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Automated Analysis of Heavy Metals and Arsenic Species in Baby Cereal, Rice Flour, Seaweed, and Fish

Highlights:

- Analysis of various types of food samples including baby cereal, rice flour, seaweed, and fish.
- Method validation using reference materials BARI-1, NIST 1568b, and TORT-3.
- Digestion procedure for foods containing As, Cd, Hg, Pb, and Se.
- Extraction procedure for foods containing arsenobetaine, arsenocholine, dimethylarsinic acid, monomethylarsonic acid, arsenite, and arsenate.
- Single sample introduction system (prep*FAST* IC), in combination with an ICPMS, used for both total metals and speciation.
- Inline dilution capabilities for easy, high-quality calibrations and reduced sample prep.

Introduction

Food safety processes ensure that consumers receive products that are safe for consumption and free of physical, chemical, or biological hazards, including adulterants, pesticides, antibiotics, and chemical contaminants. In the case of chemical contaminants, heavy metals such as arsenic (As), mercury (Hg), lead (Pb), and cadmium (Cd) may lead to adverse health effects if consumed. Table 1 displays maximum contaminant limits for select food types in the United States, Europe Union (EU), Nigeria, and Oceania (Australia and New Zealand) related to As, Pb, Cd, Hg, and Se.

As, Hg, Pb, and Cd are toxic and have no known essential roles within the human body, whereas elements like selenium (Se) are considered both essential and toxic depending on the concentrations and species present. Therefore, careful monitoring of these elements is important for maintaining food safety. Unfortunately, measuring the total amount of an element does not always provide enough information to determine its overall toxicity, so identifying which form of the species is present in a given sample is required. For example, it is well known that the inorganic forms of arsenic (As III and As V) are more lethal than the organic forms (arsenobetaine [AsB], arsenocholine [AsC], monomethylarsonic acid [MMA], and dimethylarsinic acid [DMA]); the LD_{50} for As III is 14 mg/kg, whereas AsB is ~5,000 mg/kg. Additionally, an organic form of mercury (ethyl mercury) is more toxic (LD_{50} of < 0.1 mg/kg) than inorganic mercury (LD_{50} of < 100 mg/kg).

An inductively coupled plasma mass spectrometer (ICPMS) is typically employed to determine heavy metal content in samples due to its superior detection limits as compared to other technologies. In order to measure the form of arsenic or mercury species present in a sample, liquid chromatography is required to separate the species based on charge or polarity prior to introduction into the ICPMS. Traditionally, this requires two instrument platforms to obtain both total metals content and elemental species information. To simplify and improve these analyses, Elemental Scientific has designed the prep*FAST* IC that combines both the total metals and elemental speciation measurements into a single platform. To demonstrate the capabilities of the prep*FAST* IC, food samples were analyzed for both heavy metals and arsenic speciation in combination with an ICPMS.

Table 1. Government-mandated regulation limits for element contaminants in food

		United States	European Union	Nigeria	Oceania
		mg/kg			
iAs	White Rice	*	0.2	*	*
	Parboiled and husked rice	*	0.25	*	*
	Rice	0.075-0.15	*	*	*
	Rice wafers, crackers	*	0.3	*	*
	Rice for infants/young children	0.1	0.2	*	*
	Apple Juice	0.01	*	*	*
	Seaweed	*	*	*	1
	Fish	*	*	*	2
As total	Cereal Grains	*	*	*	1
	Cocoa Products	*	*	0.5	*
	Fruit Juice	*	*	0.2	*
	Bottled Water	0.01	*	*	*
Pb	Bottled Water	0.005	*	*	*
	Juice	0.05	0.05	0.1	*
	Cocoa Products	*	*	1	*
	Candy	0.1	*	*	*
	Fish	*	0.3	*	0.5
	Seaweed	*	0.1	*	*
	Cereal, pulses, legumes	*	*	*	0.2
	Wine	*	0.2	*	*
Cd	Rice cereal	*	0.1	*	0.1
	Rice	*	0.2	*	0.1
	Root and tuber Vegetables	*	*	*	0.5
	Cocoa Products	*	*	*	*
	Fish (except mackerel, tuna, bichique)	*	0.1	*	*
	Mackerel, tuna, bichique	*	0.15	*	*
	Bullet tuna	*	0.15	*	*
	Anchovy, Swordfish, Sardine	*	0.25	*	*
Hg	Fish	*	1	*	1.5
Se	Feed	0.1	*	*	*
	Daily intake (µg)	*	*	*	15-135
	Additive	*	0.5	*	*

*No government regulations were reported at this time.

Experimental

Food samples were obtained from local markets near Omaha, NE, USA, while the reference materials were obtained from NIST and NRC (Table 2). Two different procedures were used to prepare the samples for analysis and to preserve both the total metals and the species of interest. The procedure details of the digestion (for total metals) and extraction (for elemental speciation) can be found in Table 3.

Table 2. List of samples investigated in this study.

Sample ID	Manufacturer	Sample Type
SW-1	NusaPure	Brown Seaweed
SW-2	Swanson	Brown Seaweed
SW-3	AHANA Nutrition	Brown Seaweed
SW-4	Only Natural	Brown Seaweed
SW-5	Beyond Fresh	Ocean Fresh Master Blend Seaweed
BF-1	Gerber	Rice Cereal
BF-2	Earth's Best	Organic Rice Cereal
BF-3	Parent's Choice	Rice Cereal
BARI-1	***NRC CRM***	***Baby Cereal Coarse Rice Flour***
RF-1	Arrowhead	Brown Rice Flour
RF-2	Betty Crocker	Rice Flour Blend
RF-3	Great Value	Gluten Free Flour
NIST 1568b	***NIST SRM***	***Rice Flour***
F-1	Chicken of the Sea	Albacore White Tuna
F-2	Star Kiss	Albacore White Tuna
F-3	Chicken of the Sea	Pink Salmon
F-4	Chicken of the Sea	Crabmeat
F-5	Pampa	Giant Calamari
TORT-3	***NRC CRM***	***Lobster Hepatopancreas***

Figure 1. prep*FAST* IC.

