18 | 88 ± 2 |
| ⁷⁸ Se | – | – | 0.123 ± 0.007 | 104 ± 10 | – | – |
| Element | CRM BCR 063 milk powder | | Rice flour SRM 1568b | | Fish CRM DORM-3 | |
| | n = 16 | Dilution factor = 100 | n = 21 | Dilution factor = 100 | n = 10 | Dilution factor = 100 |
| | Certified value (mg·L ⁻¹) | Average recovery (%) | Certified value (mg·L ⁻¹) | Average recovery (%) | Certified value (mg·L ⁻¹) | Average recovery (%) |
| ²³ Na | 4370 ± 31 | 102 ± 8 | – | – | – | – |
| ²⁴ Mg | 1263 ± 24 | 108 ± 9 | 559 ± 10 | 91 ± 7 | – | – |
| ²⁷ Al | – | – | 4.21 ± 0.34 | 96 ± 8 | – | – |
| ³¹ P | 11100 ± 130 | 93 ± 8 | 1530 ± 40 | 83 ± 6 | – | – |
| ³⁹ K | 17680 ± 190 | 102 ± 7 | 1282 ± 11 | 95 ± 5 | – | – |
| ⁴⁴ Ca | 13490 ± 100 | 103 ± 8 | 118.4 ± 3.1 | 103 ± 10 | – | – |
| ⁵⁵ Mn | – | – | 19.2 ± 1.8 | 93 ± 6 | – | – |
| ⁵⁷ Fe | 2.32 ± 0.23 | 93 ± 4 | 7.42 ± 0.44 | 99 ± 8 | 347 ± 20 | 92 ± 3 |
| ⁶⁵ Cu | 0.602 ± 0.019 | 115 ± 5 | 2.35 ± 0.16 | 96 ± 9 | 15.5 ± 0.63 | 96 ± 11 |
| ⁶⁶ Zn | 49 ± 0.6 | 82 ± 6 | 19.42 ± 0.26 | 101 ± 9 | 51.3 ± 3.1 | 91 ± 3 |
| ⁷⁵ As | – | – | 0.285 ± 0.014 | 93 ± 6 | 6.88 ± 0.30 | 91 ± 10 |
| ⁷⁸ Se | 0.129* | 90 ± 14 | 0.365 ± 0.029 | 95 ± 9 | – | – |
| ¹¹¹ Cd | – | – | 0.0224 ± 0.0013 | 91 ± 5 | 0.290 ± 0.020 | 104 ± 4 |
| ²⁰² Hg | – | – | 0.00591 ± 0.00036 | 102 ± 15 | 0.38 ± 0.06 | 109 ± 8 |
| ²⁰⁸ Pb | 0.0185 ± 0.001 | 107 ± 10 | – | – | 0.395 ± 0.050 | 96 ± 6 |

Robustness

The robustness of the developed method in this application note was subsequently demonstrated by the successful measurement of 1878 different samples over a period of almost two weeks. On each day, the total analysis time was about 8 to 10 hours, typically allowing the uninterrupted analysis of ~220–270 samples (Tables 2 and 6). A single calibration block at the beginning of the entire sequence was sufficient to obtain accurate data throughout the run, and all sequences showed excellent stability of both the internal standard response as well as all applicable QC checks. The calibration block was followed by QC standards (Std 4) repeated after every ten unknown samples to check for calibration validity. The above-mentioned CRMs and RMs were also included in every sequence to demonstrate the accuracy.

Figure 2 shows a representative example of the internal standard recovery typically obtained. The results shared in Table 6 further demonstrate the excellent signal stability and absence of drift, regardless of the sample composition. This table summarizes the average internal standard recovery across all groups of sample matrices that were analyzed individually on days 1 through 5, as well as mixed on days 6–9. The recoveries were extremely stable, not only on individual measurement days but also consistent when compared over all of the different matrix types (Table 6), which further proves that different types of food and beverage sample matrices can be flexibly analyzed and also mixed in a single method without any negative impact on the data quality or measurement reliability.

