Leveraging innovation in ICP-MS for efficient and sensitive analysis of foods and beverages

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Abstract

Elemental analysis of food is crucial to ensure the safety of food and nutritional adequacy for humans. Inductively Coupled Plasma Mass Spectrometry (ICP-MS) offers a powerful and sensitive method for detecting and quantifying trace elements and potential contaminants in food products. This study utilizes ICP-MS to analyze a variety of baby food and beverage samples, focusing on the detection of essential nutrients as well as toxic elements. The method involves sample preparation through microwave digestion, followed by precise and accurate measurement using ICP-MS.

Introduction

Many countries have established their own regulations and guidelines to ensure the safety and quality of food and beverages, based on scientific research and risk assessments conducted by health and food safety authorities. Among a range of other contaminants, toxic heavy metals, such as lead, arsenic, cadmium, and mercury, are subject to screening in a variety of different food and beverage products.

Foods intended for infants and young children are especially strictly regulated. The Baby Food Safety Act of 2021 requires manufacturers and the FDA to take long overdue action by setting maximum levels of inorganic arsenic (10 µg·kg^{-1}, 15 µg·kg^{-1} for cereal), lead (5 µg·kg^{-1}, 10 µg·kg^{-1} for cereal), cadmium (5 µg·kg^{-1}, 10 µg·kg^{-1} for cereal), and mercury (2 µg·kg^{-1}) allowed in baby food. As the FDA works on a "closer to zero plan", the European Union has established regulations and maximum limits for various contaminants, including toxic elements, in baby food.

ICP-MS is the commonly used analytical technique to measure trace elements in various foods and beverages. The specific trace elements to be checked by ICP-MS in the foods may vary depending on the purpose of the analysis and the specific concerns related to the safety and quality of the product.

Figure 1. The Thermo Scientific™ iCAP™ MTX triple quadrupole ICP-MS

Materials and methods

Instrumentation

A Thermo Scientific™ iCAP™ MTX ICP-MS and a Thermo Scientific™ iSC-65 Autosampler were used for analysis. The ICP-MS was operated using the parameters highlighted in Table 1. To facilitate the robustness to analyze a high number of potentially very different sample types, the instrument was operated using argon gas dilution (AGD), applying a mild (approximately 5 times) dilution to all samples. This ensures the productivity and uptime requirements in a high productivity laboratory environment can be met. To meet the stringent detection limit requirements put in place by regulatory authorities, the iCAP MTX ICP-MS was operated in SQ-KED and TQ-O_2 modes to confidently eliminate polyatomic and isobaric interferences, but also deliver the sensitivity needed to reach the requested quantification limits for the critical elements.

Table 1. Instrument parameters used on the iCAP MTX ICP-MS

Parameter	Value
Nebulizer	iCAP MX Series Nebulizer
Interface cones	Ni – tipped sample and skimmer
Spray chamber	Cyclonic quartz
Injector	Quartz, 1.5 mm ID
Torch	Quartz
Auxiliary Flow (L·min^{-1})	0.8
Cool Gas Flow (L·min^{-1})	14
Automatic dilution	Level-5
AGD Humidifier	ON
Nebulizer Flow (L·min^{-1})	0.45
Argon Gas dilution Flow (L·min^{-1})	0.50
QCell KED Flow (mL·min^{-1})	4.91
QCell O2 Flow (mL·min^{-1})	0.31
RF Power (W)	1550
Sampling depth (mm)	8
Number of Replicates	3
Spray Chamber Temp (0°C)	2.7

Sample and standard preparation

Six baby foods (infant biscuit, carrot, yoghurt etc.) and 13 different beverages, including a variety of fruit juices and soft drinks, were selected for this study. Products with different compositions (i.e., content of fat, moisture content, or dry powders) were chosen to obtain a range of different matrices and analyte concentrations. Certified reference materials (TFV002RM - Skimmed milk powder, LGC7103 - Sweet digestive biscuit) were analyzed to check the method accuracy and precision.

Despite the different characteristics, all samples were prepared using microwave assisted acid digestion to apply a single preparation method across all types of sample materials. An UltraWAVE microwave system (Milestone Srl., Sorisole, Bergamo, Italy) was used for the sample preparation. The sample preparation details are given in Table 3.

