

Development of an Eco-Friendly Microwave-Assisted Acid Digestion Method for the Determination of Elements in General Foodstuffs by ICP-MS and ICP-OES

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Table S1a

Information of CRMs and QC materials used.

Sample description	CRM code/QC material code	Refer to the sector of AOAC triangle
Peanut butter	NIST SRM 2387	Sector 1
Vegetable oil	FAPAS T07533QC	
Avocado puree	FAPAS T07566QC	Sector 2
Sunflower seeds	FAPAS T07567QC	Sector 3
Offal (liver)	FAPAS T07446QC	Sector 4
Powdered rice	FAPAS T07461QC	Sector 5
Powdered rice	FAPAS T07553QC	
Infant/adult formula	NIST SRM 1869	Sector 6
Spinach powder	NRC CRM SPIN-1	Sector 7
Soya flour	FAPAS T07458QC	Sector 8
Fish protein	NRC CRM DORM-5	Sector 9

Table S1b

Mass fractions of analytes in CRMs used.

CRM sample	Analyte (certified mass fraction, range of certified value)
NIST SRM 2387 Peanut butter	Ca (411 mg/kg, 393-429 mg/kg)
	Cu (4.93 mg/kg, 4.78-5.08 mg/kg)
	Fe (16.4 mg/kg, 15.6-17.2 mg/kg)
	Mn (16.0 mg/kg, 15.4-16.6 mg/kg)
	Na (4890 mg/kg, 4750-5030 mg/kg)
	Zn (26.3 mg/kg, 25.2-27.4 mg/kg)
NIST SRM 1869 Infant/adult formula	Ca (4560 mg/kg, 4430-4690 mg/kg)
	Cu (19.00 mg/kg, 18.62-19.38 mg/kg)
	Fe (164.7 mg/kg, 161.0-168.4 mg/kg)
	Mn (46.0 mg/kg, 44.4-47.6 mg/kg)
	Na (1877 mg/kg, 1824-1930 mg/kg)
NRC CRM SPIN-1 Spinach powder	Zn (144.0 mg/kg, 140.8-147.2 mg/kg)
	As (0.70 mg/kg, 0.58-0.82 mg/kg)
	Cd (0.265 mg/kg, 0.235-0.295 mg/kg)
NRC CRM DORM-5 Fish protein	Pb (2.33 mg/kg, 1.99-2.67 mg/kg)
	As (13.3 mg/kg, 12.6-14.0 mg/kg)
	Cd (0.148 mg/kg, 0.141-0.155 mg/kg)
	Pb (0.058 mg/kg, 0.052-0.064 mg/kg)
	Hg (2.33 mg/kg, 1.99-2.67 mg/kg)
	Cr (2.33 mg/kg, 1.99-2.67 mg/kg)

Table S1c

Mass fractions of analytes in QC materials.

QC material	Analyte (assigned value, acceptable range)
FAPAS T07533QC Vegetable oil	As (878 µg/kg, 592-1164 µg/kg)
	Cu (547 µg/kg, 356-739 µg/kg)
	Fe (797 µg/kg, 533-1060 µg/kg)
FAPAS T07566QC Avocado puree	Pb (307 µg/kg, 190-425 µg/kg)
	Cd (57.2 µg/kg, 32.0-82.3 µg/kg)
	Pb (61.0 µg/kg, 34.2-87.8 µg/kg)
FAPAS T07567QC Sunflower seeds	As (126 µg/kg, 71-180 µg/kg)
	Cd (279 µg/kg, 171-387 µg/kg)
	Pb (115 µg/kg, 64-166 µg/kg)
	Hg (41.9 µg/kg, 23.5-60.3 µg/kg)
FAPAS T07446QC Offal (liver)	As (857 µg/kg, 576-1138 µg/kg)
	Cd (675 µg/kg, 446-904 µg/kg)
	Pb (454 µg/kg, 291-618 µg/kg)
FAPAS T07461QC Powdered rice	As (223 µg/kg, 134-313 µg/kg)
	Cd (250 µg/kg, 152-349 µg/kg)
	Pb (131 µg/kg, 74-188 µg/kg)
FAPAS T07553QC Powdered rice	As (217 µg/kg, 130-305 µg/kg)
	Cd (93.2 µg/kg, 52.2-134.2 µg/kg)
	Pb (187 µg/kg, 110-264 µg/kg)
	Hg (122 µg/kg, 68-175 µg/kg)
FAPAS T07458QC Soya flour	As (767 µg/kg, 512-1022 µg/kg)
	Cd (445 µg/kg, 284-606 µg/kg)
	Pb (549 µg/kg, 357-741 µg/kg)

Table S2a

Typical ICP-MS conditions of Perkin Elmer 300X.

