APPLICATION NOTE

000209

Using triple quadrupole ICP-MS to improve the speed, sensitivity, and accuracy of the analysis of toxic and nutritional elements in baby foods

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Goal

To demonstrate the applicability of the Thermo Scientific™ iCAP™ TQe ICP-MS incorporating a discreet sample valve for the fast, sensitive, accurate, and reliable determination of nutritional elements, at mg·kg^{-1} concentrations, and toxic trace elements, at µg·kg^{-1} concentrations, in baby food samples in a single analysis.

Introduction

Food and food supplements supply the human body not only with energy, but also essential macro- and micronutrients for a long and healthy life. While several elements, such as calcium, potassium, and phosphorous, are essential nutritional building blocks, exposure to heavy metals, like arsenic, mercury, and lead, potentially present in food as contaminants may lead to serious negative effects on health. Other elements, such as selenium, can be essential in a narrow concentration window, leading to adverse effects when they are deficient or present in excess. Thus, regular control and testing of food substances for their total nutritional and potential toxic/hazardous elemental content is essential for long-term community health.

An especially vulnerable group susceptible to different illnesses and potential lifelong neurological damage through exposure to toxic heavy metals are infants and young children between the ages of one and three years.[1] Due to their low body mass, high nutritional needs, and underdeveloped immune system, daily exposure to toxic elements, even at very low levels, may lead to brain function decline and cause negative long term effects.[2]

Several regulations exist globally for protecting the health of consumers, for example the Food, Drug, and Cosmetic Act (FDCA)[3] in the US and GB-2762-2017 in China[4]. Such regulations typically set reference values for nutrients and define permissible limits of toxic elements in foods. Within the European Union, foods for babies and young children are governed specifically by EU regulations 609/2013[1] and EC No 1881/2006[5], setting low limits for arsenic, lead, and cadmium in food products. Such regulations are subject to regular amendments and expected to become more stringent worldwide, as evidenced by the recently introduced Baby Food Safety Act of 2021[6] proposing extremely low limits of lead, mercury, etc. in baby foods. The lowest limits for toxic elements currently found globally are 10 $\mu g \cdot kg^{-1}$ for arsenic, 5 $\mu g \cdot kg^{-1}$ for cadmium and lead each, and 2 $\mu g \cdot kg^{-1}$ for mercury.

In view of the decreasing permissible limits for toxic elements in baby foods, it is critical for analytical laboratories to be able to successfully quantify these at the required levels to confidently conclude that samples comply with existing regulations and are safe for consumption. Quantifying the true concentration of toxic elements in baby foods over a "less than" threshold also requires detection and correction for any potential interferences caused by the sample matrix. It is also important to have a simplified method capable of accurately measuring both the $\mu g \cdot kg^{-1}$ levels of toxic elements and the $mg \cdot kg^{-1}$ levels of nutritional elements, ideally using a single analysis of the sample. In this study we present a simple and fast analytical method for highly accurate and sensitive analysis of both nutritional and toxic elements in different types of baby foods and reference materials. The method is based on ICP triple quadrupole MS with oxygen as the sole collision / reaction cell gas used. An iCAP TQe ICP-MS was operated in conjunction with a discreet sample valve based automation system to reduce the sample turnaround time, while increasing confidence in results.

Experimental

Instrument parameters

All measurements in this study were conducted using an iCAP TQe ICP-MS, coupled with an ASX-560 autosampler and the ASXpress® plus Rapid Sample Introduction System (Teledyne Cetac Technologies), the operation of the latter being fully integrated within the Qtegra ISDS Software. A standard aqueous sample introduction system and the high matrix skimmer cone insert were used for all measurements. The details of the sample introduction system and other parameters are listed in Table 1.

Table 1. Instrument parameters

Parameter	Value
Nebulizer	Borosilicate glass Micromist, 400 $\mu L \cdot min^{-1}$, pumped at 40 rpm
Spray chamber	2.5 mm i.d., quartz
Injector	Quartz cyclonic, cooled at 2.7 °C
Interface	Nickel sampler and nickel skimmer cone with high matrix insert
Plasma power	1,550 W
Nebulizer gas	1.13 $L \cdot min^{-1}$
QCell setting	TQ-O_2
Gas flow	100% O_2, 0.34 $mL \cdot min^{-1}$ and 0.7 $mL \cdot min^{-1}$
Scan setting	Dwell times: 0.2 s for As, Hg, Se; 0.1 s for Cd, Fe, Pb, Sb, Sn; 0.05 s for others. 5 sweeps, 3 main runs
Autosampler and discreet sample valve	CETAC ASX-560 with ASXpress
Lens setting	Optimized using autotune
Time per sample incl. 3 repeats, uptake, and rinse	1 min 19 s

All measurements were performed in a single mode using oxygen as the sole reaction gas. The instrument was tuned daily for highest sensitivity across the mass range and optimized interference removal. For all analytes not reacting with oxygen or undergoing a mass shift reaction (i.e., arsenic or selenium), the tuned oxygen flow was applied. For analytes undergoing an on-mass reaction (with the interference undergoing a chemical reaction in the CRC), the oxygen flow was further increased compared to the tuning result to improve interference removal. The full runtime was 1 minute and 19 s per sample, including three repeat measurements and uptake / wash times.

