

## Supplementary Data

### Inorganic arsenic speciation analysis in food using HPLC/ICP-MS: Method development and validation

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**Supplementary Table 1.** Total arsenic concentration mg kg<sup>-1</sup> for reference materials in extracts.

Reference material	Total As <sup>a</sup> (mg/kg)	Solvent	Total As <sup>b</sup> (mg/kg)	Extraction rate <sup>c</sup> (%)
Rice flour (SRM-1568b)	0.29 ± 0.01	H <sub>2</sub> O	0.27 ± 0.03	94.2
		0.28M HNO <sub>3</sub>	0.31 ± 0.01	108.2
		50% EtOH	0.31 ± 0.03	107.5
Seaweed (Hijiki) (CRM-7405a)	36.4 ± 0.97	H <sub>2</sub> O	25.7 ± 1.24	70.6
		0.28M HNO <sub>3</sub>	26.1 ± 1.10	71.7
		50% EtOH	46.8 ± 1.03	128.5
Fish protein (CRM-DORM4)	6.77 ± 0.18	H <sub>2</sub> O	4.81 ± 0.05	71.0
		0.28M HNO <sub>3</sub>	4.98 ± 0.03	73.6
		50% EtOH	9.91 ± 0.09	146.4
Lobster hepatopancreas (CRM-TORT3)	60.3 ± 2.25	H <sub>2</sub> O	57.7 ± 2.03	95.8
		0.28M HNO <sub>3</sub>	60.4 ± 2.07	100.2
		50% EtOH	128.1 ± 1.60	212.6

<sup>a</sup> Total arsenic in microwave digestion by ICP/MS.

<sup>b</sup> Total arsenic in extraction by ICP/MS.

<sup>c</sup> Extraction rate (%) =  $b / a \times 100$

**Supplementary Table 2.** Concentrations of arsenic compounds in fish oil as measured by HPLC/ICP-MS (n = 3).

Extraction solution	Arsenic compounds (mg/kg) <sup>a</sup>						Sum of species
	AsC	AsB	As(III)	DMA	MMA	As(V)	(%) <sup>b</sup>
H <sub>2</sub> O	0.18 ± 0.005	0.18 ± 0.006	0.17 ± 0.006	0.18 ± 0.004	0.18 ± 0.004	0.18 ± 0.008	89.2
0.28M HNO <sub>3</sub>	0.20 ± 0.006	0.19 ± 0.007	0.18 ± 0.010	0.19 ± 0.007	0.18 ± 0.011	0.21 ± 0.014	95.8
2% Triton X-100	0.24 ± 0.017	0.22 ± 0.011	0.22 ± 0.008	0.23 ± 0.014	0.22 ± 0.013	0.24 ± 0.021	114.2
80% Methanol	0.30 ± 0.004	0.17 ± 0.001	0.24 ± 0.006	0.25 ± 0.002	0.24 ± 0.003	0.21 ± 0.011	117.5

<sup>a</sup> Spike level of 0.2 mg/kg for each As species.

<sup>b</sup> Sum of species (%) = sum of the extracted arsenic compounds / total spiked arsenic compounds × 100

**Supplementary Table 3.** Recoveries of As(V) spiked into seafood CRMs separated on the PRP-X100 column by HPLC/ICP-MS.

Reference material	As compound	Conc. (mg/kg)	Spiked level (mg/kg)	Recovery (%)	CV (%)
Lobster hepatopancreas (CRM-TORT3)	As(V)	0.21	0.10	95.5	3.65
Fish protein (CRM-DORM4)		0.30	0.10	113.9	6.75
Cod Fish Tissue (CRM-7402a)		N.D. <sup>a</sup>	0.10	110.1	2.89
Swordfish Tissue (CRM-7403a)		N.D. <sup>a</sup>	0.10	115.4	1.37

<sup>a</sup> N.D., not detected; LOQ = 0.02 mg/kg.

**Supplementary Table 4.** Total arsenic contents in marine oil samples analyzed by ICP-MS.

Sample	Total As (mg/kg)
Salmon oil_#1	N.D. <sup>a</sup>
Tuna oil_#1	N.D. <sup>a</sup>
Tuna oil_#2	N.D. <sup>a</sup>
Refined fish oil_#1	N.D. <sup>a</sup>
Refined fish oil_#2	N.D. <sup>a</sup>
Deep-sea fish oil_#1	N.D. <sup>a</sup>
Deep-sea fish oil_#2	N.D. <sup>a</sup>
Deep-sea fish oil_#3	N.D. <sup>a</sup>
Deep-sea fish oil_#4	N.D. <sup>a</sup>
Deep-sea fish oil_#5	N.D. <sup>a</sup>
Deep-sea fish oil_#6	N.D. <sup>a</sup>
Deep-sea fish oil_#7	N.D. <sup>a</sup>
Deep-sea fish oil_#8	N.D. <sup>a</sup>
Deep-sea fish oil_#9	N.D. <sup>a</sup>
Deep-sea fish oil_#10	N.D. <sup>a</sup>

<sup>a</sup> N.D., not detected; LOQ = 0.02 mg/kg.