Trace metals analysis in baby food using triple quadrupole inductively coupled plasma mass spectrometry (TQ-ICP-MS)

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Abstract

This poster will highlight the complete and comprehensive workflow for toxic elements analysis in various baby foods. An automated and reproducible acid digestion sample preparation combined with a sensitive and robust method based on triple quadrupole ICP-MS allows a wide range of trace elements present in the samples to be quantified without any compromise in respect to applicable regulations.

Introduction

Many countries have established their own regulations and guidelines to ensure the safety and quality of baby food products. These regulations are generally 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.

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.

Since January 2022, baby food manufacturers need to meet these maximum levels for toxic elements by testing their final products (because only ingredient testing significantly underestimates toxic heavy metal levels in the final product), and to post the results of their product testing online twice per year. Similarly, as the FDA is working 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 (inductively coupled plasma mass spectrometry) is the commonly used analytical technique to measure trace elements in various samples, including baby food. The specific trace elements to be checked by ICP-MS in baby food may vary depending on the purpose of the analysis and the specific concerns related to the safety and quality of the product.

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 all 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 O_2 flow (mL·min^{-1})	0.31
RF power (W)	1550
Sampling depth (mm)	8
Number of replicates	3
Spray chamber temp (degree C)	2.7

Sample and standard preparation

Six baby food products were selected for this study and were purchased in a local supermarket. 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. Three certified reference materials were analyzed to check the method accuracy and precision. All the samples are described in Table 2.

Table 2. List of baby food samples investigated in this study

Sample	Type	Comment	Sample preparation strategy
TFV002RM - Skimmed milk powder	CRM	Carbohydrates	Dry
TM07413 - Infant cereal (rice based)	CRM	Carbohydrates	Dry
LGC7103 - Sweet digestive biscuit	CRM	Carbohydrates, fat	Dry
Yogurt	Sample	Carbohydrates, fat	Wet
Apple-Nectarine-Banana	Sample	Carbohydrates, mostly fruit sugar, dietary fiber	Wet
Carrot	Sample	Dietary fiber	Wet
Salmon	Sample	High fat and protein	Wet
Lamb	Sample	High fat and protein	Wet
Infant biscuit	Sample	Carbohydrates, fat	Dry

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.

Due to the capacity of the ultraWAVE system to digest high sample masses, and the ability of the iCAP MTX ICP-MS to analyze samples with high amounts of dissolved solid without further dilution, 0.5 g of dry or fat samples and 1.0 g of wet samples were weighed into the Quartz ultraWAVE tubes. Digested samples were finally made up to a final volume of 50 mL with ultra-pure water and were directly placed on the autosampler for analysis.

During all analyses, an internal standard 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 with a multi-element stock solution containing 34 elements. A low QC (0.2 µg·L^{-1} and 0.004 µg·L^{-1} for Hg) and a high QC (25 µg·L^{-1} and 0.5 µg·L^{-1}) were independently prepared to verify the validity of the calibration curve throughout the run. For the analysis of common nutrients such as Na, Mg, K, Ca, P, S, and Si, an additional calibration curve was prepared alongside an own QC check (1 mg·L^{-1}).

Results

Table 3 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.

Robustness

Due to the variability of the sample composition, the instrument's robustness to run all the different samples in one sequence was investigated thoroughly. This was accomplished by analyzing the different samples periodically within the same sequence. To check the internal standard (IS) response during a working day of analysis (8 hours) and over several days, work was carried out 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 Thermo Scientific™ Qtegra™ Software (Figure 2).

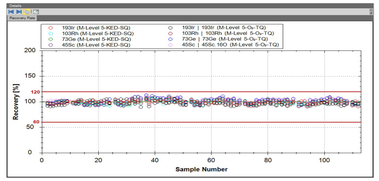

Figure 2. Internal standard stability (Sc, Ge, Rh, Ir) for 8-hour analysis

Accuracy

The workflow accuracy for determining the concentration level 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.