Software

The Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software was used for all data acquisition and data processing in this study. Qtegra ISDS Software is a workflow orientated software package that supports the user in all steps from method creation to data evaluation. It controls not only the instrument, but also peripheral devices or accessories such as autosamplers. For example, control and configuration of the ASXpress valve system is accomplished directly from the main user interface. Qtegra ISDS Software comes with a broad range of tools to apply quality control (QC) testing in every analysis. This includes, for example, constant control of the validity of the calibration curve using Initial Calibration Verification (ICV) and Continuous Calibration Verification (CCV) checks, as well as appropriate automated actions in the event of failure.

Additionally, spike recoveries and results for reference materials can be fully set up as QC standards, so that results are automatically calculated and reported.

Samples and certified reference materials

A total of ten infant and baby food samples comprising two milk powders (infant formula and follow-on formula), two cereals, four meal-substitute purees (organic, non-organic, vegetarian, and non-vegetarian), and two juice/fruit puree samples were purchased in a local supermarket.

A milk powder certified reference material (CRM) - BCR063 (European Commission - Joint Research Center, Belgium) and one baby food composite CRM - 2383a (National Institute of Standards and Technology, USA), as well as an infant formula reference material - TYGRM082 (Merck, Germany) were analyzed in this study. Details on the elemental composition of the CRMs and RM are described in the results section.

Sample preparation

Aliquots of 0.4 ± 0.05 g of homogenized powder sample or 2 ± 0.2 g of homogenized puree sample were weighed accurately. After the addition of 5 mL HNO_3 and 1 mL HCl (67 and 35–37 weight %, Optima™ grade, Fisher Scientific), the digestion of the samples was accomplished using an Ethos microwave digestion system (Milestone SRL, Italy). After cooling, the digested solutions were quantitatively recovered and made up to a final volume of 50 mL using ultra-pure water. All food samples and reference materials were digested using the same method, irrespective of sample type.

Additionally, four simulated samples, closely mimicking the matrix of the real baby foods and CRMs, were prepared in 2% (v/v) HNO_3 and 2% (v/v) HCl in order to be used as technical replicates in a long-term analysis alongside CRMs and real baby food sample digests. These samples included the typically abundant elements in the following concentration ranges: Ca = 7,500 $mg{\cdot}kg^{-1}$, K = 3,000–15,000 $mg{\cdot}kg^{-1}$, Mg = 500–1,500 $mg{\cdot}kg^{-1}$, Na = 200–300 $mg{\cdot}kg^{-1}$, and Fe = 100–500 $mg{\cdot}kg^{-1}$.

Standard solutions

All calibration standards were prepared using single element standards (SPEX CertiPrep, Metuchen, NJ, USA) in an acid matrix of 2% (v/v) HNO_3 and 2% (v/v) HCl with the concentrations given in Table 2. The concentration range for each analyte was selected to cover the typical concentrations expected for major elements (P, Na, K, Ca, Fe) in baby food samples as well as concentrations at least 70% below the lowest permitted limit (including dilution factors) according to current and proposed future regulated limits. The calibration range covered a total of nine orders of magnitude across all analytes investigated in this study.

An internal standard solution containing Ga, In, Tb, and Tl at 10 $\mu g{\cdot}L^{-1}$ in 2% (v/v) HNO_3 was added to all samples and standards, on-line, via the additional port available on the ASXpress discreet sample valve system.

Calibration standard 4 was used as a QC quality control standard for regularly checking the data quality during an uninterrupted analysis of a large number of samples.

Method validation: pre-digestion spiking

Food samples are a mixture of various organic compounds, macro- and minor-nutrients, essential nutritional elements in varying amounts, and sometimes trace quantities of harmful or potentially toxic elements. To ensure that relevant levels of toxic elements are detected and accurately quantified, two aliquots of one milk powder and one puree sample were spiked with 2 $\mu g{\cdot}kg^{-1}$ and 400 $\mu g{\cdot}kg^{-1}$ of As, Cd, Hg, Pb and other trace elements before digestion. The spike levels were chosen such that the expected concentrations of the spiked elements in the final sample solution would be (considering the overall dilution factor) equivalent to 0.016 $\mu g{\cdot}kg^{-1}$ and 3.2 $\mu g{\cdot}kg^{-1}$ in powdered food samples. The lower concentration equates to the lowest regulatory limit across these analytes.

