Application Note
Food

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Analysis of Inorganic Arsenic, Cadmium, Lead, and Mercury in Baby Foods by ICP-MS

Addressing action levels in US Baby Food Safety Act 2021 using an Agilent 7850 ICP-MS and HPLC-ICP-MS

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Introduction

During the critical early stages of physical and neurological development, babies and young infants require access to safe and nutritious food. However, an investigative committee in the U.S. House of Representatives issued a report in February 2021 showing that many baby foods sold in US supermarkets contained unacceptably high concentrations of As, Cd, Pb, and Hg (*1*). Infants and children are at greater risk from food contaminants because they consume more food than adults do relative to body weight and their diets tend to be less varied.

To protect the health and development of babies and infants, and to assure parents and caregivers about the safety of manufactured baby foods, the US government has proposed new, lower limits for trace elements in food. The Baby Food Safety Act 2021 (*2*) sets out maximum levels of inorganic As (the most toxic form of As), Pb, Cd, and Hg permitted in baby foods sold in the US (Table 1). There are different levels for cereal and non-cereal based foods since cereal plants are known to accumulate heavy metals if they are grown in contaminated agricultural conditions (*4*). Rice plants are especially efficient at accumulating heavy metals because the flooded fields in which they are grown make it easier to take up metal compounds (*5*).

Table 1. Proposed maximum allowable levels of toxic heavy metals in infant and toddler foods. Units: ppb.

Element	Proposed Action Level in Baby Food Safety Act 2021		Current FDA Action Levels or Current Guidance Levels for Baby Foods
	Cereal	Non-Cereal	
*Inorganic arsenic (iAs)	15	10	100 for infant and toddler rice (*3*)
Cadmium	10	5	None
Lead	10	5	None
Mercury	2	2	None

**Speciation analysis for iAs commonly uses HPLC-ICP-MS.*

Before the legislation is passed, baby food manufacturers are encouraged to be proactive, and to test all ingredients for heavy metals before use, as well as testing finished products. If current ingredients do not meet the new levels, manufacturers are expected to find alternatives or to reformulate their products. In response to the Act, the US Food and Drug Administration (FDA) has published the Closer to Zero plan. The aim of the plan is to reduce the levels of As, Pb, Cd, and Hg in foods consumed by babies and young children per the following timeframe (*6*):

- Phase 1: April 2021 – April 2022: propose a draft action level for Pb.
- Phase 2: April 2022 - April 2024: finalize action level for Pb. Propose draft action level for As.
- Phase 3: April 2024 – beyond: finalize action level for As. Propose draft action levels for Cd and Hg.

Meeting any final action levels will require a sensitive analytical technique to achieve low detection limits for total element concentrations in complex food matrices and speciation analysis for iAs. ICP-MS is the obvious choice, due to its sensitivity and ability to handle varied food matrices. ICP-MS can also be easily connected to HPLC for the speciation analysis of iAs.

For guidance on method development and method validation for the analysis of foods by ICP-MS, laboratories can refer to the FDA Elemental Analysis Manual (EAM). Section 4.7 of the EAM describes how to determine 12 elements in food by ICP-MS, including As, Cd, Pb, and Hg, following microwave-assisted acid decomposition. EAM 4.7 also outlines a series of quality control (QC) tests to ensure instrument performance and data accuracy (*7*).

Inorganic arsenic

Under the proposals in the Baby Food Safety Act, food manufacturers will be required to analyze all baby foods for total As content. Samples that contain more than 10 ppb (or 15 ppb for cereal-based foods) of total As would then be analyzed for iAs to assess compliance with the action limit for iAs. FDA EAM section 4.11 recommends the use of HPLC-ICP-MS to determine iAs (as the sum of As(III) and As(V)) in infant rice cereals (*8*).

The proposed action levels and public concern about baby food contamination are expected to increase demand for routine monitoring of As, Cd, Pb, Hg, and iAs in baby foods.

ICP-MS and HPLC-ICP-MS

Agilent has been providing robust and reliable solutions for the measurement of multiple elements by ICP-MS and speciation studies requiring HPLC-ICP-MS for many years. The Agilent 7850 ICP-MS with Octopole Reaction System (ORS^4) is an ideal instrument for food-testing laboratories, including labs that are new to the ICP-MS technique or new to Agilent systems. The 7850 combines proven hardware capabilities with helpful software features that simplify all aspects of the analytical workflow.

The 7850 controls common spectral interferences using helium (He) collision cell mode and Kinetic Energy Discrimination (KED), while doubly-charged interferences can be addressed using half-mass correction in the ICP-MS MassHunter software (*9, 10*). These methods lead to more accurate results, reducing the need for sample remeasurements. The 7850's 10 orders linear dynamic range also simplifies method setup, as major and trace analytes can be measured in a single run, meaning fewer reruns due to over-range results. The 7850 features Agilent Ultra High Matrix Introduction (UHMI) aerosol dilution technology as standard. UHMI improves plasma robustness, enabling the 7850 to handle samples with percent level dissolved solids (TDS) content (*11*). Analysts can find out the TDS content of new sample types within a few seconds using the IntelliQuant function in ICP-MS MassHunter software (*12*). IntelliQuant is based on the QuickScan full mass spectrum data, enabling the identification and confirmation of semiquantitative results for up to 78 elements. IntelliQuant's periodic table “heat map” view provides a quick and simple overview of the concentration of all elements within the sample.