During all analyses, an internal standard (IS) solution was automatically added (500 µg·L^{-1} Sc and Ge; 20 µg·L^{-1} Rh and Ir) before nebulization to compensate for any sample matrix effects in the plasma. Calibration curves were prepared from a multielement stock (34 elements). QC standards were independently prepared to verify the validity of the calibration curve throughout the run.

Table 3. Sample preparation protocols for baby foods and beverages

Sample type	Sample amount	Acid mixture	Final volume (mL)	Final acidity (V/V)
Dry samples	0.5 g	2 mL HNO3 + 0.5 mL HCl	50	4% HNO3 + 1% HCl
Wet samples	1 g			
Beverage samples	5 g			

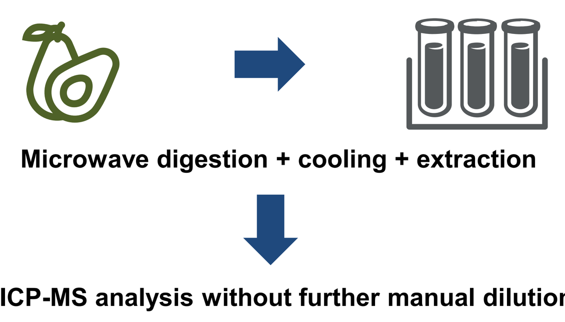


Table 4. Calibration and QC standards for trace elements

Trace elements	STD 1	STD 2	STD 3	STD 4	STD 5	STD 6	STD 7
Multielement solution	0.1 µg·L^{-1}	0.5 µg·L^{-1}	1.0 µg·L^{-1}	10 µg·L^{-1}	20 µg·L^{-1}	50 µg·L^{-1}	100 µg·L^{-1}
Hg	0.002 µg·L^{-1}	0.01 µg·L^{-1}	0.02 µg·L^{-1}	0.2 µg·L^{-1}	0.4 µg·L^{-1}	1.0 µg·L^{-1}	-
Na, Mg, Si, P, S, K, Ca	0.02 mg·L^{-1}	0.1 mg·L^{-1}	0.5 mg·L^{-1}	2.0 mg·L^{-1}	10 mg·L^{-1}	50 mg·L^{-1}	-

Results and discussion

Calibration

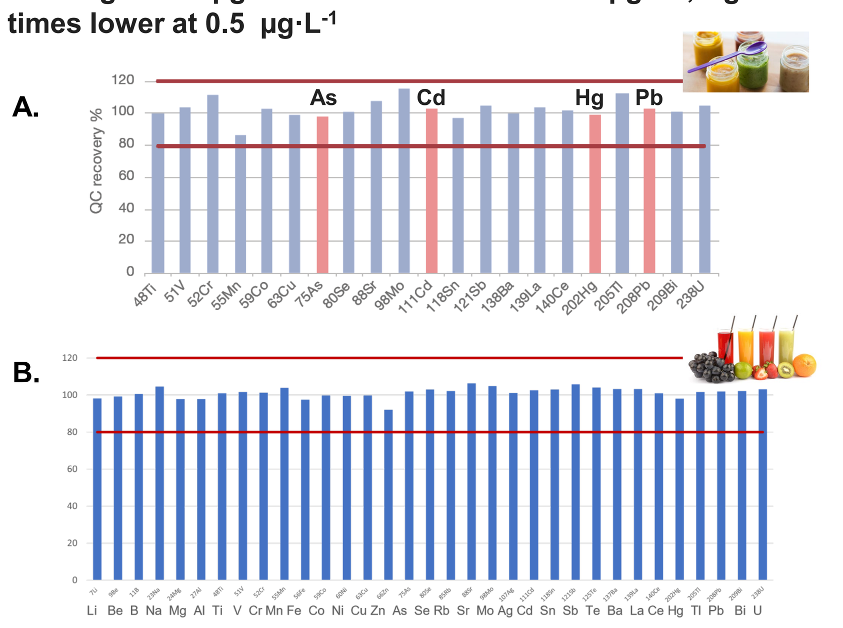
Table 5 summarizes the analytical figures of merit for all analytes including the correlation coefficients, the limits of quantification, and the quantification limits of the method (MLOQ). Whereas the limit of quantification (LOQ) only considers what can be quantified by the instrument, the method quantification limit (MLOQ) also accounts for the complete sample preparation, i.e., the sample weight and the dilution factor. Specifically highlighted in the table are the results obtained for As, Cd, Hg, and Pb, elements which are of specific regulatory concern due to their toxicity and risk for bioaccumulation. As can be seen, the achieved MLOQs are at least a factor two lower compared to the nominal limits specified in EAM 4.7.