Table 2. Calibration standards and their respective concentrations

	Concentrations ($\mu g{\cdot}L^{-1}$)						
Elements	**Blank**	**Std 1**	**Std 2**	**Std 3**	**Std 4**	**Std 5**	**Std 6**
Hg	0	0.005	0.010	0.050	0.100	0.500	1
Ag, As, Cd, Ce, Co, Cr, Cu, Li, Mo, Ni, P, Pb, Pt, Sb, Se, Sn, Zn	0	0.050	0.100	0.500	1	5	10
Al, B, Be, Fe	0	5	10	50	100	500	1,000
Mn	0	250	500	2,500	5,000	25,000	50,000
Ca, K, Mg, Na, P	0	500	1,000	5,000	10,000	50,000	100,000

Results and discussion

Sensitivity

The coefficient of determination (R^2), background equivalent concentration (BEC), instrument detection limit (IDL), instrumental quantification limit (ILOQ), and method limit of quantification (MLOQ) (including all dilution factors incurred during sample preparation) are listed in Table 3. The LOQ and MLOQ were calculated as follows; with IDL and BEC readily available in the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software.

$\text{LOD or IDL} = 3 \times s_0$ *Equation 1*

$\text{ILOQ} = 3\,X_{LD} = 9\,s_0$ *Equation 2*

$\text{MLOQ} = \text{method dilution factor} \times \text{LOQ}$ *Equation 3*

where s_0 is the standard deviation of the concentration of three measurements of the calibration blank.

Table 3. List of target analytes and corresponding IDLs and LOQs for individual elements in solution as well as the same in real samples (MLOQ)

Element	R^2	BEC (µg·L^{-1})	IDL (µg·L^{-1})	ILOQ = 3 × IDL (µg·L^{-1})	MLOQ in dry samples (125 × LOQ) (µg·kg^{-1})	MLOQ in puree or wet samples (25 × LOQ) (µg·kg^{-1})
^{107}Ag	0.9999	0.002	0.002	0.006	0.81	0.16
^{27}Al	0.9995	0.450	0.156	0.465	58.1	11.63
^{75}As	0.9995	0.002	0.004	0.012	1.44	0.29
^{11}B	0.9994	0.114	0.062	0.187	23.4	4.68
^{138}Ba	0.9999	0.049	0.067	0.202	25.2	5.04
^{9}Be	0.9994	0.024	0.126	0.377	47.1	9.43
^{44}Ca	0.9995	1.50	1.48	4.46	556.8	111.4
^{111}Cd	> 0.9999	0.0073	0.003	0.009	1.09	0.22
^{140}Ce	0.9997	0.0032	0.002	0.005	0.64	0.13
^{59}Co	> 0.9999	0.0009	0.001	0.004	0.49	0.097
^{52}Cr	0.9993	0.028	0.032	0.096	12.0	2.39
^{63}Cu	0.9996	0.024	0.013	0.040	5.02	1.00
^{56}Fe	0.9994	0.190	0.058	0.174	21.8	4.35
^{57}Fe	> 0.9999	0.057	0.068	0.205	25.6	5.12
^{202}Hg	> 0.9999	0.0020	0.0003	0.001	0.11	0.02
^{39}K	0.9994	2.85	0.440	1.32	165.1	33.0
^{7}Li	0.9999	0.025	0.130	0.390	48.8	9.75
^{24}Mg	0.9999	0.109	0.078	0.234	29.2	5.84
^{55}Mn	0.9994	0.013	0.009	0.026	3.19	0.64
^{98}Mo	> 0.9999	0.005	0.024	0.073	9.09	1.82
^{23}Na	0.9986	1.98	0.282	0.846	105.8	21.2
^{60}Ni	0.9993	0.060	0.033	0.099	12.4	2.48
^{31}P	0.9999	0.139	0.275	0.826	103.2	20.6
^{208}Pb	> 0.9999	0.006	0.001	0.004	0.45	0.09
^{195}Pt	> 0.9999	0.0006	0.001	0.003	0.43	0.09
^{121}Sb	0.9999	0.003	0.002	0.007	0.87	0.18
^{78}Se	0.9999	0.005	0.010	0.030	3.71	0.74
^{118}Sn	0.9998	0.015	0.003	0.009	1.11	0.22
^{232}Th	0.9994	0.746	0.776	2.327	290.9	58.2
^{238}U	> 0.9999	0.0003	0.0001	0.000	0.025	0.005
^{51}V	0.9995	0.059	0.021	0.064	8.04	1.61
^{66}Zn	0.9996	0.254	0.097	0.290	36.3	7.26

Method accuracy

The accuracy of the method was tested using the reference materials mentioned above, which were representative of the different sample types analyzed in this study. The measured concentrations of the elements showed recoveries ranging from 85 to 119% in both CRMs NIST 2383a and BCR063 (Table 4) compared to certified concentrations. The recoveries in the case of the RM TYG082fall between 86 and 119% for all elements, again well within the acceptable range (Table 4).