The 7850 ICP-MS also links with Agilent chromatography systems, such as the Agilent 1260 HPLC, using an optimized interface and integrated software control. The coupled system is set up and operated from the ICP-MS MassHunter software, giving simple, automated analysis.

This study describes the use of the 7850 ICP-MS and Agilent SPS 4 autosampler for the analysis of critical elements in different baby food samples using a simple, single He cell gas method. The list of elements included the 12 elements that are specified in EAM 4.7: arsenic, cadmium, chromium, copper, lead, manganese, mercury, molybdenum, nickel, selenium, thallium, and zinc. The quality of the data for these elements was assessed through the measurement of three food standard reference materials (SRMs), a fortified method blank (FMB), and two fortified analytical portions (FAPs).

Experimental

Calibration standards

The calibration standards were prepared in 3% nitric acid (HNO_3) and 0.5% hydrochloric acid (HCl). HCl is routinely added to samples being prepared for ICP-MS analysis, as it ensures that chemically unstable elements such as Hg are retained in solution. Any Cl-based polyatomic overlaps formed are easily controlled on Agilent ICP-MS systems using the standard He cell mode. Calibration standards were prepared from Agilent standard solutions including environmental calibration standard, p/n 5183-4688, multi-element calibration standard-1, p/n 8500-6944, and 1000 µg/mL single calibration standard for Hg, p/n 5190-8485. Most elements were calibrated from 0.1 to 25 ppb. Cu, Mn, and Zn were calibrated up to 250 ppb. Hg was calibrated from 0.01 to 2.5 ppb. Continuing calibration verification (CCV) standards were prepared at 1 ppb (2 ppb for Hg), and/or 10 ppb.

An Agilent internal standard (ISTD) solution (p/n 5188-6525) containing 2 ppm Sc, Ge, Rh, In, Tb, Lu, and Bi, was prepared in 1% HNO_3, 0.5% HCl, and 10% isopropanol (IPA). Per the 4.7 method, IPA was added to the ISTD to help ensure consistent sensitivity for As and Se, as ionization of these elements is affected by variable levels of residual carbon in the digested samples. The ISTD solution was added automatically online at a flow rate approximately 16 times lower than the sample flow.

Reference materials and samples

Three varied food matrix SRMs from National Institute of Standards and Technology (NIST, Gaithersburg, US) were used to validate the method. The SRMs were NIST 2383a Baby Food Composite, NIST 1546a Meat Homogenate, and NIST 2385 Slurried Spinach.

A good representation of fruit, vegetable, and meat products packaged in different types of containers such as glass, plastic tubs, and pouches were bought in a supermarket in North Carolina, USA. The products included single foods like green beans or mango, blends such as broccoli, carrot, banana, pineapple, and an "organic" beef medley. Some popular brand products and store-own branded foods were included.

Standard and sample preparation

The samples were prepared for analysis according to the digestion procedure outlined in the EAM 4.7 method using a MARS 6 iWave closed-vessel microwave digestion system (CEM Corporation, Matthews, NC US). All of the foods were already puréed, so no further sample preparation was necessary before digestion. However, baby foods contain a high moisture content, and the moisture content of the foods included in this study ranged from 60 to 93%. 2 g of the pureed sample was calculated to be equivalent to dry weights between 0.14 g and 0.8 g.

2 g of each of 11 food samples and three SRMs was accurately weighed into a 75 mL PFA MARS Xpress vessel. 8 mL of HNO_3 and 1 mL of 30% H_2O_2 was added to each vessel. Duplicates of the samples, SRMs, spiked samples, method blanks, and fortified method blanks (method spikes), 40 vessels in total, were prepared and digested in a single batch, using the heating program shown in Table 2. Finally, 0.5 mL concentrated HCl was added to the digests, followed by de-ionized water to a final weight of 100 g.

Table 2. Microwave digestion parameters.

Parameter	Setting
Power (W)	1800
Ramp Time (min)	25
Hold Time (min)	15
Temperature (°C)	200
Cooling Time (min)	~20

Fortified analytical portions

Two FAPs were prepared to test the robustness of the sample preparation method. Before digestion, the two FAP samples - a beef medley and a blend of broccoli, carrot, banana, and pineapple - were each fortified in duplicate with a low-level spike and a high-level spike as shown in Table 3.

Table 3. Spike concentrations used for the fortified analytical portion test.

Analyte	Low-Level Spike Conc (µg/kg)	High-Level Spike Conc (µg/kg)
Hg	0.1	50
As	10	50
Cr	1	200
Ni	1	50
Cd	1	50
Ba	1	50
Tl	1	50
Mo	1	200
Se	1	200
Pb	1	200
Mn	5	200
Cu	10	200
Zn	10	200

The analytical sequence of calibration standards, samples, and QC solutions is shown in Figure 1. The sample block was analyzed repeatedly with automatic insertion of the periodic QC block after every 10 samples.