Table 5. Analytical figures of merit for all analytes, including R^2, IDL, and MQL for dry and wet sample types. Values in bold indicate concentrations given in mg·kg^{-1}. Highlighted are the calculated limits for toxic elements As, Cd, Hg, and Pb.

	EAM 4.7 Measured Limits iCAP MTX ICP-MS				Typical ICP-SQMS EAM 4.7 Nominal Analytical Limits
Element	R^2	LOQ (µg·kg^{-1})	MLOQ (dil 50) Wet Baby Food (µg·kg^{-1})	MLOQ (dil 100) Dry Baby Food (µg·kg^{-1})	LOQ (µg·kg^{-1})
^{11}B	**0.9913**	**0.013**	**0.668**	**1.336**	
^{23}Na	**0.9999**	**0.001**	**0.072**	**0.145**	
^{24}Mg	**0.9999**	**0.001**	**0.043**	**0.086**	
^{28}Si	**0.9998**	**0.019**	**0.974**	**1.947**	
^{27}Al	>0.9999	3.161	158.0	316.1	
^{28}Si	**0.9998**	**0.019**	**0.974**	**1.947**	
^{39}K	**0.9999**	**0.026**	**1.325**	**2.651**	
^{44}Ca	**0.9999**	**0.013**	**0.625**	**1.250**	
^{48}Ti	0.9999	0.023	1.147	2.295	
^{51}V	0.9999	0.091	4.550	9.101	
^{52}Cr	0.9998	0.107	5.390	10.78	48.9
^{55}Mn	0.9999	0.060	3.035	6.071	21.2
^{56}Fe	0.9999	0.153	7.679	15.36	
^{59}Co	0.9997	0.004	0.235	0.471	
^{60}Ni	0.9985	0.372	18.63	37.26	58
^{63}Cu	0.9995	0.093	4.690	9.381	54.7
^{66}Zn	0.9998	0.610	30.47	60.95	340
^{75}As	0.9999	0.054	2.718	5.436	11.6
^{80}Se	0.9999	0.084	4.204	8.408	66.1
^{85}Rb	0.9998	0.094	4.697	9.395	
^{88}Sr	0.9999	0.051	2.538	5.077	
^{98}Mo	0.9999	0.018	0.895	1.791	47.1
^{107}Ag	0.9999	0.008	0.378	0.758	
^{111}Cd	0.9999	0.008	0.412	0.824	3.71
^{118}Sn	0.9999	0.037	1.831	3.662	
^{121}Sb	0.9999	0.011	0.562	1.124	
^{125}Te	0.9999	0.042	2.109	4.218	
^{138}Ba	0.9999	0.042	2.116	4.233	
^{139}La	0.9999	0.002	0.085	0.170	
^{140}Ce	0.9999	0.003	0.136	0.272	
^{202}Hg	0.9995	0.007	0.355	0.712	7.82
^{205}Tl	0.9998	0.002	0.087	0.175	
^{208}Pb	0.9998	0.004	0.195	0.391	10.9
^{209}Bi	0.9999	0.004	0.214	0.428	
^{238}U	0.9997	0.004	0.189	0.379	

The calibration curve accuracy was monitored throughout the sequence. Figure 2 represents the recoveries obtained for the QC check at a concentration of QC 0.004 µg·L^{-1} for Hg 0.2 µg·L^{-1} QC for all trace elements in a baby food matrix; the QC was run every ten unknown samples. For the beverage matrices, QC checks were setup at 0.2 µg·L^{-1}, 25 µg·L^{-1}, and 1 mg·L^{-1} and monitored five times in the sequence. All QC recoveries were found to be within the ±20% range in both baby food as well as beverages matrices. This is specifically important for toxic elements such as arsenic, cadmium, mercury, and lead.