Method accuracy for four toxic and thirteen trace elements is additionally demonstrated by pre-digestion spiking of real baby food samples with amounts corresponding to 2 μg·kg^{-1} and 400 μg·kg^{-1}, respectively in one milk powder (125× diluted solution) and one puree sample (25× diluted solution) (Table 5). The 2 μg·kg^{-1} spike is particularly important because it equates to the lowest proposed permissible limit for the maximum amount of lead and mercury allowed in baby foods[6]. Recoveries for the 2 μg·kg^{-1} spike were between 90 and 122% for As, 93 and 105% for Cd, 84 and 108% for Hg and 98 and 101% for Pb. This was true in all cases where the spiked amount corresponded to at least 70% of the inherent concentration of the spiked element in the samples (Table 5). For the other spiked elements, with higher incurred concentration in the samples, the recoveries of the pre-digestion spikes were between 76 and 125%.

Table 4. Certified and measured elemental concentrations two baby food CRMs and one RM. Values marked with * are known reference values and not certified values.

	CRM NIST 2383a – baby food composite			CRM BCR063 – milk powder			RM TYG082 – infant formula		
Element	Certified value (mg·kg^{-1})	Determined value (mg·kg^{-1})	Recovery (%)	Certified value (mg·kg^{-1})	Determined value (mg·kg^{-1})	Recovery (%)	Certified value (mg·kg^{-1})	Determined value (mg·kg^{-1})	Recovery (%)
Ba	0.278 ± 0.020	0.243	**88 ± 1.6**	-	-	**-**	-	-	**-**
Ca	342.6 ± 5.0	377.3	**110 ± 1.6**	13490 ± 100	14205	**105 ± 4.8**	5355 ± 77	5700	**106 ± 12.6**
Ce	-	-	**-**	1.5	1.2	**83 ± 4.0**	-	-	**-**
Co	0.048 ± 0.005	0.051	**106 ± 3.9**	-	-	**-**	-	-	**-**
Cu	0.758 ± 0.082	0.648	**85 ± 3.2**	0.602 ± 0.019	0.609	**101 ± 6.2**	3.69 ± 0.08	4.00	**108 ± 8.2**
Fe	4.420 ± 0.51	5.116	**88 ± 1.5**	2.32 ± 0.23	2.02	**88 ± 1.8**	74.8 ± 1.4	80.53	**108 ± 4.0**
K	2910 ± 220	3341	**115 ± 0.4**	17680 ± 190	17556	**102 ± 4.5**	5408 ± 90	5719	**106 ± 3.2**
Mg	212.2 ± 4.0	229.0	**108 ± 0.4**	1263 ± 24	1362	**110 ± 6.3**	505 ± 7	505	**112 ± 6**
Mo	-	-	**-**	0.33*	0.25	**89 ± 2.2**	-	-	**-**
Mn	0.963 ± 0.064	1.153	**119 ± 0.5**	-	-	**-**	0.623 ± 0.022	0.739	**119 ± 4.0**
Na	195 ± 29	202.495	**104 ± 0.6**	4370 ± 31	4329	**95 ± 2.7**	1698 ± 29	1776	**105 ± 2.7**
P	453 ± 11	402	**89 ± 2.2**	11100 ± 130	10598	**95 ± 5.5**	2898 ± 60	2894	**100 ± 13.4**
Pb	-	-	**-**	0.0185 ± 0.001	0.0175	**95 ± 4.1**	-	-	**-**
Se	-	-	**-**	0.129*	0.121	**93 ± 3.4**	0.123 ± 0.007	0.106	**86 ± 3.1**
Zn	2.22 ± 0.18	1.77	**89 ± 2.0**	49 ± 0.6	50.1	**104 ± 3.9**	52.1 ± 0.9	52	**101 ± 4.4**

Table 5. Recovery for pre-digestion spikes in baby foods—milk powder and puree samples. Recovery is not reported for cases where the spiked amount was too low compared to the original concentration in the sample.

Sample	Milk powder - 1				Puree vegetable and meat based - 1			
Element	Original concentration in sample	1σ error	Spike Recovery 1 – conc. 2 µg·kg^{-1} (0.016 µg·L^{1} in solution)	Spike Recovery 2 – conc. 400 µg·kg^{1} (3.2 µg·L^{1} in solution)	Original concentration in sample	1σ error	Spike Recovery 1 – conc. 2 µg·kg^{1} (0.080 µg·L^{1} in solution)	Spike Recovery 2 – conc. 400 µg·kg^{1} (16 µg·L^{-1} in solution)
As#	6.10	*2.6*	-	**96**	0.68	*0.4*	**107**	**90**
Ba	0.18	*0.01*	-	104	0.48	*0.02*	-	117
Cd#	<DL	-	**105**	**93**	4.98	*0.3*	-	**95**
Ce	0.02	*0.01*	-	91	0.001	*0.001*	81	110
Co	0.01	*0.0003*	103	118	0.003	*0.0001*	117	120
Cr	0.04	*0.01*	-	92	0.05	*0.01*	-	93
Cu	4.01	*0.01*	-	-	0.34	*0.01*	-	115
Hg#	<DL	-	**84**	**87**	0.32	*0.03*	**108**	**84**
Mo	0.11	*0.003*	-	113	0.02	*0.001*	-	117
Ni	0.22	*0.3*	-	107	0.04	*0.01*	-	103
Pb#	2.73	*4.1*	**109**	**98**	2.30	*0.05*	-	**101**
Pt	<DL	-	95	95	<DL	-	110	95
Sb	0.42	*0.01*	-	97	<DL	-	-	95
Se	0.14	*0.04*	-	91	<DL	*0.00004*	82	76
Sn	0.24	*0.2*	-	102	0.18	*0.01*	-	107
Th	<DL	-	105	98	<DL	-	-	99
V	<DL	-	108	121	<DL	-	93	124

Values in blue are in µg·kg^{-1}. All other concentration values are in mg·kg^{-1}.