Table 3. List of samples investigated in this study.

	Digestion Procedure	Extraction Procedure
Sample Preparation	• ~ 0.5 g of fish, baby food, and rice flour samples were weighed out into individual vials. Due to the nature of the seaweed breakdown during digestion, only 0.1 g of these samples were used. • 5 mL of concentrated nitric acid was added to each vial, followed by 1 mL concentrated Hydrogen Peroxide.	• ~ 0.5 g of all samples were weighed out into individual vials. • 10 mL of 1% nitric acid (v/v) were added to each of the vials.
Procedure Method	• The samples were digested to completion (no visible sample residue, and no more bubbles forming). This took about 1 to 3 hours to complete depending on the sample type. No heating was required to complete this step. • The samples were then added to 50 mL vials and diluted to 50 mL with DI water. These samples were analyzed using the prep*FAST* IC in total metals mode with sample dilutions of 2x and 5x.	• The vials were heated at 90°C for 90 minutes. Vials were taken off the heat block and allowed to cool to room temperature prior to sample preparation. • The samples were then spun in a centrifuge for 30 minutes at 3000 RPM. • 5 mL of the supernatant solutions were then placed in new vials and diluted to 10 mL with DI water. • These resulting samples were then run using the prep*FAST* IC in speciation mode with sample dilutions of 10x and 20x.

Experimental *(continued)*

The prep*FAST* IC (Fig. 1) is a single-platform, metal-free system capable of performing total metals and elemental speciation in an automated fashion. This system includes an autosampler, inline dilution system (prep*FAST*), column valve, and a syringe-driven high-pressure chromatography system. The inline dilution system allows for autocalibration using a single stock standard as well as inline dilutions of samples, saving time and reducing potential preparation error. When operating in total metals mode, the chromatographic column is bypassed, whereas in speciation mode, the sample passes through the chromatographic column prior to introduction to the ICPMS. For total metals determination, the carrier and diluent consisted of 2% HNO_3 (v/v), while in speciation mode, the eluent and diluent were ammonium carbonate and DI water, respectively.

An ICPMS was connected to the prep*FAST* IC. For the total metals analysis, ^{75}As and ^{114}Cd were measured in collision-cell mode using He gas, ^{202}Hg and ^{208}Pb were measured in standard mode, and $^{80}Se^{16}O$ was measured using triple-quad mode using O_2 gas. For arsenic speciation, ^{75}As was measured in collision-cell mode using He gas (alternative method would be to use O_2 gas and measure $^{75}As^{16}O$). These methods were automated such that the total metals and arsenic speciation were queued together and the analyses were performed with no user interaction to switch from total metals with inline sample prep to arsenic speciation with inline sample prep.

Results and Discussion

Typical calibration curves for the total metals mode can be seen in Fig. 2. These calibration curves were prepared inline with the prep*FAST* IC utilizing a single stock standard of 200 ppb As, 200 ppb Cd, 50 ppb Se, 5 ppb Hg, and 5 ppb Pb with 1–100x dilutions defined in the software. Detection limits were determined to be between 1–10 ng/L (Table 4). Example calibration curves for the arsenic speciation method can be found in Fig. 3. Since many of the samples were expected to be high in AsB, the calibration range for this species was set to 1–100 ppb, while all other arsenic species were calibrated from 25–500 ppt. The detection limit for each of the arsenic species is between 1–4 ng/L. Figure 4 displays a chromatogram that highlights the easy detection of 25 ppt As III, As V, DMA, MMA, and AsC from a 50 µL injection (AsB is also displayed but at a concentration of 5 ppb for reasons mentioned previously). One advantage of the inline dilution technique is that any samples with concentrations higher than the highest calibration point can be easily diluted inline at a higher dilution factor without the need for physical user interaction.

To validate the method, three different reference materials, BARI-1 (baby cereal coarse rice flour, NRC), NIST 1568b (rice flour, NIST), and TORT-3 (lobster hepatopancreas, NRC), were evaluated using the previously described digestion and extraction methods. Table 5 displays the results for these three reference materials for As, Cd, Hg, Pb, iAs (total inorganic arsenic—which is defined as the sum of As III and As V), AsB, DMA, and MMA. The "As Total" value was determined during the total metals mode measurements, whereas the iAs, AsB, DMA, and MMA were determined using the arsenic speciation method. The measured values were all within the reference ranges reported for each reference material tested. Figure 5 displays example arsenic speciation chromatograms for BARI-1 and NIST 1568b along with an overlay of a standard containing all species for reference purposes. For seafood or seaweed samples, the amount of AsB is expected to be routinely high. One significant advantage of the prep*FAST* IC is that any sample can be diluted inline, which for samples that are high in AsB can be extremely useful. If the AsB level is too high in a particular sample, it can overload the column and result in poor chromatographic separation and recovery. Figure 6 displays an example chromatogram for TORT-3 that is high in AsB, but a 50x inline dilution improves the chromatographic separation and recovery. Any sample can be easily reanalyzed with additional dilution by increasing the dilution factor in the software prior to reanalysis; no manual dilutions are required when analyte concentrations are higher than the highest calibration point.

Table 4. Limits of detection (LOD) for the "total metals" and "speciation" methods used in this study.

	Element	Measured	LOD (ng/L)
Total Metals	As	^{75}As	3
	Cd	^{114}Cd	2
	Hg	^{202}Hg	4
	Pb	^{208}Pb	1
	Se	$^{80}Se^{16}O$	10
Speciation	As	AsB	2
		AsC	3
		DMA	1
		MMA	3
		As III	2
		As V	4

LOD = $(3^* \sigma_{blank})/m$

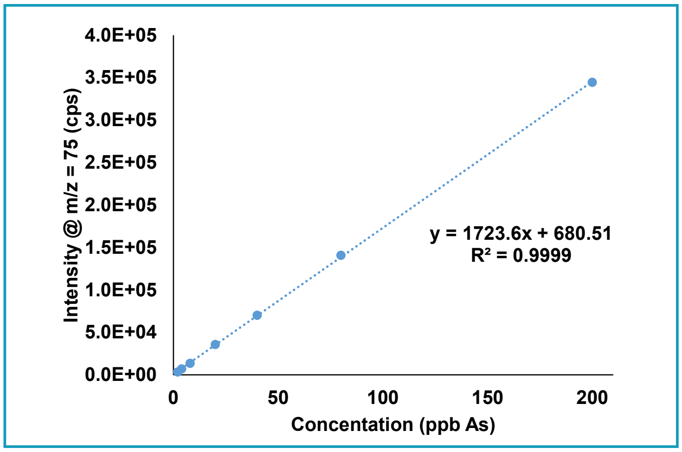


Figure 2a. Typical calibration curve for As measured in “total metals” mode.