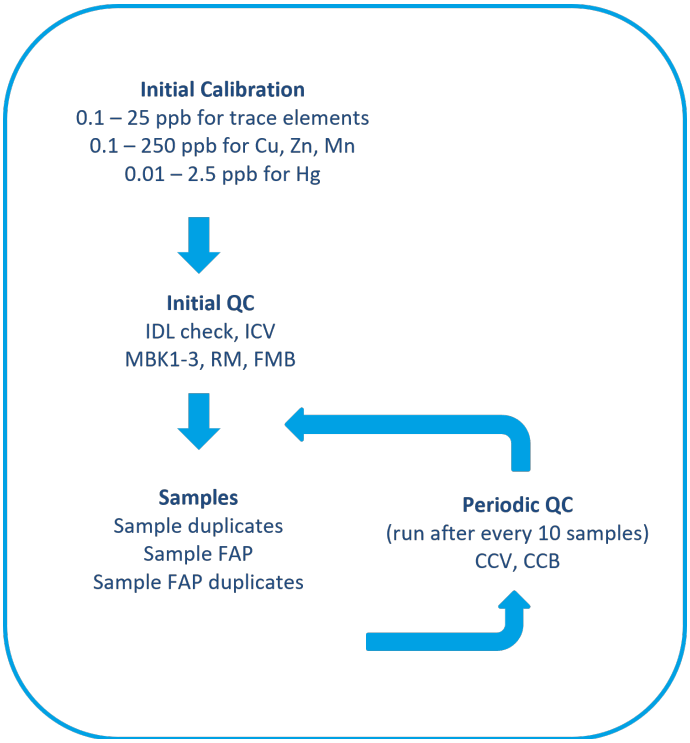

Figure 1. Analytical sequence. Key: Instrument detection limit (IDL), initial calibration verification (ICV), method blank (MBK), reference material (RM), fortified method blanks (FMB), fortified analytical portion (FAP), continuing calibration verification (CCV), continuing calibration blank (CCB).

Instrumentation

An Agilent 7850 ICP-MS equipped with the ORS^4 collision cell and UHMI was used for the analysis. Sampling was performed using an Agilent SPS 4 autosampler. The 7850 ICP-MS was configured with the standard sample introduction system consisting of a MicroMist glass concentric nebulizer, quartz spray chamber, and quartz torch with 2.5 mm id injector. A nickel plated sampling cone with a copper core was used, together with a nickel skimmer cone.

The IntelliQuant function in the ICP-MS MassHunter software performs a full mass-spectrum scan with only two seconds additional measurement time. IntelliQuant was used to check the concentration of Rare Earth Elements (REEs), along with most of the other elements in the periodic table, in the baby food samples. REEs have relatively low second ionization potentials, so form a small percentage of doubly charged ions (M^{2+}) in the plasma. If REEs such as Nd, Sm, Gd, and Dy are present in a sample at a high enough concentration, M^{2+} interferences can affect the accuracy of the measurement of arsenic (As) and selenium (Se). Therefore, the EAM 4.7 method recommends that analysts monitor the following isotopes: ^{146}Nd, ^{147}Sm, ^{155}Gd, ^{163}Dy. Selecting half-mass correction in the ICP-MS MassHunter method wizard enables real-time correction of unknown samples that may contain REEs at high enough concentration to cause M^{2+} interferences (*10*).

The IntelliQuant results also provided valuable information about the total matrix solids (TMS) level of the food samples. The TMS function is especially useful when dealing with unknown and potentially complex food samples by helping the analyst to decide if a sample needs to be diluted or a higher UHMI setting is needed. The measured TMS levels for the samples analyzed in this study are shown in Table 4.

Table 4. Total matrix solids data for baby food samples (n=2, in duplicate) obtained by the TMS function of ICP-MS MassHunter. Units: ppm

Beef Medley	Green Beans	Mango	Chicken Noodle	Broccoli, Carrot, Banana, Pineapple	Banana, Carrot, Strawberry	Apple, Pear, Green Pea	Pear, Apple, Broccoli	Carrot, Zucchini, Broccoli	Sweet Potato, Turkey	Prunes
37.7	35.8	29.8	32.9	51.5	53.5	24.5	26.3	36.8	44.1	51.5

Other instrument operating settings were optimized automatically using the ICP-MS MassHunter autotune function. All analytes were acquired in He mode (enhanced He mode for As and Se). Based on the TMS levels (Table 4), plasma setting HMI-4 was used, which applies an aerosol dilution factor of four times to the sample aerosol. When UHMI is selected, all related settings are autotuned as appropriate for the matrix levels of the target sample types. Instrument operating conditions are listed in Table 5.

Table 5. ICP-MS operating conditions*.

ICP-MS Parameter	Setting
RF Power (W)	1600
Sampling Depth (mm)	10
Nebulizer Gas Flow (L/min)	0.6
Dilution (UHMI) Gas Flow (L/min)	0.35
Lens Tune	Autotune
Helium Cell Gas Flow (mL/min)	4.3 (10**)
Energy Discrimination (V)	5 (7**)

** Shaded parameters are defined in the method and HMI-4 plasma presets; all parameters were automatically optimized during start-up and autotuning. ** Enhanced He mode settings used for As and Se.*

Results and discussion

Typical 7850 ICP-MS detection limits (DLs) calculated from the ICP-MS MassHunter calibrations are shown in Table 6. The EAM method limits of detection (LOD) - also known as method detection limits (MDL) - and quantification limits (LOQ) were calculated based on method blanks measured at the end of the run, n=10 (*13*).