Interference removal

Of specific importance in ICP-MS analysis is to make sure that analytes are detected free from interferences. Often of concern are interferences on arsenic, both polyatomic or caused by the presence of lanthanides, like neodymium or samarium, positively biasing the results by formation of doubly charged interferences. While these interferences can be corrected for using triple quadrupole ICP-MS, or, within limits, by mathematical correction[7,8], there are other interferences that can be a potential cause of false positives. For example, molybdenum and tungsten can form $^{98}Mo^{16}O^+$ and $^{186}W^{16}O^+$ in the plasma leading to false positive data on ^{111}Cd and ^{202}Hg, respectively. The TQ-O_2 mode used in this study enables confident quantitation of the true concentration of these critical toxic elements in baby food samples through removal of such interferences. With single quadrupole instruments, the conventional kinetic energy discrimination (KED) approach used is less efficient in removing polyatomic interferences having masses larger than 100 amu. Consequently, false positive results can easily arise and the fact this is due to interferences may not be immediately noticed. The ability of triple quadrupole ICP-MS, operated with oxygen collision / reaction cell gas, to remove MoO^+ and WO^+ interferences on Cd and Hg, respectively, is illustrated for a pooled baby food sample in Table 6.

Table 6. Resolving interferences of W and Mo on toxic elements Cd and Hg, respectively, in a baby food sample using TQ-O_2 high gas flow mode.

Kinetic energy discrimination (KED)	^{111}Cd (µg·kg^{-1})	^{202}Hg (µg·kg^{-1})
Original sample	**2.41**	**0.044**
Sample + 5 µg·kg^{-1} W + 0.5 mg·kg^{-1} Mo	2.62	0.262
Sample + 10 µg·kg^{-1} W + 1 mg·kg^{-1} Mo	2.81	0.440
Sample + 20 µg·kg^{-1} W + 10 mg·kg^{-1} Mo	7.42	0.878
TQ O_2 mode		
Original sample	**2.33**	**0.047**
Sample + 5 µg·kg^{-1} W + 0.5 mg·kg^{-1} Mo	2.37	0.047
Sample + 10 µg·kg^{-1} W + 1 mg·kg^{-1} Mo	2.32	0.047
Sample + 20 µg·kg^{-1} W + 10 mg·kg^{-1} Mo	2.45	0.050

Note that both conventional analyses using kinetic energy discrimination and the proposed method using TQ-O_2 mode return identical results for the unspiked sample. However, even with low concentrations of molybdenum and tungsten present in the sample, false positive results arise on the critical elements Cd and Hg. These interferences can only be efficiently removed using triple quadrupole technology.

Robustness validation and real sample analysis

A critical requirement for analytical laboratories is to maintain the high sensitivity, accuracy, and precision during the analysis of long sequences of hundreds of samples. Thus, instrument performance was tested by analysis of a long sequence of injections.

An extended batch measurement was started with 10 calibration blanks and a single calibration block. An ICV was carried out by immediately following this by the analysis of Std 4, which was used as a QC standard throughout the experiment. Subsequently, blocks of ten unknown samples, which included two real baby food samples and eight simulated samples with matched matrix composition, were analyzed. The QC standard was repeated after each block of 10 unknown samples as a CCV to ensure the validity of the calibration. For the determination of accuracy throughout the experiment, a NIST 2383a CRM sample was also regularly analyzed.

The recovery of the internal standard, QC samples, and CRMs remained stable throughout the experiment with recoveries between 75 and 110% (Figures 1, 2, and 3, respectively). After 8 hours of continuous measurements, only gallium (one of the internal standards, denoted by red circles in Figure 1) started to drift negatively outside this range. Almost all QC checks were found to be within 80 to 120% over the full 11 hours of continuous analysis. The results for individual analysis of NIST CRM 2383a are displayed in Figure 3, and again show that the results match the assigned concentrations for this reference material, demonstrating the potential of the proposed method to deliver accurate results even during extended unattended analysis.

The concentrations obtained for major and trace elements in the ten baby food samples are presented in Table 7.