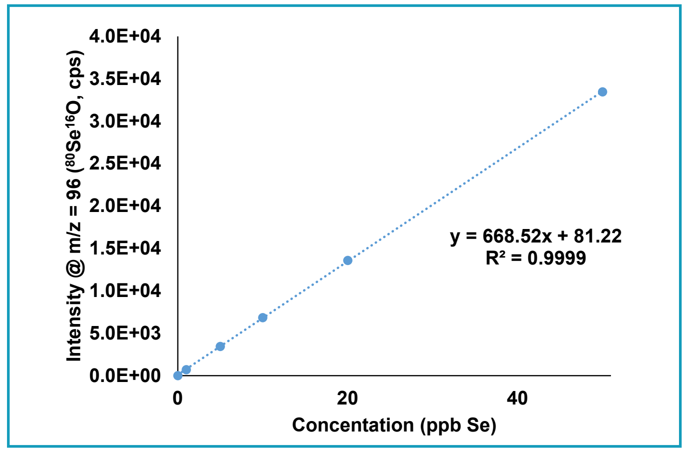


Figure 2b. Typical calibration curve for Se measured in “total metals” mode.

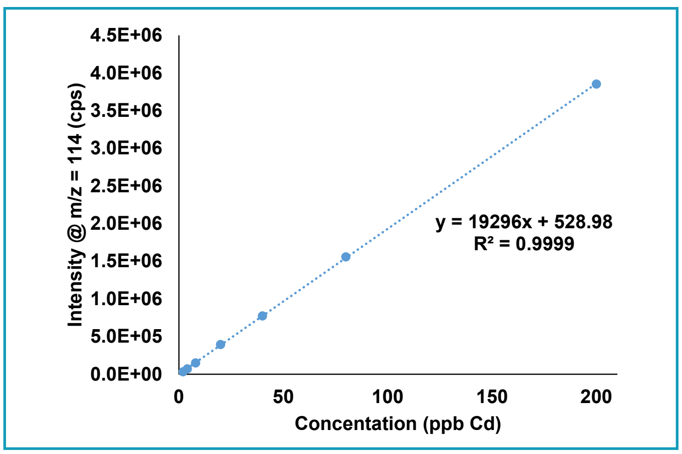


Figure 2c. Typical calibration curve for Cd measured in “total metals” mode.

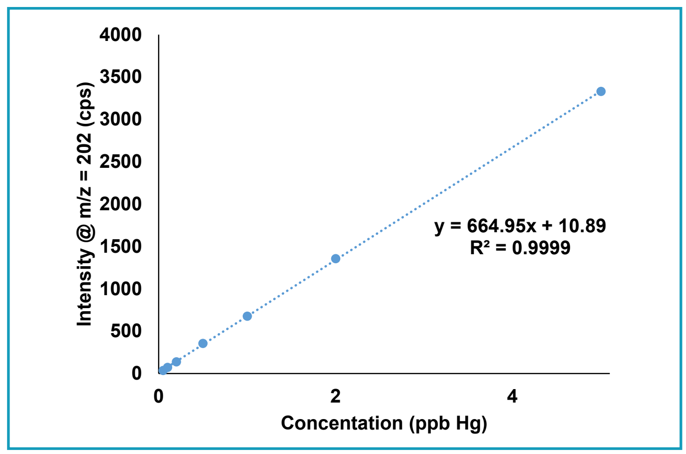


Figure 2d. Typical calibration curve for Hg measured in “total metals” mode.

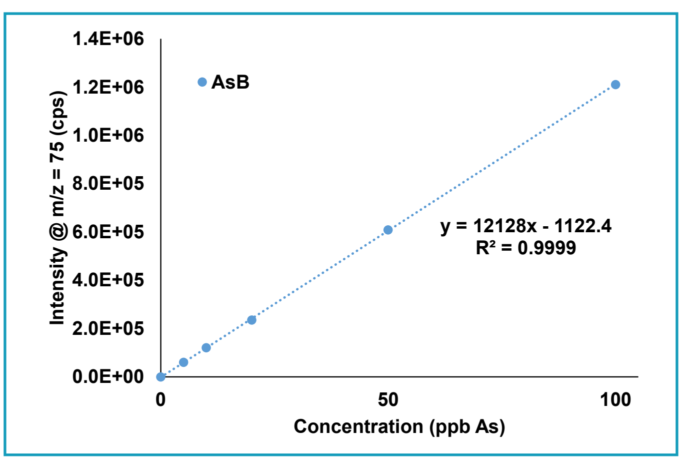


Figure 3a. Typical calibration curve for AsB measured in “speciation” mode.

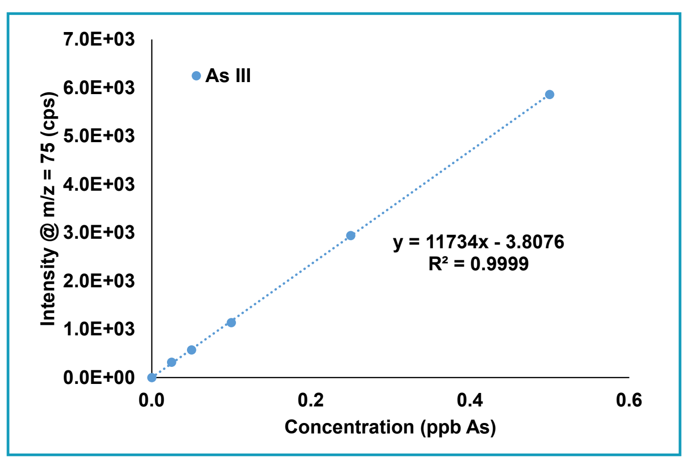


Figure 3b. Typical calibration curve for As III measured in “speciation” mode.