Figure 2. QC recoveries (%) A. QC in baby foods - 0.004 µg·L^{-1} for Hg and 0.2 µg·L^{-1} for other trace elements; B. QC in beverages - 25 µg·L^{-1} – Trace elements in 25 µg·L^{-1}, Hg 50 times lower at 0.5 µg·L^{-1}

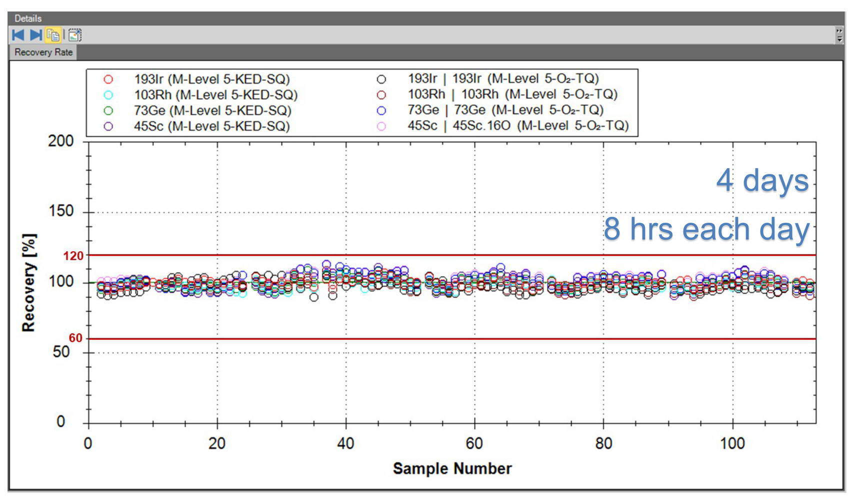


Robustness

Due to the variability of the sample composition, the instrument's ability to run all samples in one sequence was investigated thoroughly. To check the internal standard (IS) response during a working day of analysis (8 hours) and over several days, work was carried out with different beverages together on one day and different baby foods together on four different days.

The IS recovery graph is automatically generated for ^{45}Sc, ^{73}Ge, ^{103}Rh, and ^{193}Ir in both modes SQ-KED and TQ-O_2 through Qtegra Software (Figure 3).

Figure 3. Internal standard (IS) stability (Sc, Ge, Rh, Ir) for an 8-hour analysis of baby food samples



Accuracy

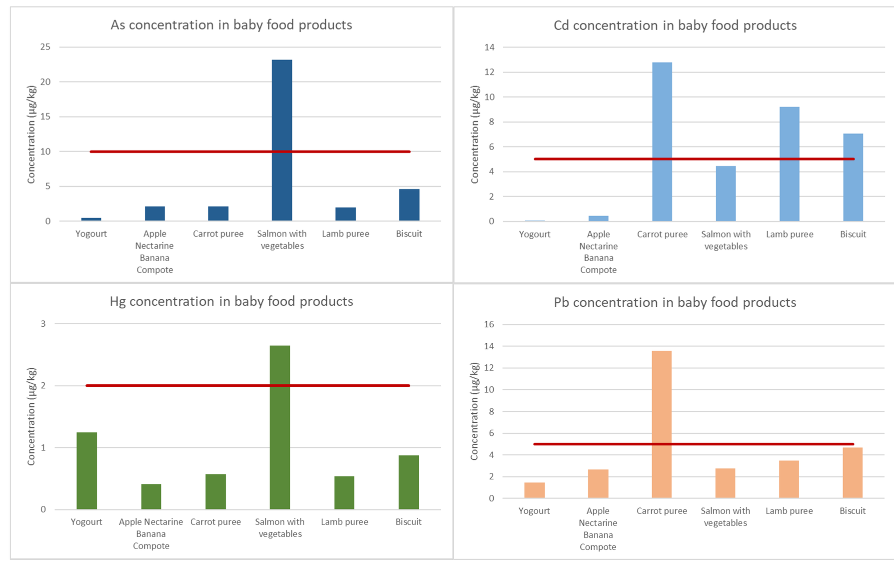
The workflow accuracy for determining the concentration levels of toxic elements was checked by the analysis of three certified reference materials. Each CRM was prepared in triplicate to evaluate the reproducibility of the sample preparation and the analysis. The iCAP MTX ICP-MS instrument shows excellent accuracy for the different CRMs. For the elements As, Cd, Hg, and Pb, the recovery calculation is between 90% and 120% for the three sample preparations, thus ensuring excellent accuracy of the results on these highly toxic elements for children.