Table 3. Analytical figures of merit for all analytes, including R², IDL, and MQL for dry and wet sample types (as µg·kg^{-1}). 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			EAM 4.7 Nominal Limits
	R2	LOQ	MLOQ (Wet baby food)	MLOQ (Dry Baby Food)	LOQ
11B	**0.9913**	**0.013**	**0.668**	**1.336**	
23Na	**0.9999**	**0.001**	**0.072**	**0.145**	
24Mg	**0.9999**	**0.001**	**0.043**	**0.086**	
28Si	**0.9998**	**0.019**	**0.974**	**1.947**	
27Al	> 0.9999	3.161	158.0	316.1	
28Si	**0.9998**	**0.019**	**0.974**	**1.947**	
31P	**0.9999**	**0.002**	**0.079**	**0.159**	
32S	**0.9999**	**0.004**	**0.203**	**0.405**	
39K	**0.9999**	**0.026**	**1.325**	**2.651**	
44Ca	**0.9999**	**0.013**	**0.625**	**1.250**	
48Ti	0.9999	0.023	1.147	2.295	
51V	0.9999	0.091	4.550	9.101	
52Cr	0.9998	0.107	5.390	10.78	48.9
55Mn	0.9999	0.060	3.035	6.071	21.2
56Fe	0.9999	0.153	7.679	15.36	
59Co	0.9997	0.004	0.235	0.471	
60Ni	0.9985	0.372	18.63	37.26	58
63Cu	0.9995	0.093	4.690	9.381	54.7
66Zn	0.9998	0.610	30.47	60.95	340
75As	0.9999	0.054	2.718	5.436	11.6
80Se	0.9999	0.084	4.204	8.408	66.1
85Rb	0.9998	0.094	4.697	9.395	
88Sr	0.9999	0.051	2.538	5.077	
98Mo	0.9999	0.018	0.895	1.791	47.1
107Ag	0.9999	0.008	0.378	0.758	
111Cd	0.9999	0.008	0.412	0.824	3.71
118Sn	0.9999	0.037	1.831	3.662	
121Sb	0.9999	0.011	0.562	1.124	
125Te	0.9999	0.042	2.109	4.218	
138Ba	0.9999	0.042	2.116	4.233	
139La	0.9999	0.002	0.085	0.170	
140Ce	0.9999	0.003	0.136	0.272	
202Hg	0.9995	0.007	0.355	0.712	7.82
205Tl	0.9998	0.002	0.087	0.175	
208Pb	0.9998	0.004	0.195	0.391	10.9
209Bi	0.9999	0.004	0.214	0.428	
238U	0.9997	0.004	0.189	0.379	

Unknown sample analysis

The results obtained were compared to the maximum levels of arsenic, lead, cadmium, and mercury allowed in baby food recommended by The Baby Food Safety Act of 2021 (Figure 4). More than 23 µg·kg^{-1} of total arsenic was detected in the salmon and vegetables puree. While the maximum limit of 10 µg·kg^{-1} has been established for the highly toxic inorganic forms of As, it is not unusual to also find this element incorporated in organic molecules, especially in fish and seafood. These species are of far lower toxicity, so the overall amount found for As may not be of immediate concern. To verify the chemical form of As, it would be required to conduct speciation analysis using ion chromatography (IC) hyphenated to the iCAP MTX ICP-MS.

The same sample was also found to slightly exceed the permitted amount of Hg. Again here, this is most likely related to salmon, as it is well-known that mercury can accumulate in the muscle tissues of fish following its absorption from surrounding waters or from the consumption of preys that contain mercury. Thus, it is not surprising to detect more than 2 µg·kg^{-1} in fish-based baby foods.

Three samples contained cadmium concentrations above 5 µg·kg^{-1}. Cadmium is an element that is frequently found in the environment in its natural state, but due to industrial and agricultural activities it can also contaminate plants. It thus enters the food chain and can be found in baby food products. Similarly, it is not surprising to find lead concentrations greater than 5 µg·kg^{-1} in e.g. carrot puree since carrots are a vegetable that grows underground, and, depending on the region, may have grown in soil with elevetated levels of lead.

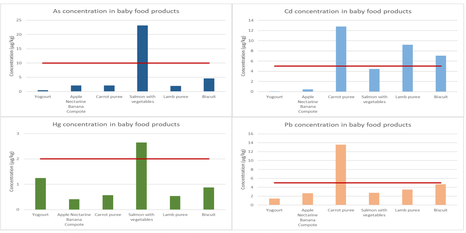

Figure 4. Concentration of As, Cd, Hg, and Pb compared with recommended maximum limits

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, dangerous for children, as well as the essential elements, necessary for the good growth and health of children, in various baby food products.

- The automatic and constant 5-times dilution allows robust analysis of different types of baby food products, whether dry, liquid, or fatty, without any compromise on sensitivity to reach the regulated limits.
- The stability of the iCAP MTX ICP-MS allows a large variety of different food samples can 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.
- Qtegra ISDS Software with the Reaction Finder functionality, allows simplified handling for the operator by automatically defining the isotopes and the acquisition modes to obtain reliable results.
- The maintenance assistant also ensures alerts to the user when actions need to be carried out by the operator to guarantee an instrument with best performance throughout instrument lifetime.

References

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