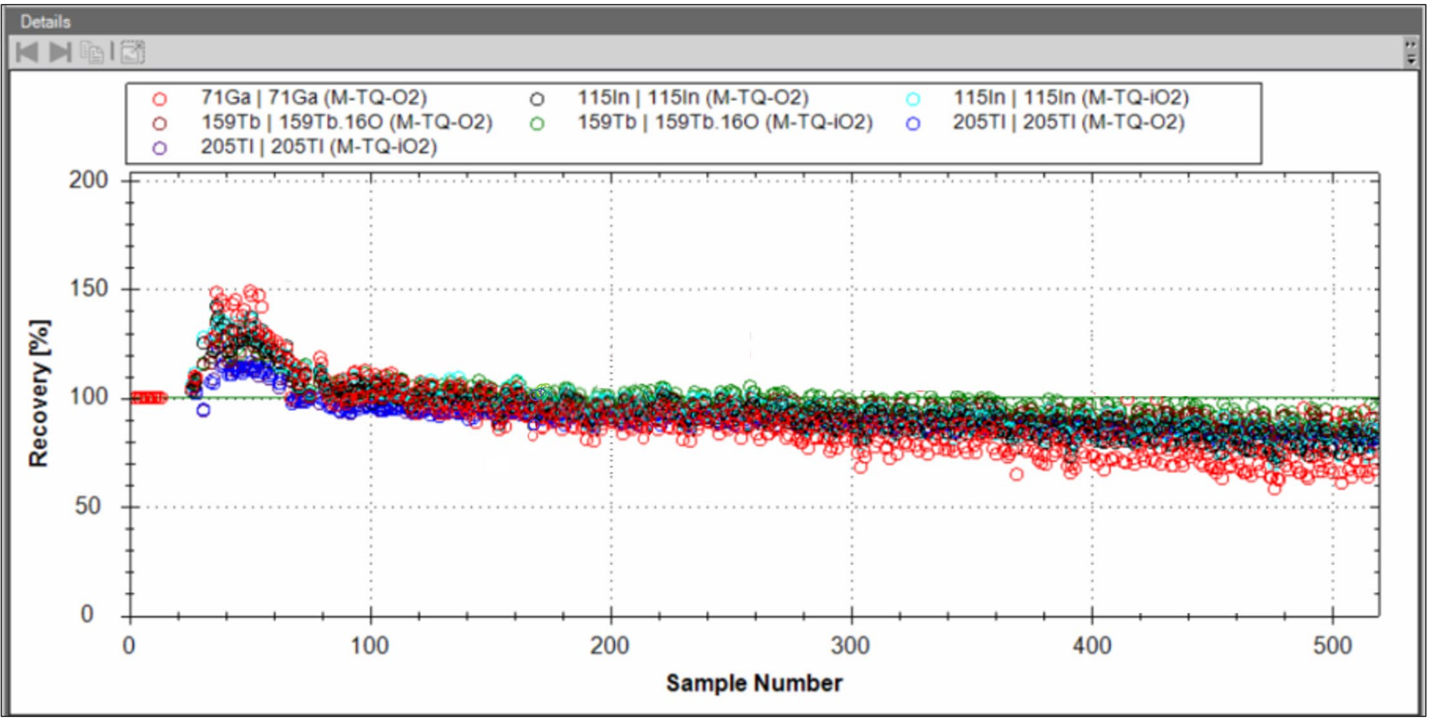

Figure 1. Internal standard recovery in real samples during an extended run covering 11 hours. The increase of the internal standard response between samples 25–60 is due to initial conditioning of the cones with the sample matrix.