It should be noted that the recovery calculations are also extremely accurate for chromium and selenium, which are also elements that can cause problems with children's health. At the same time, accurate recoveries within the limit of ±20% were also obtained in CRM LGC7103, which shows that the workflow not only guarantees the results on toxic elements but also meets the same requirements for essential and major elements necessary to be present at high concentrations.

Baby food samples

The results of unknown baby food samples were compared to the maximum levels of arsenic, lead, cadmium, and mercury allowed in baby food as recommended by The Baby Food Safety Act of 2021 (Figure 4). The salmon and vegetables puree contained more than 23 µg·kg^{-1} of total arsenic. While the maximum limit for inorganic arsenic is 10 µg·kg^{-1}, the presence of organic arsenic species, especially in fish, may lower toxicity concerns. Speciation analysis using ion chromatography (IC) coupled with iCAP MTX ICP-MS would be required to verify the chemical form of arsenic. The same sample also slightly exceeded the permitted mercury level, likely due to mercury accumulation in salmon. Three samples had cadmium concentrations above 5 µg·kg^{-1}, which can result from environmental contamination and agricultural activities. Additionally, the carrot puree showed lead concentrations greater than 5 µg·kg^{-1}, which is common for ground-grown vegetables in lead-contaminated soils.

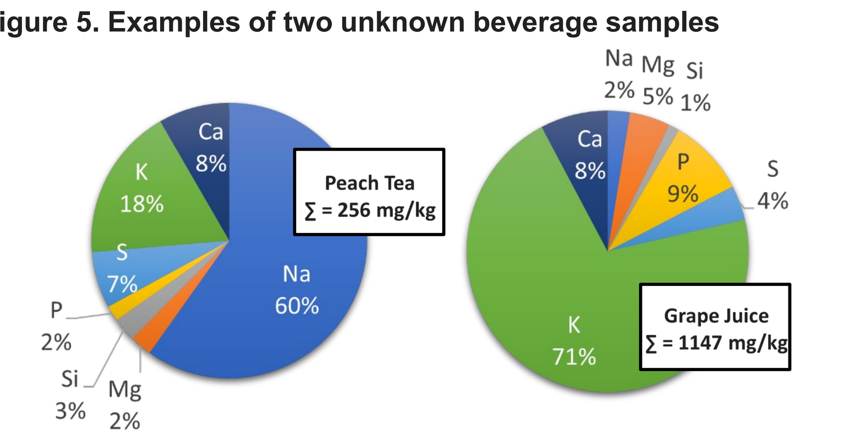
Figure 4. Concentration of As, Cd, Hg, and Pb compared with recommended maximum limits



Beverage samples

Wide range of concentrations of different elements were found in the different beverage samples – fruit juices, carbonated drinks and sweet teas – 79 to 2300 mg·L^{-1} (examples in Figure 5). This reflects the large TDS range included and successfully measured in the study without any QC or IS failures.

Figure 5. Examples of two unknown beverage samples



Conclusions

This workflow, combining sample preparation using microwave assisted digestion and analysis using the iCAP MTX ICP-MS, allows accurate measurement of the concentration of toxic elements as well as the essential nutritional elements in various food and beverage products.

- The automatic and consistent 5-times dilution enables robust analysis of various baby food products—dry, liquid, or fatty—without compromising sensitivity to meet regulated limits.
- The stability of the iCAP MTX ICP-MS allows a large variety of different food samples to be analyzed in the same run, ensuring productivity in an applied testing laboratory.
- All types of interferences (polyatomic as well as isobaric interference, including doubly charged ions) are effectively suppressed with either helium collision gas or by using oxygen as a reactive gas in triple quadrupole mode.
- The maintenance assistant of Qtegra ISDS software provides alerts for required maintenance actions, ensuring optimal instrument performance throughout its lifespan.

References

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