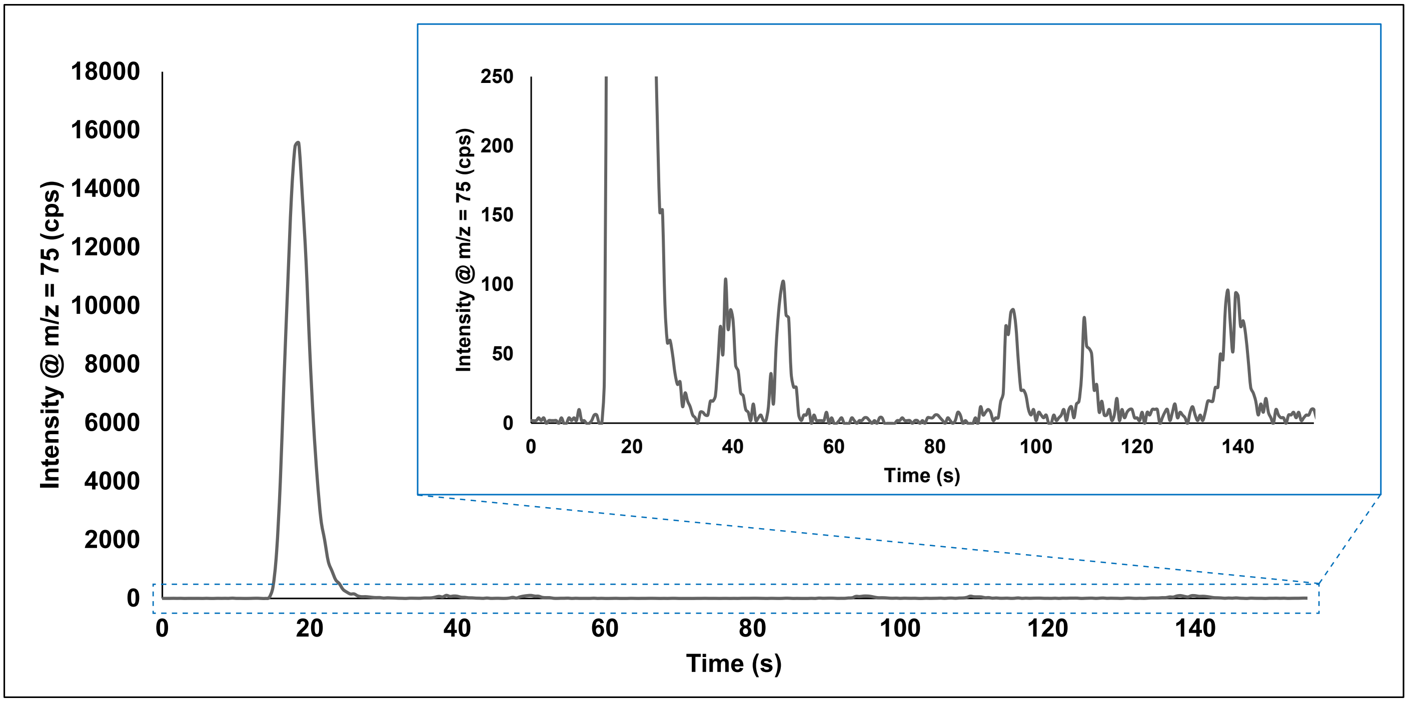


Figure 4. Chromatogram of standard 1, which equates to 5 ppb AsB and 25 ppt As III, As V, AsC, DMA, and MMA.

Table 5. Results from the total metals and speciation measurements for baby cereal coarse rice flour (BARI-1), rice flour (NIST 1568b), and lobster hepatopancreas (TORT-3). prep*FAST* IC represents data collected in this study (n = 3).

Reference Material		As Total (mg/kg)	Cd (mg/kg)	Hg (mg/kg)	Pb (mg/kg)	iAs (mg/kg)	AsB (mg/kg)	DMA (mg/kg)	MMA (mg/kg)
BARI-1	Target Value	0.248 ± 0.018	0.0134 ± 0.0014	0.0026 ± 0.0003	0.0064 ± 0.0016	0.113 ± 0.016	n/a	0.115 ± 0.010	0.0045 ± 0.0008
	Reference Range (min - max)	0.230 - 0.266	0.0120 - 0.0148	0.0023 - 0.0029	0.0048 - 0.0080	0.097 - 0.129	n/a	0.105 - 0.125	0.0037 - 0.0053
	prep*FAST* IC	**0.262 ± 0.010**	**0.0124 ± 0.0010**	**0.0024 ± 0.0003**	**0.0065 ± 0.0012**	**0.110 ± 0.003**	**-**	**0.106 ± 0.005**	**0.0040 ± 0.0010**
NIST 1568b	Target Value	0.285 ± 0.014	0.0224 ± 0.0013	0.00591 ± 0.00036	0.008 ± 0.003	0.092 ± 0.010	n/a	0.180 ± 0.012	0.0116 ± 0.0035
	Reference Range (min-max)	0.271 - 0.299	0.0211 - 0.0237	0.00555 - 0.00627	0.005 - 0.011	0.082 - 0.102	n/a	0.168 - 0.192	0.0081 - 0.0151
	prep*FAST* IC	**0.299 ± 0.006**	**0.0214 ± 0.0009**	**0.00600 ± 0.00080**	**0.007 ± 0.002**	**0.084 ± 0.013**	**-**	**0.171 ± 0.004**	**0.0113 ± 0.0050**
TORT-3	Target Value	59.5 ± 3.8	42.3 ± 1.8	0.137 ± 0.012	0.225 ± 0.018	n/a	54.9 ± 2.5	n/a	n/a
	Reference Range (min-max)	55.7 - 63.3	40.5 - 44.1	0.125 - 0.149	0.207 - 0.243	n/a	52.4 - 57.4	n/a	n/a
	prep*FAST* IC	**62.3 ± 1.6**	**41.7 ± 0.9**	**0.128 ± 0.015**	**0.209 ± 0.010**	**-**	**53.2 ± 4.5**	**-**	**-**