Data was acquired for the 12 elements required by EAM 4.7, including As, Cd, Hg, and Pb, using only helium as a cell gas. For all elements, the 7850 ICP-MS analytical limits are lower by around one or two orders of magnitude compared to the nominal limits provided in EAM 4.7.

Table 6. Agilent 7850 detection limits and EAM 4.7 nominal analytical limits.

Element	ISTD	ICP-MS MassHunter	Calculated Nominal Analytical Limits	
		LOD (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
^{52}Cr	^{103}Rh	0.0329	1.079	3.597
^{55}Mn	^{103}Rh	0.0343	0.344	1.147
^{60}Ni	^{103}Rh	0.0105	0.741	2.470
^{63}Cu	^{103}Rh	0.0112	0.138	0.461
^{66}Zn	^{103}Rh	0.2961	3.502	11.674
^{75}As	^{74}Ge	0.0235	0.628	2.094
^{78}Se	^{103}Rh	0.0259	0.686	2.286
^{95}Mo	^{103}Rh	0.0034	0.400	1.335
^{111}Cd	^{103}Rh	0.0042	0.087	0.289
^{201}Hg	^{209}Bi	0.0013	2.747	9.156
^{205}Tl *	^{209}Bi	0.0055	0.450	1.502
Pb ‡	^{103}Rh	0.0438	0.540	1.799

*All elements were acquired in He mode (enhanced He mode for As and Se). The Nominal Analytical Limits are given in EAM 4.7 and are based on method blanks measured during the single lab validation over one year; n=143. *Based on a single lab validation (n=27).*
‡ Sum of Pb isotopes.

Verification of instrument calibration and sample digestion process

As part of the method quality control procedure specified in EAM 4.7, and to ensure the ongoing validity of the calibration, a CCV standard was analyzed seven times during the analytical sequence. As shown in Figure 2, all elements in the seven CCVs and the initial ICV were recovered within the EAM acceptance criteria of ±10% of the actual concentration.

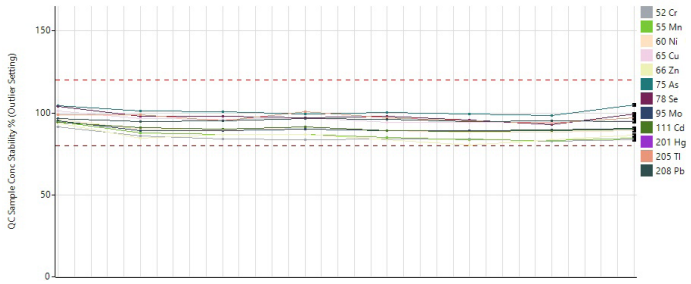

Figure 2. CCV recoveries over the course of the 48-hour sequence, including at the end of the analytical sequence.

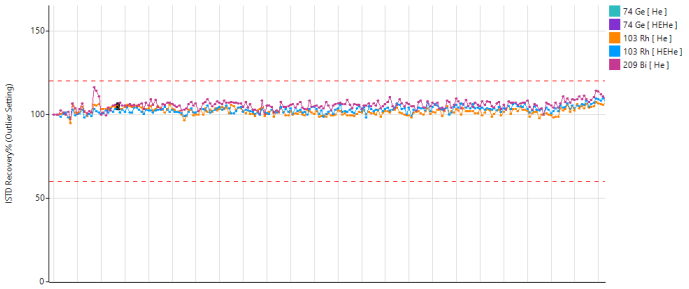

Figure 3. Stability of ISTD measurements over 48 hours. The ISTD recoveries have been normalized to the calibration blank for all samples. All measurements were within the method requirements of 60–120%.

To verify the sample digestion process, each of the three NIST SRMs was prepared in duplicate and each preparation was analyzed twice (with three replicates per analysis) using the 7850 ICP-MS. As shown in Table 7, the mean concentrations were in good agreement with the certified concentrations, meeting the QC criteria requirements of the FDA EAM method of 80–120%. Since not all SRMs are certified for all analytes, blank cells indicate the absence of a certified or reference value.

Instrument robustness and stability: ISTD recovery (%)

The analytical sequence outlined in Figure 1 was analyzed repeatedly over 48 hours. All the ISTD recovery plots were within ±20%, with no internal standard failures throughout the run, meeting the criteria specified in EAM 4.7 (Figure 3). The results demonstrate the robustness of the plasma and high matrix tolerance of the 7850 ICP-MS with UHMI over long runs.

Spike recoveries

A spike recovery (fortified analytical portion - FAP) test was carried out to check the sample preparation method and the accuracy of the 7850 ICP-MS analysis of baby foods, especially for elements not present in the SRMs. Two food samples (beef medley and a product made from a blend of broccoli, carrot, banana, and pineapple) were selected at random. Both samples were spiked with all elements as detailed in Table 3 and measured using ICP-MS. The recoveries for all elements in the fortified food samples were within the EAM 4.7 method QC criteria of ±20%, as shown in Table 8.

Table 7. Mean measured concentrations in three food SRMs corrected for dilution, n=2.