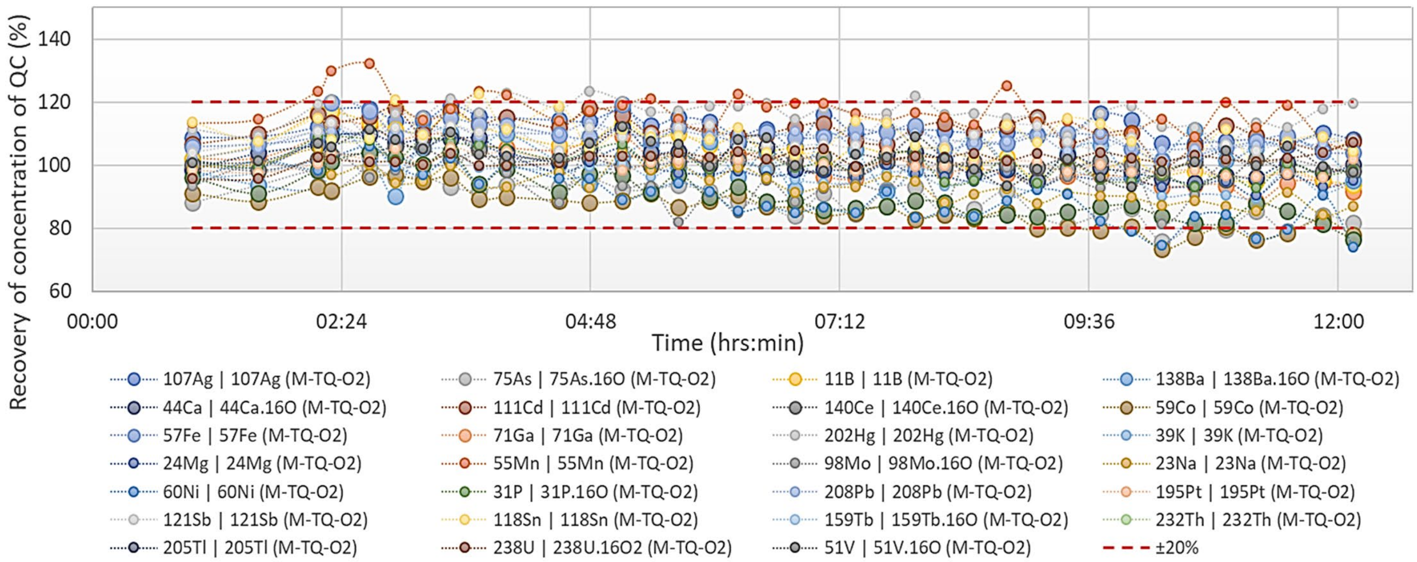

Figure 2. CCV recoveries for all analytes at a concentration equal to standard solution 4. The dashed line represents the accepted recovery interval (± 20%).

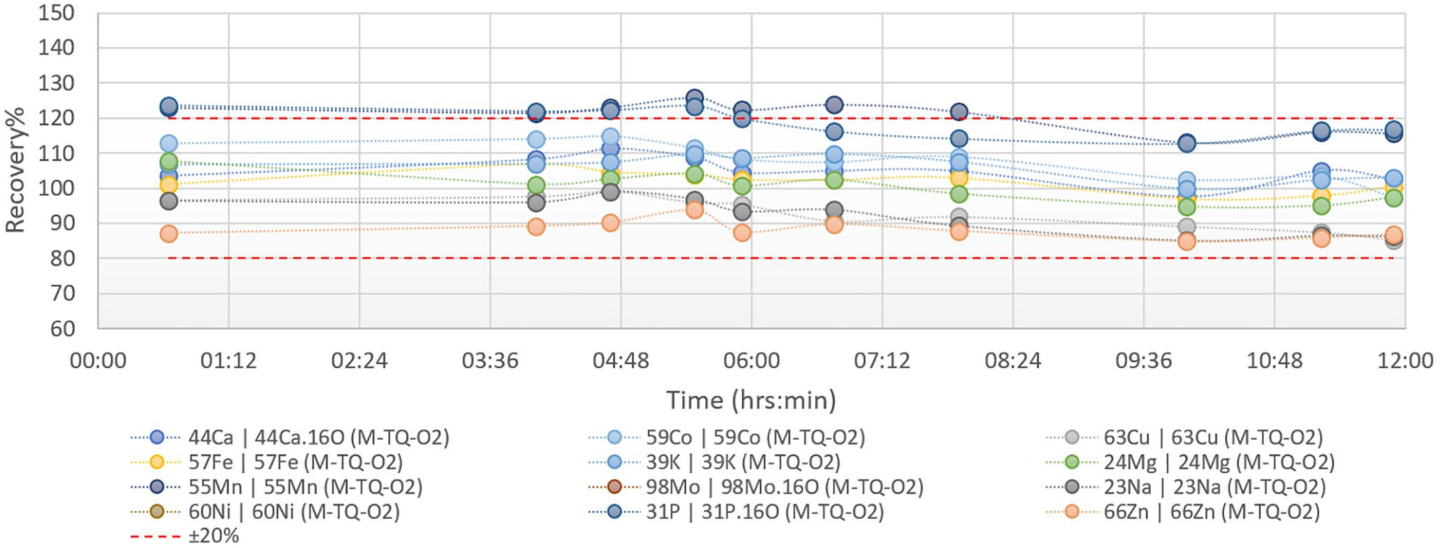

Figure 3. Trueness: the results obtained for the analysis of CRM 2383a throughout an extended analysis

Table 7. Compositions of ten different baby food samples. Values of As, Cd, Hg, and Pb (in blue) are in µg·kg^{-1}, whereas all other values are in mg·kg^{-1}.