Results and Discussion *(continued)*

After validation of the methods using the reference materials, five seaweed (SW), three rice cereal (BF), three rice flour (RF), and five seafood (F) samples were analyzed. Table 6 displays the results from these samples, which were chosen because they are readily available to any consumer. Considering the allowable limits listed in Table 1, there are a few samples that were found to exceed these limits. The amount of Cd measured in seaweed (SW-1, SW-2, and SW-4) was found to exceed the limits for food and fish products (0.1–0.5 mg/kg Cd, Table 1), but it should be noted that there is no specific regulation for Cd in seaweed. The calamari sample tested (F5) was found to be just above the allowable limit of 0.1 mg/kg Cd. The amount of Pb found in 4 of the 5 seaweed samples (SW-1, SW-2, SW-4, and SW-5) was above the EU allowable limit of 0.1 mg/kg Pb. Of these samples, SW-4 was found to be 1.68 ± 0.23 mg/kg Pb, which is ~17 times higher than the allowable limit. None of the samples had a level of Hg that exceeded the limits listed in Table 1. Canned tuna (F2) had the highest concentration of Hg, which was measured as 0.521 ± 0.068 mg/kg Hg; however, this is still below the 1 mg/kg limit set by the EU. The regulations for selenium are less stringent and it is less prevalent in food. The only samples that were found to be high in Se were the fish samples, especially the canned tuna samples, which contained 0.575 and 0.405 mg/kg Se.

One of the most powerful aspects of the prep*FAS*T IC is that it can determine both total metals and elemental species with a single analytical platform. The results from these two determinations should correlate well, which is shown for As in Table 7. The linear regression (Fig. 7) also shows excellent correlation between the two As measurements. For the samples analyzed in this study, the brown seaweed (SW-2) and brown rice flour (RF-1) samples were found to be above the allowable limits for iAs listed in Table 1. The amount of arsenic varied greatly among the different food groups tested, but most of the arsenic detected was organic arsenic. Example arsenic speciation chromatograms for seaweed, rice cereal, rice flour, and seafood can be found in Figs. 8, 9, 10, and 11, respectively.

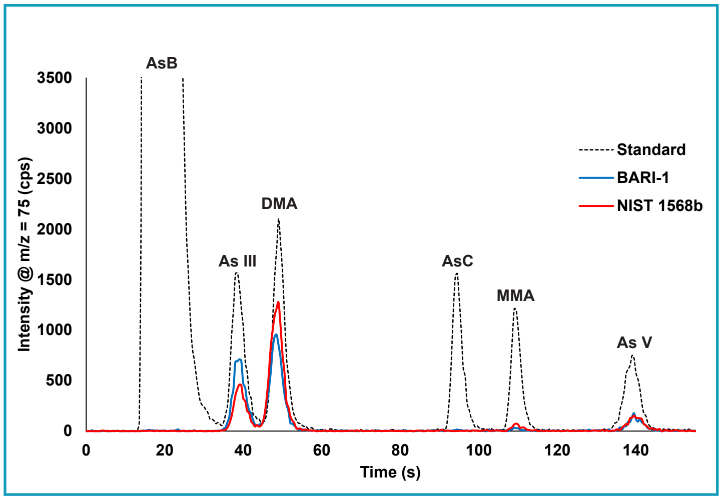


Figure 5. BARI-1 and NIST 1568b chromatograms overlaid with standard 5 for species identification purposes. Standard = 100 ppb AsB and 0.5 ppb As III, As V, AsC, DMA, and MMA.

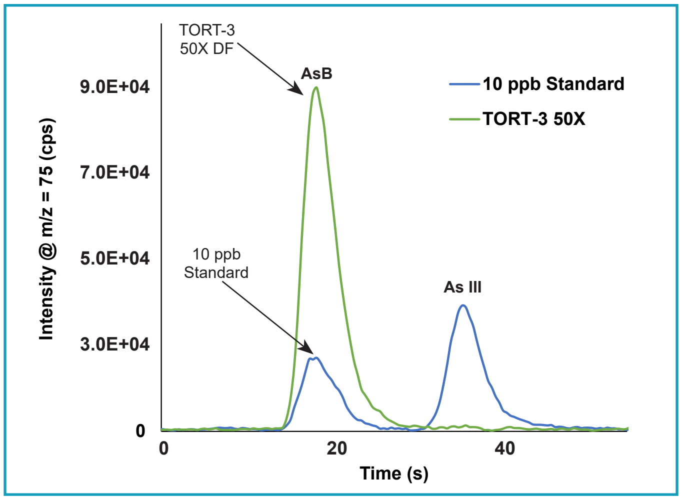


Figure 6. Example of how the inline dilution function can be used for samples with high levels of AsB, such as seafood samples.

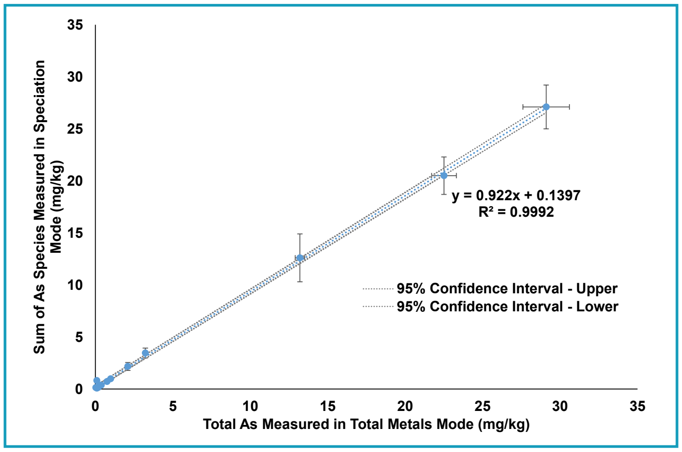


Figure 7. Linear regression comparing the sum of the arsenic species compared to the total arsenic in each of the food samples analyzed.