	NIST 2383a Baby Food Composite			NIST 1546a Meat Homogenate			NIST 2385 Slurried Spinach		
Element	Certified Conc (µg/kg)	Measured Conc (µg/kg)	Recovery (%)*	Certified Conc (µg/kg)	Measured Conc (µg/kg)	Recovery (%)*	Certified Conc (µg/kg)	Measured Conc (µg/kg)	Recovery (%)*
^{55}Mn	963	972	101	286	285	100	3810	3397	89
^{63}Cu	-	-	-	605	602	100	17100	16556	97
^{66}Zn	758	749	99	17880	18990	106	900^{R}	818	-
^{75}As	2220	2156	97	-	-	-	-	-	-
^{78}Se	-	-	-	281	301	107	8370	6956	83
^{98}Mo	28^{R}	26	-	16^{R}	19	-	-	-	-

** FDA Elemental Analysis Manual (Section 3.4 Special Calculations) 3.4 Equation 20. ^{R} Noncertified reference value.*

Table 8. Mean recovery results based on the analysis of baby food sample digests. Mean calculated from two separate digests, each measured twice in triplicate.

	Measured Concentration Beef Medley	Fortified Analytical Portion (Beef Medley)		Measured Concentration Fruit/Veg	Fortified Analytical Portion (Fruit/Veg)	
Element	ppb	Low Spike Recovery ± 1σ (%)	High Spike Recovery ± 1σ (%)	ppb	Low Spike Recovery ± 1σ (%)	High Spike Recovery ± 1σ (%)
^{52}Cr	31.19 ± 0.54	99	90	34.49 ± 0.71	96	87
^{55}Mn	631.41 ± 3.46	*	92	6434.48 ± 94.93	*	83
^{60}Ni	41.13 ± 1.97	102	94	116.61 ± 1.40	115	90
^{63}Cu	366.66 ± 5.16	87	96	556.66 ± 9.01	106	92
^{66}Zn	4865.77 ± 29.49	*	91	1788.90 ± 29.19	*	89
^{75}As **	3.84 ± 0.25	104	100	<LOQ	101	97
^{78}Se**	12.75 ± 0.74	96	93	5.68 ± 0.70	93	92
^{95}Mo	19.12 ± 0.42	98	93	35.56 ± 0.80	96	90
^{111}Cd	7.04 ± 0.37	97	95	1.69 ± 0.15	96	92
^{137}Ba	311.13 ± 8.42	86	94	315.15 ± 4.63	94	90
^{201}Hg	<LOQ	97	117	<LOQ	98	94
^{205}Tl	<LOQ	95	93	<LOQ	95	92
Pb ‡	<LOQ	92	97	<LOQ	99	96

**Spike levels were too low (<5%) relative to the unspiked concentration. ** Enhanced He and half mass correction were used for As and Se. ‡ Sum of Pb isotopes.*

Quantitative results for baby foods

Quantitative results for beef medley and the blend of broccoli, carrot, banana, and pineapple (fruit/veg) are given in Table 8. Data for nine more baby foods are given in Table 9. In addition to the 12 elements specified in EAM 4.7, data is also provided for Ba.

Table 9. Quantitative data for five baby foods. Units: µg/kg (ppb), n=6.

	Green Beans	Mango	Chicken Noodle	Banana, Carrot, Strawberry	Apple, Pear, Green Pea
^{52}Cr	26.84 ± 1.38	55.49 ± 1.64	18.12 ± 1.35	405.2 ± 4.82	24.76 ± 3.36
^{55}Mn	1928.45 ± 23.03	1252 ± 17.82	1729 ± 40.77	1884 ± 27.87	401.9 ± 4.65
^{60}Ni	121.28 ± 1.24	221.8 ± 7.38	69.08 ± 5.89	287.6 ± 9.43	63.30 ± 2.57
^{63}Cu	414.10 ± 8.10	563.9 ± 13.40	632.6 ± 10.58	631.2 ± 9.21	606.8 ± 8.94
^{66}Zn	1611.47 ± 21.12	575.8 ± 23.20	4484 ± 72.42	1632 ± 316.56	928.8 ± 91.24
^{75}As	<LOD	4.67 ± 0.40	5.78 ± 0.26	<LOD	<LOD
^{78}Se*	<LOD	6.64 ± 0.70	34.73 ± 1.39	<LOD	<LOD
^{95}Mo	93.99 ± 2.28	14.01 ± 0.57	60.40 ± 1.33	85.20 ± 1.02	76.38 ± 0.38
^{111}Cd	<LOD	2.04 ± 0.15	13.98 ± 0.60	4.19 ± 0.25	<LOD
^{137}Ba	964.10 ± 17.52	934.9 ± 23.08	1148 ± 17.56	645.3 ± 8.87	353.1 ± 6.08
^{201}Hg	<LOD	<LOD	<LOD	<LOD	<LOD
^{205}Tl	<LOD	<LOD	<LOD	<LOD	<LOD
Pb ‡	<LOD	<LOD	<LOD	<LOD	<LOD

** Enhanced He mode used for As and Se. ‡ Sum of Pb isotopes.*

Table 9 continued. Quantitative data for four more baby food products. Units: µg/kg (ppb), n=6.