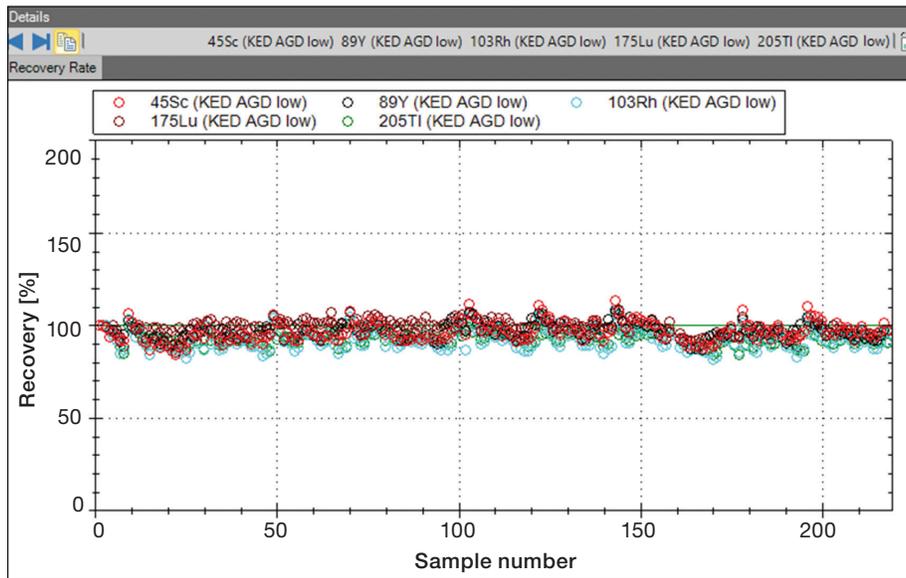


Figure 2. Stable response of the internal standards Sc, Y, Rh, Lu, and TI of 85–110% over >9 h of uninterrupted measurement of food samples from different food groups in the same analytical sequence

Table 6. Internal standard recovery (%) compared to the first blank of the sequence during the 8–10 hours long food experiments run daily and standard deviation for each internal standard throughout the whole of each run. “n” is the number of unknown food and beverage samples analyzed.

| Day | Group | n | Sc | | Y | | Rh | | Lu | | TI | |
|-------------------------|-------|-----|--------------|-------|--------------|-------|--------------|-------|--------------|-------|--------------|-------|
| | | | Recovery (%) | Stdev |
| 1 | 1 | 216 | 85 | 8.1 | 90 | 7.9 | 90 | 7.3 | 96 | 8.0 | 108 | 8.9 |
| 2 | 2 | 249 | 101 | 4.5 | 99 | 4.1 | 96 | 3.5 | 95 | 3.4 | 102 | 3.6 |
| 3 | 3 | 216 | 87 | 3.5 | 95 | 4.3 | 90 | 3.4 | 96 | 2.9 | 101 | 5.0 |
| 4 | 4 | 250 | 88 | 8.9 | 89 | 8.9 | 88 | 9.0 | 90 | 9.3 | 90 | 9.1 |
| 5 | 5 | 214 | 96 | 8.7 | 98 | 8.6 | 96 | 8.9 | 98 | 7.9 | 102 | 9.6 |
| 6 | 1–4 | 253 | 107 | 9.8 | 106 | 9.6 | 100 | 8.5 | 104 | 7.5 | 106 | 9.2 |
| 7 | 1–4 | 216 | 96 | 8.4 | 96 | 8.0 | 91 | 7.9 | 99 | 7.8 | 92 | 7.2 |
| 8 | 1–4 | 244 | 97 | 5.2 | 96 | 4.1 | 94 | 5.0 | 99 | 3.6 | 95 | 3.8 |
| 9 | 1–5 | 270 | 94 | 4.7 | 100 | 4.2 | 93 | 4.9 | 100 | 2.8 | 97 | 3.6 |
| Average of 1878 samples | | | 95 | | 97 | | 93 | | 97 | | 99 | |