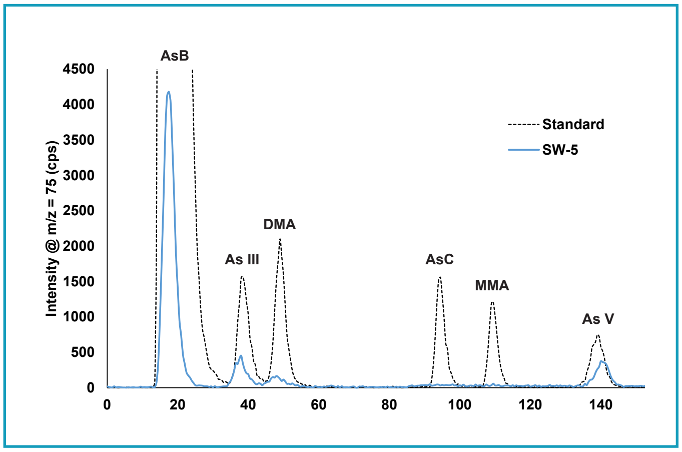


Figure 8. One of the seaweed samples overlaid with standard 5 for species identification purposes. Standard = 100 ppb AsB and 0.5 ppb As III, As V, AsC, DMA, and MMA.

Table 6. Results from the total metals and speciation measurements for seaweed, rice cereal, rice flour, and seafood samples (n = 3).

Seaweed Samples	As Total (mg/kg)	Cd (mg/kg)	Hg (mg/kg)	Pb (mg/kg)	Se (mg/kg)
SW-1	22.5 ± 0.8	0.260 ± 0.010	0.016 ± 0.008	0.204 ± 0.032	0.031 ± 0.005
SW-2	13.2 ± 0.3	0.670 ± 0.010	0.006 ± 0.001	0.300 ± 0.003	0.100 ± 0.020
SW-3	0.201 ± 0.018	0.022 ± 0.003	0.005 ± 0.001	0.007 ± 0.001	0.099 ± 0.012
SW-4	29.1 ± 1.5	0.500 ± 0.020	0.023 ± 0.003	1.68 ± 0.23	0.044 ± 0.010
SW-5	0.750 ± 0.030	0.049 ± 0.010	0.007 ± 0.003	0.128 ± 0.005	0.071 ± 0.010
Baby Food	**As Total (µg/kg)**	**Cd (µg/kg)**	**Hg (µg/kg)**	**Pb (µg/kg)**	**Se (µg/kg)**
BF-1	105 ± 10	8.5 ± 0.7	1.7 ± 0.3	7.1 ± 2.0	64 ± 6
BF-2	92 ± 5	40 ± 2	1.8 ± 0.4	22 ± 3	22 ± 5
BF-3	125 ± 1	22 ± 1	0.50 ± 0.11	9.0 ± 2.1	ND
Rice Flour	**As Total (µg/kg)**	**Cd (µg/kg)**	**Hg (µg/kg)**	**Pb (µg/kg)**	**Se (µg/kg)**
RF-1	378 ± 31	15 ± 1	3.7 ± 1.0	4.3 ± 1.0	51 ± 6
RF-2	114 ± 17	10 ± 1	2.2 ± 0.6	9.7 ± 3.0	53 ± 6
RF-3	89 ± 16	11 ± 1	1.1 ± 0.3	9.8 ± 5.0	8.5 ± 1.2
Seafood Samples	**As Total (mg/kg)**	**Cd (mg/kg)**	**Hg (mg/kg)**	**Pb (mg/kg)**	**Se (mg/kg)**
F-1	3.22 ± 0.08	0.014 ± 0.001	0.181 ± 0.002	0.004 ± 0.001	0.575 ± 0.033
F-2	2.09 ± 0.07	0.010 ± 0.001	0.521 ± 0.068	0.004 ± 0.001	0.405 ± 0.026
F-3	0.192 ± 0.024	0.005 ± 0.001	0.017 ± 0.002	0.005 ± 0.001	0.143 ± 0.016
F-4	0.133 ± 0.004	0.048 ± 0.001	0.019 ± 0.002	0.016 ± 0.003	0.059 ± 0.010
F-5	0.983 ± 0.006	0.101 ± 0.027	0.035 ± 0.004	0.009 ± 0.003	0.295 ± 0.050

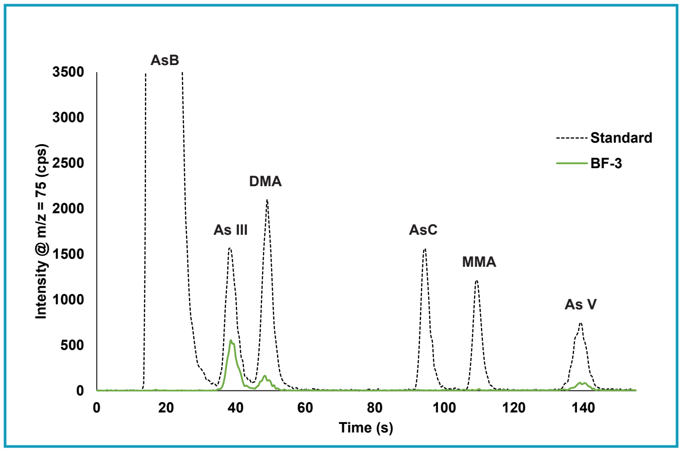


Figure 9. One of the baby food (rice cereal) samples overlaid with standard 5 for species identification purposes. Standard = 100 ppb AsB and 0.5 ppb As III, As V, AsC, DMA, and MMA.