	Pear, Apple, Broccoli	Carrot, Zucchini, Broccoli	Sweet Potato, Turkey	Prunes
^{52}Cr	21.44 ± 0.29	<LOD	13.50 ± 0.62	42.45 ± 1.34
^{55}Mn	493.2 ± 6.35	14.20 ± 0.47	1362 ± 18.96	591.8 ± 7.99
^{60}Ni	36.69 ± 1.63	1.39 ± 0.04	28.11 ± 4.49	164.6 ± 3.21
^{63}Cu	511.2 ± 7.34	5.02 ± 0.17	561.3 ± 7.94	708.3 ± 8.66
^{66}Zn	591.8 ± 13.26	24.46 ± 0.58	2105 ± 40.43	1202 ± 19.38
^{75}As	<LOD	<LOD	<LOD	<LOD
^{78}Se*	<LOD	<LOD	27.35 ± 1.08	<LOD
^{95}Mo	27.39 ± 0.65	<LOD	15.00 ± 0.34	8.91 ± 0.31
^{111}Cd	1.16 ± 0.16	<LOD	3.61 ± 0.17	<LOD
^{137}Ba	258.1 ± 3.75	11.21 ± 0.36	313.2 ± 5.95	274.0 ± 6.19
^{201}Hg	<LOD	<LOD	<LOD	<LOD
^{205}Tl	<LOD	<LOD	<LOD	<LOD
Pb ‡	<LOD	<LOD	<LOD	<LOD

** Enhanced He mode used for As and Se. ‡ Sum of Pb isotopes.*

As shown in Tables 8 and 9, the measured concentrations of As were above the detection limit in three baby foods, and Cd was above the detection limit in seven baby foods. Hg and Pb were not detected above the detection limit in any of the baby foods.

Figure 4 shows the results in relation to the action levels proposed in the Baby Food Act 2021 (Table 1). The concentration of Cd in two baby foods exceeded the action level of 5 ppb for non-cereal infant and toddler food, as indicated by the red-dotted line.

IntelliQuant data

When an analyst develops a quantitative method using a preset method, IntelliQuant Quick Scan data is acquired automatically. No special setup or separate calibration is needed, simplifying the analysis. IntelliQuant automatically acquires full mass-spectrum data for up to 78 elements in every sample with only 2 s measurement time, allowing the analyst to quickly see which elements are present in the samples. IntelliQuant data is acquired in helium collision cell mode, so analytes are largely free from errors caused by polyatomic ion overlaps, ensuring the quality of the data.

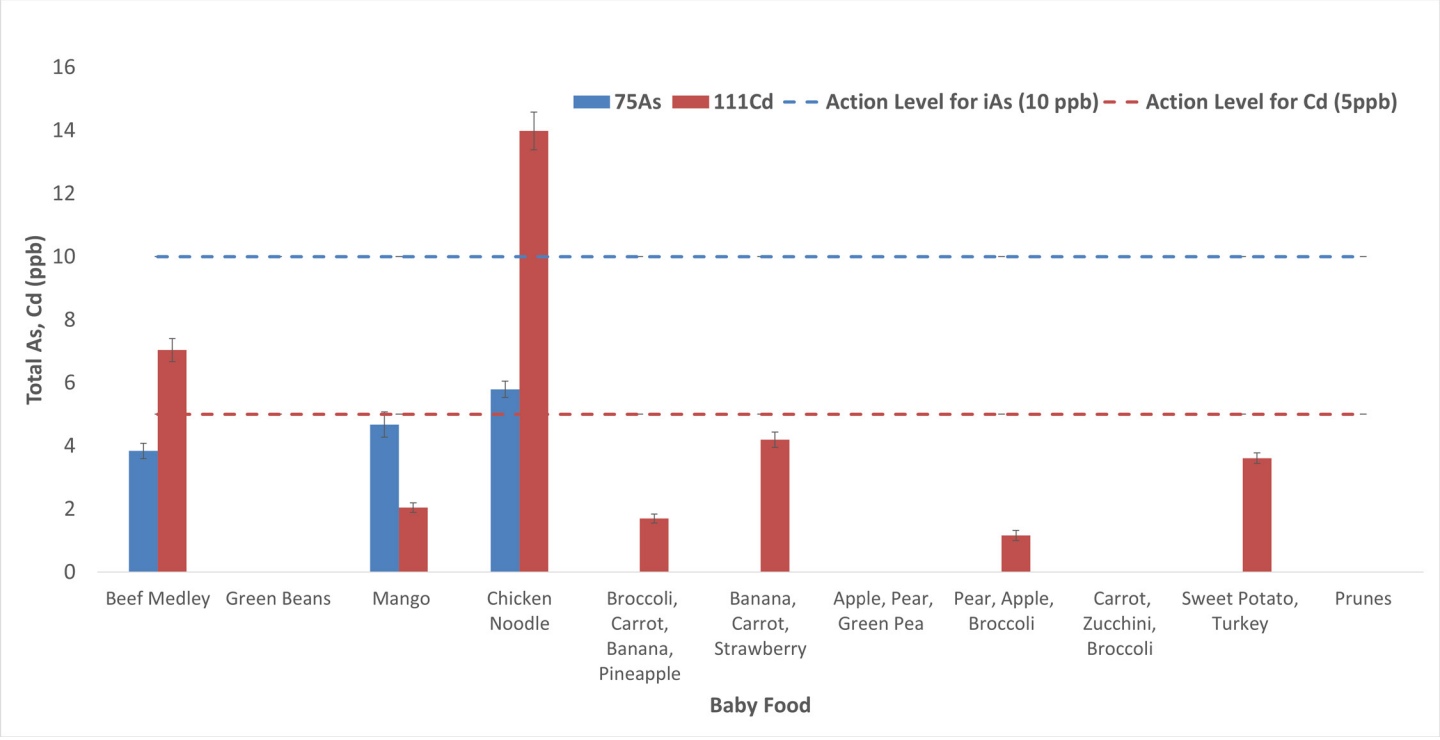

Figure 4. Measured concentrations of As and Cd compared to the proposed US government action levels (indicated by the blue and red dotted lines, respectively). No Pb or Hg was detected in any of the food samples.