This ability is further demonstrated when looking at the vastly different composition of the individual samples run. Major elemental concentrations of representative samples (one of each of the aforementioned groups) are shown in Figure 3. Concentrations of the toxic elements arsenic, cadmium, mercury, and lead quantified across the different samples are illustrated in Figure 4. It is noteworthy that elevated concentrations of arsenic are not unusual in marine samples like fish or seafood, where in contrast to other samples, it is predominantly found as part of organic compounds that are far less toxic compared to the inorganic forms of arsenic. To make a precise assessment on potential risks associated with the levels of arsenic found in the fish sample, speciation analysis, using a hyphenated system with ion chromatography (IC-ICP-MS), would be required.

Although microwave-assisted digestion degrades the carbon-based matrix components, the actual sample matrix (predominantly considering the major elements such as sodium, potassium, calcium, or phosphorous) differs significantly between the samples. For example, those elements alone make up for 200 ppm total dissolved solids (TDS) in digested solutions of samples like black pepper and milk powder, whereas the same elements only contribute 30 ppm TDS in tomato sample digest. This alone may lead to a change in the signal response, leading to varying signal suppression, and ultimately a re-run of the sample if the result lies outside of the acceptable limits for the internal standard response. The use of argon gas dilution allows the uninterrupted analysis of any type of food sample with a highly robust and reliable instrument response, so that analytical testing laboratories can fully rely on being able to return results back to clients within agreed turnaround times.

A total of 161 individual QC checks were run during days 1–9. On individual days, the QC recoveries were all within 80–120 % with an average standard deviation of $\pm 3\%$ for individual elements,

as seen in the example from day 4 in Figure 5. Compared over all nine days, the mean recoveries for the individual QC analytes were within 90–112%, with an average standard deviation of $\pm 7\%$ for each analyte. The CRM and RM samples that were interspersed regularly throughout all sequences to provide further evidence for measurement accuracy also showed very good recoveries as discussed in the accuracy section (Table 5).