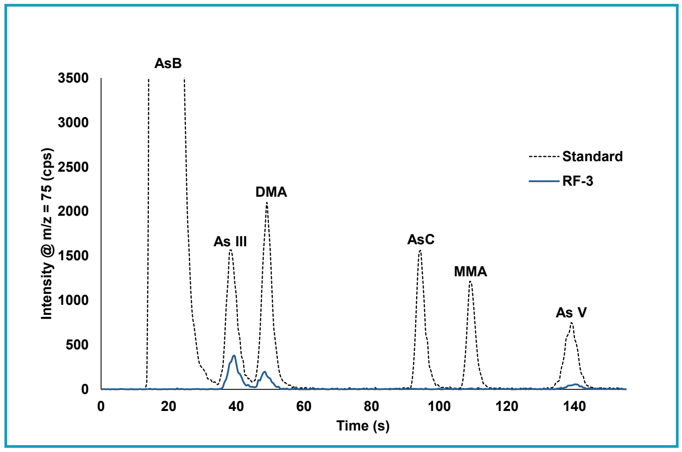


Figure 10. One of the rice flour samples overlaid with standard 5 for species identification purposes. Standard = 100 ppb AsB and 0.5 ppb As III, As V, AsC, DMA, and MMA.

iAs (mg/kg)	AsB (mg/kg)	DMA (mg/kg)	MMA (mg/kg)	AsC (mg/kg)	Sum of As Species (mg/kg)
0.640 ± 0.200	19.0 ± 2.1	0.703 ± 0.030	0.060 ± 0.020	0.120 ± 0.009	20.5 ± 1.8
2.16 ± 0.03	9.88 ± 0.80	0.409 ± 0.084	0.050 ± 0.004	0.140 ± 0.050	12.6 ± 2.3
0.078 ± 0.011	0.028 ± 0.007	0.044 ± 0.013	0.021 ± 0.003	0.020 ± 0.005	0.191 ± 0.050
0.297 ± 0.064	26.2 ± 2.0	0.390 ± 0.072	0.035 ± 0.013	0.106 ± 0.010	27.1 ± 2.1
0.120 ± 0.002	0.562 ± 0.052	0.023 ± 0.007	0.230 ± 0.007	0.011 ± 0.005	0.727 ± 0.140
iAs (µg/kg)	**AsB (µg/kg)**	**DMA (µg/kg)**	**MMA (µg/kg)**	**AsC (µg/kg)**	**Sum of As Species (µg/kg)**
88 ± 8	8 ± 2	20 ± 3	ND	ND	116 ± 12
65 ± 3	ND	17 ± 2	ND	ND	82 ± 5
85 ± 6	4 ± 1	18 ± 4	13 ± 2	12 ± 2	132 ± 14
iAs (µg/kg)	**AsB (µg/kg)**	**DMA (µg/kg)**	**MMA (µg/kg)**	**AsC (µg/kg)**	**Sum of As Species (µg/kg)**
87 ± 4	2.1 ± 0.3	280 ± 20	1.2 ± 0.3	ND	374 ± 38
48 ± 4	28 ± 5	50 ± 9	ND	ND	126 ± 12
49 ± 6	6.0 ± 0.9	26 ± 9	ND	ND	81 ± 14
iAs (mg/kg)	**AsB (mg/kg)**	**DMA (mg/kg)**	**MMA (mg/kg)**	**AsC (mg/kg)**	**Sum of As Species (mg/kg)**
0.016 ± 0.002	3.42 ± 0.41	0.014 ± 0.002	ND	0.013 ± 0.004	3.46 ± 0.47
0.007 ± 0.001	2.15 ± 0.36	0.009 ± 0.001	ND	0.0013 ± 0.0008	2.17 ± 0.38
0.013 ± 0.005	0.156 ± 0.001	ND	ND	0.0013 ± 0.0009	0.171 ± 0.015
0.014 ± 0.004	0.115 ± 0.015	0.011 ± 0.003	ND	0.006 ± 0.001	0.145 ± 0.051
0.023 ± 0.004	0.956 ± 0.120	0.0011 ± 0.0005	ND	0.0017 ± 0.0002	0.982 ± 0.110

Table 7. Comparison of the As total and sum of As species for seaweed, rice cereal, rice flour, and seafood samples (n = 3).

Seaweed Samples	As Total (mg/kg)	Sum of As Species (mg/kg)	% iAs
SW-1	22.5 ± 0.8	20.5 ± 1.8	3.1%
SW-2	13.2 ± 0.3	12.6 ± 2.3	17.1%
SW-3	0.201 ± 0.018	0.191 ± 0.050	40.8%
SW-4	29.1 ± 1.5	27.1 ± 2.1	1.1%
SW-5	0.750 ± 0.030	0.727 ± 0.140	16.5%
Baby Food	**As Total (µg/kg)**	**Sum of As Species (µg/kg)**	
BF-1	105 ± 10	116 ± 12	75.9%
BF-2	92 ± 5	82 ± 5	79.3%
BF-3	125 ± 1	132 ± 14	64.4%
Rice Flour	**As Total (µg/kg)**	**Sum of As Species (µg/kg)**	
RF-1	378 ± 31	374 ± 38	23.3%
RF-2	114 ± 17	126 ± 12	38.1%
RF-3	89 ± 16	81 ± 14	60.5%
Seafood Samples	**As Total (mg/kg)**	**Sum of As Species (mg/kg)**	
F-1	3.22 ± 0.08	3.46 ± 0.47	0.5%
F-2	2.09 ± 0.07	2.17 ± 0.38	0.3%
F-3	0.192 ± 0.024	0.171 ± 0.015	7.6%
F-4	0.133 ± 0.004	0.145 ± 0.051	9.7%
F-5	0.983 ± 0.006	0.982 ± 0.110	2.3%