In this study, IntelliQuant data was acquired for each food sample with the 7850 ICP-MS operating in He mode. The data can be displayed in a periodic table view, as shown in Figure 5. The periodic table "heat map" shows which elements are present in the sample. The color intensity indicates the concentration of elements in the sample i.e. the darker the red, the higher the concentration of that element.

IntelliQuant provides a complete picture of the elements present in the sample. The results for green beans (Figure 5) confirm the absence of As, Cd, Pb, and Hg in the sample.

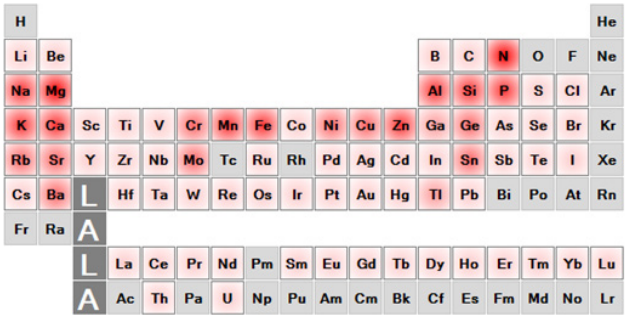

Figure 5. Periodic table heat map view of ICP-MS IntelliQuant data acquired for a green beans baby food.

Inorganic arsenic in baby rice cereals

In a previous study using an Agilent HPLC-ICP-MS system, the EAM 4.11 method was applied to the determination of four As species – including iAs (sum of As(III) and As(V))– in 31 infant rice cereals (*14, 15*). The four species were separated using isocratic anion exchange HPLC, and the chromatographic peaks were detected using ICP-MS, as shown in Figure 6. The LODs ranged from 0.9 to 1.8 ppb for the different As species and the LOQs were 7 to 14 ppb (these limits include the dilution factor). The measured concentrations are shown in Figure 7. The orange dashed line represents the US government proposed action limit for iAs in rice-based baby foods of 15 ppb (*2*). The green-dashed line represents the FDA proposed action limit of 100 ppb (*3*).

Figure 7 shows that all of baby rice samples would fail the action limit for iAs proposed in the Baby Food Safety Act 2021 (orange-dotted line). Many of the samples would also fail the 100 ppb limit proposed by the FDA (green-dotted line).

A faster screening method for the determination of iAs in baby rice cereal in under two minutes has also been developed using an Agilent HPLC-ICP-MS system (*16, 17*).

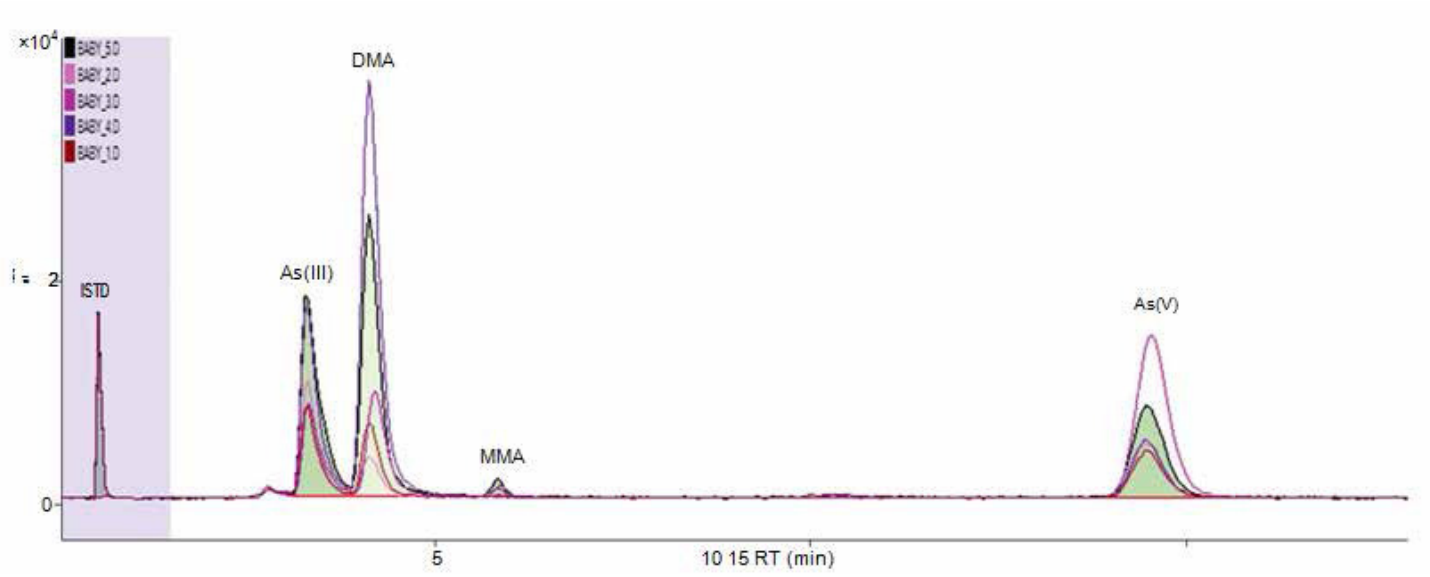

Figure 6. Overlay of As chromatograms in five rice cereals measured using HPLC-ICP-MS.