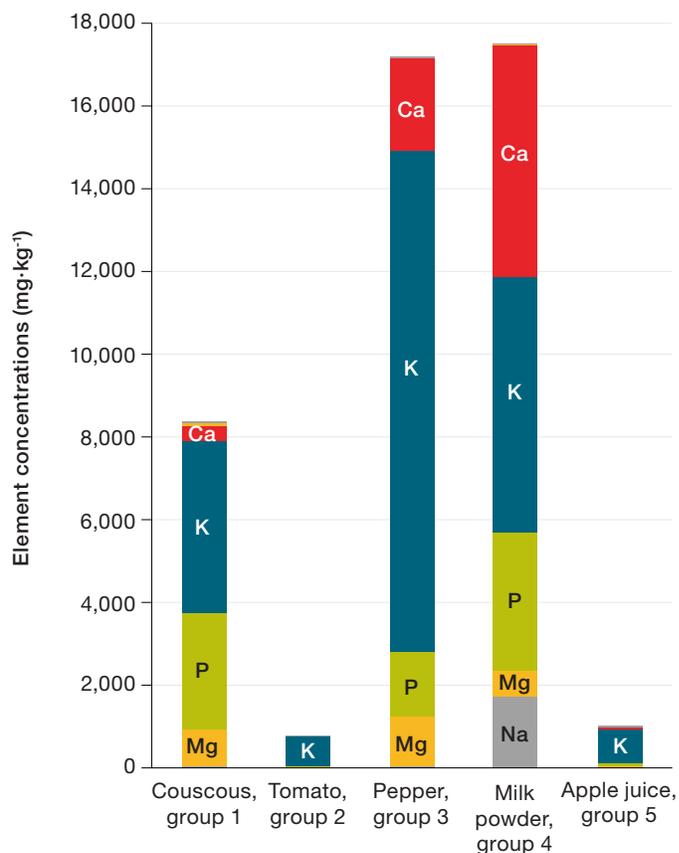


Figure 3. Elemental compositions of representative samples from Groups 1–5

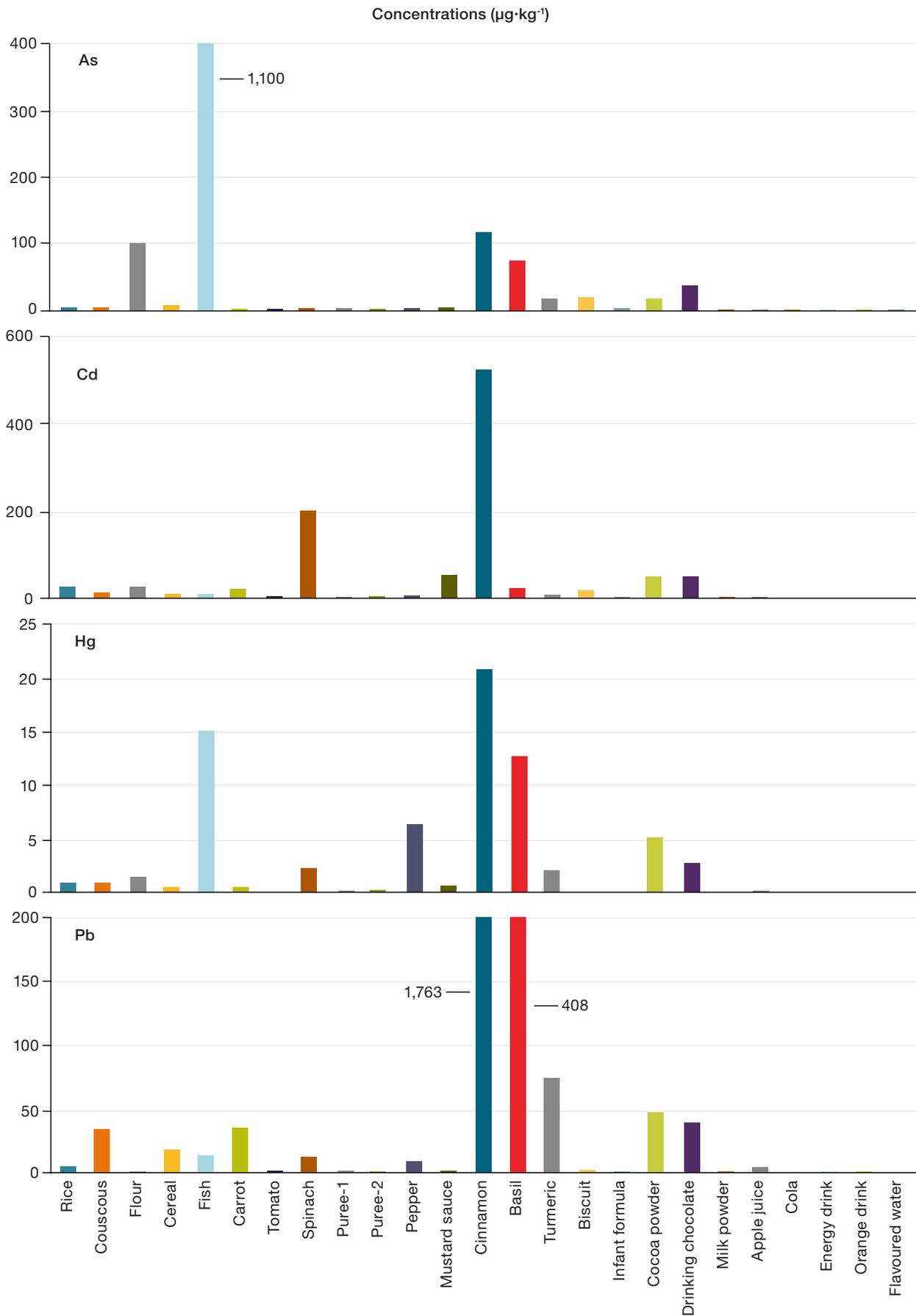


Figure 4. Concentrations of toxic elements arsenic, cadmium, mercury, and lead in the samples