Element	Milk – I, infant formula powder	Milk – II, follow-on formula powder	Puree – I, vegetables and meat base	Puree – II, vegetable base	Organic puree – I, vegetable base	Organic puree – II, vegetable and meat base	Juice/fruit puree – I	Juice/fruit puree – II	Cereal – I	Cereal – II
Ag	<DL	<DL	0.000	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Al	3.46	1.44	<DL	0.46	0.53	0.36	1.30	1.67	1.83	1.09
As	6.10	4.77	0.68	1.42	<DL	1.53	1.34	1.44	9.10	34.86
B	0.36	0.52	1.16	<DL	1.26	0.92	<DL	<DL	<DL	<DL
Ba	0.18	0.19	0.48	2.30	1.41	0.53	0.95	3.16	10.56	1.25
Ca	4036	4929	310	70	223	172	61	108	268	5405
Cd	N.A.	N.A.	4.98	1.38	N.A.	N.A.	<DL	<DL	<DL	4.71
Ce	0.02	0.004	0.001	0.001	0.001	0.001	0.002	0.001	0.002	0.003
Co	0.01	0.01	0.003	0.002	0.004	0.001	0.003	0.003	0.014	0.007
Cr	0.04	0.04	0.05	0.04	0.05	0.02	0.04	0.01	0.02	0.05
Cu	4.01	3.92	0.34	0.61	0.47	0.31	0.35	0.83	3.24	0.85
Fe	32.69	55.03	1.45	0.00	0.27	0.26	1.66	3.17	20.64	76.93
Hg	<DL	N.A.	0.32	0.07	N.A.	N.A.	0.06	0.06	0.26	0.80
K	5491	5512	1745	1215	2351	2037	819	2954	3091	5196
Li	<DL	0.01	0.01	0.01	0.01	3.24	<DL	<DL	0.01	0.01
Mg	381	438	0.11	142	109	112	49	303	772	462
Mn	<DL	0.42	0.67	3.13	1.24	0.67	1.05	2.67	33.79	4.56
Mo	0.11	0.12	0.02	0.05	0.03	14.79	0.03	0.06	0.52	0.24
Na	1631	1419	101	6.70	92	220	4.21	2.77	8.23	999
Ni	0.22	0.11	0.04	0.07	0.07	9.60	0.03	0.04	0.16	0.08
P	2920	3100	402	218	250	220	69	280	1874	2905
Pb	2.73	5.85	2.30	2.83	4.33	3.45	2.13	1.61	5.56	6.22
Pt	<DL	<DL	<DL	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Sb	0.42	0.11	<DL	<DL	<DL	0.48	<DL	<DL	<DL	0.001
Se	0.14	0.15	<DL	<DL	<DL	0.09	<DL	<DL	0.04	0.09
Sn	0.24	0.02	0.18	0.05	0.07	0.05	<DL	0.003	0.04	0.06
Th	<DL	<DL	<DL	<DL	<DL	<DL	<DL	<DL	<DL	<DL
U	<DL	<DL	<DL	<DL	<DL	<DL	<DL	<DL	<DL	<DL
V	<DL	<DL	<DL	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Zn	34.34	35.18	2.14	1.65	1.67	2.11	0.26	1.72	20.03	16.54

Conclusions

The current study presents a simple, fast, highly sensitive, and accurate analytical methodology for analyzing a suite of elements typically monitored in foods intended for babies and young children. The results from the different experiments demonstrate the iCAP TQe ICP-MS can provide confidence in the analysis results of nutritional and toxic elements in a variety of different baby foods in a single analysis. This confidence can be achieved consistently in analytical testing laboratories where long, daily measurement sequences consisting of hundreds of samples need to be turned around.

The most important results and highlights of this study are summarized below:

- A single method allowing the analysis of 30 elements, covering nutritional and toxic elements in baby food, was developed and tested.
- All analytes were measured using a single measurement mode on the iCAP TQe ICP-MS using oxygen as the collision / reaction cell gas. This resulted in highly sensitive and accurate analysis of all analytes of interest, including the potentially challenging analytes arsenic, selenium, cadmium, and mercury, which were measured free of any interferences with high precision.
- A wide linear dynamic range, starting from 0.005 $\mu g.kg^{-1}$ for trace elements and extending up to 100,000 $\mu g.kg^{-1}$ for major elements, was achieved, enabling the analysis of all 30 elements in a single measurement without any extra dilution or calibration steps.
- Excellent detection limits of <1 $\mu g{\cdot}L^{-1}$ (sub-ppb or ppt level) for 29 elements and 1.5 $\mu g{\cdot}L^{-1}$ for the remaining element calcium were achieved. Limits of quantification for the toxic elements As, Cd, Hg, and Pb in real samples (MLOQ) were at least 70% lower than the lowest global regulatory limits.
- Excellent interference removal was demonstrated not only by the results obtained for the reference materials analyzed in this study, but also through the intentional addition of elements known to cause interferences. At the same time, detection limits were absolutely comparable to established methods, even for analytes that do not readily benefit from the use of oxygen as a reactive gas.
- Method validity and accuracy were demonstrated with the analysis of appropriate CRMs and RMs, typically obtaining a result between 85 and 120% of the certified concentration. Accuracy of determination was confirmed by pre-digestion spiking, which yielded recoveries between 80 and 125%.
- The Qtegra ISDS Software serves as a single solution to all data acquisition and data processing needs of the application. Different automatic evaluations of the acquired data, for example, analytical figures of merit or QC checks are all automated, eliminating the need for additional manual evaluation.
- The iCAP TQe ICP-MS proved to be an extremely robust system capable of producing reliable and accurate data, as demonstrated over at least 11 hours of uninterrupted measurements of high matrix baby foods and CRM samples.
- The combination of iCAP TQe ICP-MS with the CETAC ASXPRESS discreet sample valve module reduced the analysis time enabling a very quick run time of 1 minute 19 s per sample. This ensures high sample throughput, comparable to well-established methods using single quadrupole ICP-MS, making this setup perfectly suited for high sample throughput analytical testing laboratories.

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