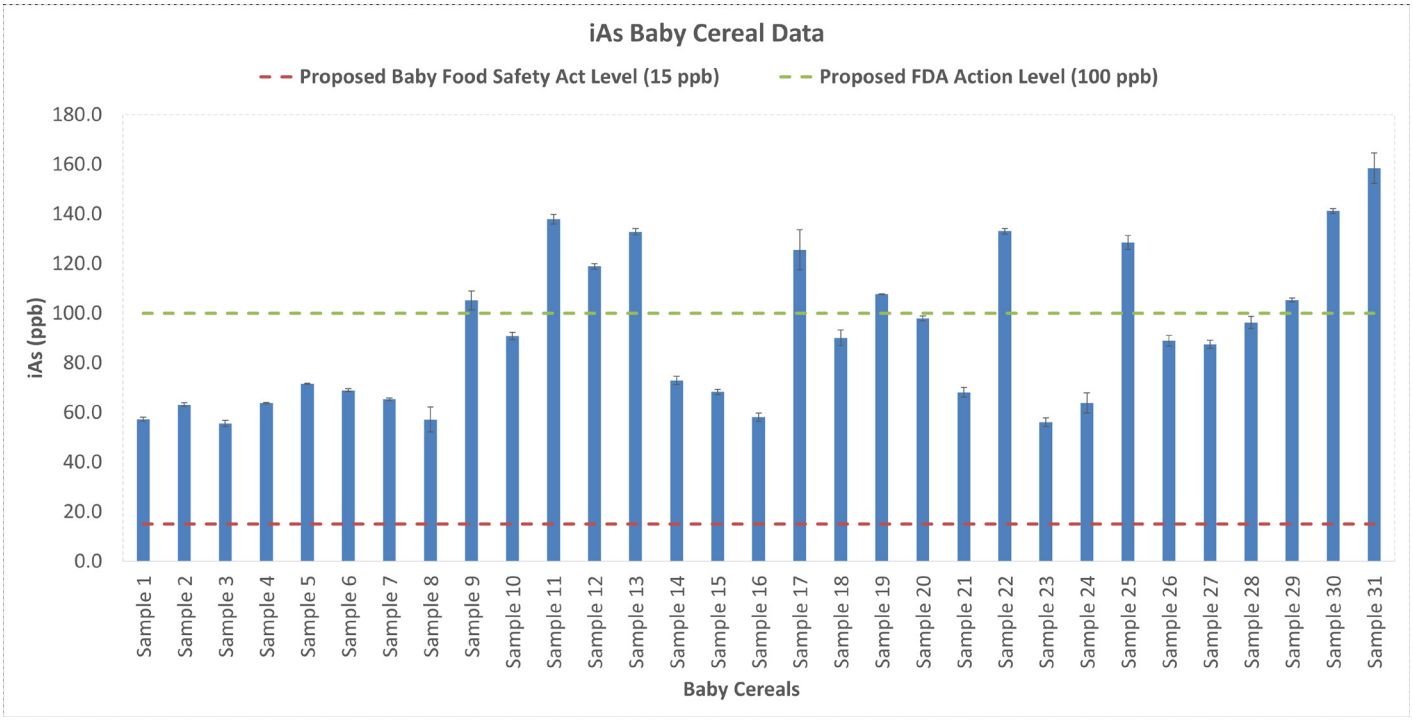

Figure 7. iAs in baby rice samples determined by HPLC-ICP-MS compared to proposed action levels for iAs (*2, 3*).

Conclusion

The US government is expected to introduce stringent action levels for iAs, Cd, Hg, and Pb in baby and infant foods. Therefore, manufacturers of baby food products urgently require easy-to-use, reliable, and accurate methods for the routine testing of these metals and compounds in ingredients and final products. This study has demonstrated the suitability of the Agilent 7850 ICP-MS for the analysis of multiple elements in a range of baby foods in accordance with EAM method 4.7, including total As, Cd, Hg, and Pb. All samples were prepared in the same batch using a single microwave digestion method.

The accuracy of the method was evaluated by analyzing three food-based SRMs and conducting a spike recovery test for 12 elements in two food samples. Excellent recoveries were achieved in all cases. The 7850 ICP-MS also met the nominal detection limit requirements specified in the EAM method and showed excellent stability over a 48-hour run. Only Cd was found to exceed the action level of 5 ppb for non-cereal infant and toddler food in two of the baby foods.

To comply with the action levels stated in the Baby Food Safety Act 2021, food labs would need to measure inorganic arsenic in samples with a total As concentration more than 10 ppb. The limit for cereal-based foods is >15 ppb. Based on the findings of a previous study, it is likely that iAs will need to be measured in many rice-based baby and infant cereals. The 7850 ICP-MS can easily be coupled to HPLC, providing labs with a routine HPLC-ICP-MS capability to monitor iAs in baby foods.

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