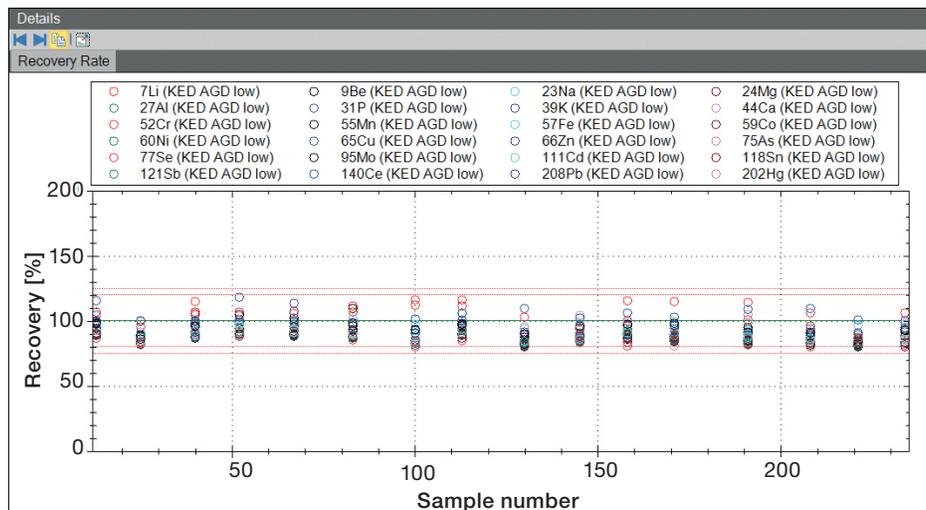


Figure 5. Accuracy of quality control standards (Continuing Calibration Verification, CCV, n=16) spanning >10 hours of continuous measurements of a variety of different food samples (day 8, Groups 1–4)

Conclusions

The current study presents a single highly sensitive and robust analytical method using the iCAP RQplus ICP-MS with argon gas dilution (AGD) for multi-element analysis of different food and beverage samples available for human consumption. Typical nutritional and toxic elements, regularly monitored in foods and beverages, can all be measured using this method without worrying about matrix effects. Automatic instrument tuning and low maintenance requirements easily allow analysis of thousands of samples as has been demonstrated. This application note includes data acquired over a period of nearly two weeks and the most important results of this study are summarized below:

- This study proposes a simple ICP-MS method based on AGD for the accurate, precise, and efficient quantitation of a wide range of analytes. A total of 26 different elements—present as both high concentration nutritional elements and extremely low-level toxic metals—were analyzed in 25 different food and beverage samples.
- The dry and moisture containing food samples were chosen to cover a wide range of food groups including high carbohydrate containing foods, vegetable-based foods, spices, milk, high fat containing products, and baby foods. The beverages, on the other hand covered fruit juices, flavored beverages, and energy drinks.

- The dilution of all samples inside the ICP-MS with a direct argon gas supply is an easy and convenient approach to achieving the dilution levels required to minimize matrix effects. This was demonstrated by excellent internal standard stability over the entire mass range and also over consecutive days of analysis, with >200 samples and >8 hours of data acquisition per day. A total of 1878 unknown samples were analyzed together with all required QC checks.
- Excellent sensitivity and a wide linear dynamic range were achieved, enabling the user to easily comply with global regulations and ensure the safety and quality of the food supply.
- The method was validated using six different CRM / RM samples run 153 times during the different experiments. Recoveries of between 82% and 115% were obtained across all analytes, demonstrating the accuracy of the measurements.
- Minimal user interaction and only limited maintenance were necessary during the entire duration of the measurements made in this study, demonstrating that the iCAP RQplus ICP-MS is an ideal elemental analysis tool for analytical food testing facilities.

References

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2. GB-2762-2017 National Food Safety Standard for Maximum Levels of Contaminants in Foods, China.
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