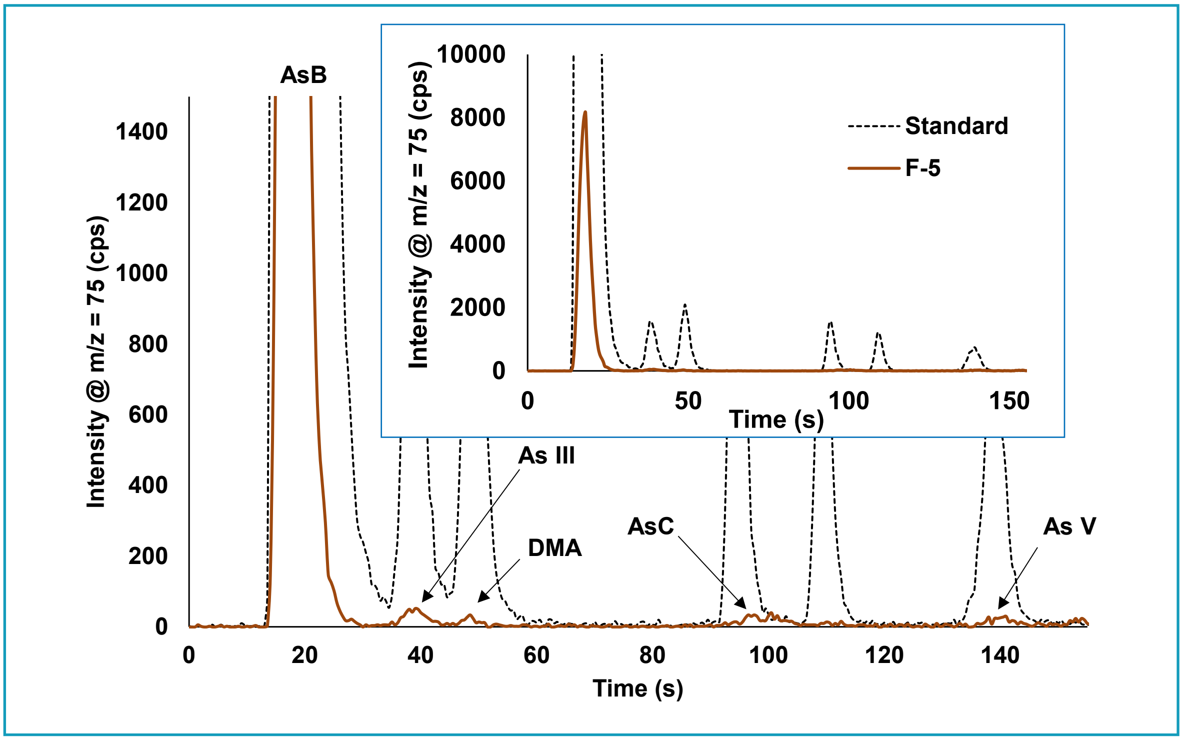
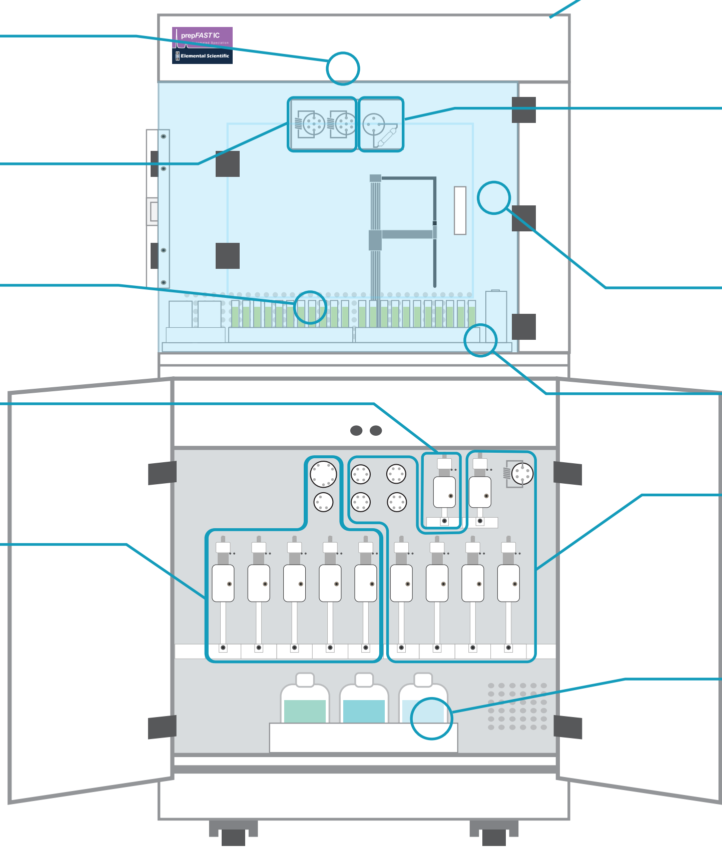


Figure 11. The giant calamari sample overlaid with standard 5 for species identification purposes. Standard = 100 ppb AsB and 0.5 ppb As III, As V, AsC, DMA, and MMA.

Conclusions

The prep*FAST* IC is a versatile instrument that automates laboratory work flow and easily performs both total metals analysis and arsenic speciation when connected to an ICPMS. Laboratories save time and reduce costs by using a single platform and not requiring an operator to manually switch between a regular autosampler (total metals) and a traditional LC system (speciation) to perform these two analyses. The methods were validated using the BARI-1 (NRC), NIST 1568b (NIST), and TORT-3 (NRC) for both total metals and As species. Real-word samples of seaweed, rice cereal, rice flour, and seafood were analyzed using the validated methods. The seaweed and seafood samples were found to have the highest concentrations of Cd, Pb, and organic arsenic as compared to the other samples tested. Good correlation was found between the total As and sum of As species, with all values falling within a 95% confidence interval.

prep*FAST* IC



Optional ULPA Filter

Autodilution Valves

Inline automated calibration and dilution system

Sample Containers

<1 mL up to 500 mL

Autotuning

Autodilution Syringe Module

- Sample autodilution at time of analysis
- Species-specific autocalibration capability
- Micro-volume sample syringe-loading
- Calibration matrix matching
- Inline sample chemical reactions
- Inline sample derivatization

Electronics in Rear Plenum

Speciation/Total Metals Selection Valve

Auto switching of total metals or speciation (chromatography mode)

Exhausted Enclosure

High-precision DX Autosampler

High-pressure Speciation Module

-Ultra-high-purity, metal free pump
-Gradient elution
-Post-column internal standard
-Post-column dilution
-Post-column derivatization

Reagent Reservoirs

For autodilution and elution reagents.