RF power	1500 W
Plasma gas flow rate	17 L/min
Auxiliary gas flow rate	1.3 L/min
Helium gas flow rate	4 mL/min
Sample uptake rate	-3 rpm (anti-clockwise)
Nebulizer	ST nebulizer
Spray chamber	Quartz, cyclonic, chilled to 2 °C
Acquisition mode	Spectrum
Replicates	3

Table S2b

Typical ICP-MS conditions of Agilent 8900.

RF power	1550 W
Plasma gas flow rate	15 L/min
Auxiliary gas flow rate	0.9 L/min
Helium gas flow rate	4 mL/min
Sample uptake rate	0.1 rps
Nebulizer	MicroMist nebulizer
Spray chamber	Quartz, Scott double pass, chilled to 2 °C
Acquisition mode	Spectrum
Replicates	3

Table S2c

Typical ICP-OES conditions of Spectro Arcos.

RF power	1410 W
Plasma gas flow rate	13 L/min
Auxiliary gas flow rate	0.7 L/min
Sample uptake rate	2.0 mL/min
Nebulizer	SeaSpray nebulizer
Spray chamber	Quartz, Zyclon Burgener
Replicates	3

Table S3a

Typical calibration ranges for analysis of As, Cd, Cr, Cu, Fe, Hg, Mn, Pb and Zn by ICP-MS.

Analyte	Calibration range (µg/L)	Internal standard (µg/L)
As	0.2 to 20	Ge (5)
Cd	0.1 to 10	In (5)
Cr	0.5 to 50	Ge (5)
Cu	0.8 to 80	Ge (5)
Fe	0.8 to 80	Ge (5)
Hg	0.05 to 5	Ir (5)
Mn	0.8 to 80	Ge (5)
Pb	0.05 to 5	Bi (5)
Zn	0.8 to 80	Ge (5)

Table S3b

Typical calibration ranges for analysis of Ca and Na by ICP-OES.

Analyte	Calibration range (µg/L)	Internal standard (µg/L)
Ca	100 to 2000	Lu (100)
Na	100 to 2000	Lu (100)

Table S4

Limits of quantification (LOQ) for various analytes.

Analyte	LOQ (mg/kg)	Technique
As	0.1	ICP-MS
Cd	0.01	ICP-MS
Cr	0.05	ICP-MS
Cu	0.08	ICP-MS
Fe	0.08	ICP-MS
Hg	0.005	ICP-MS
Mn	0.08	ICP-MS
Pb	0.005	ICP-MS
Zn	0.08	ICP-MS
Ca	10	ICP-OES
Na	10	ICP-OES

Table S5

Results obtained under two different sets of digestion conditions.

FAPAS QC material	Analyte (assigned value, acceptable range)	Results of 2.5 mL of conc. HNO ₃ & 2.5 mL of 30% H ₂ O ₂ (n = 2)	Results of 5 mL of conc. HNO ₃ (n = 2)
T07458QC Soya flour	As (767 µg/kg, 512-1022 µg/kg)	Mean = 774 µg/kg Recovery = 101 % RPD = 2.8 %	Mean = 754 µg/kg Recovery = 98 % RPD = 1.5 %
	Cd (445 µg/kg, 284-606 µg/kg)	Mean = 450 µg/kg Recovery = 101 % RPD = 1.6 %	Mean = 442 µg/kg Recovery = 99 % RPD = 0.8 %
	Pb (549 µg/kg, 357-741 µg/kg)	Mean = 555 µg/kg Recovery = 101 % RPD = 0.2 %	Mean = 545 µg/kg Recovery = 99 % RPD = 2.2 %
T07461QC Powdered rice	As (223 µg/kg, 134-313 µg/kg)	Mean = 226 µg/kg Recovery = 101 % RPD = 0.6 %	Mean = 225 µg/kg Recovery = 101 % RPD = 1.1 %
	Cd (250 µg/kg, 152-349 µg/kg)	Mean = 247 µg/kg Recovery = 99 % RPD = 0.4 %	Mean = 250 µg/kg Recovery = 100 % RPD = 0.9 %
	Pb (131 µg/kg, 74-188 µg/kg)	Mean = 125 µg/kg Recovery = 96 % RPD = 0.6 %	Mean = 124 µg/kg Recovery = 94 % RPD = 0.2 %
T07446QC Offal (liver)	As (857 µg/kg, 576-1138 µg/kg)	Mean = 846 µg/kg Recovery = 99 % RPD = 0.3 %	Mean = 841 µg/kg Recovery = 98 % RPD = 0.4 %
	Cd (675 µg/kg, 446-904 µg/kg)	Mean = 685 µg/kg Recovery = 101 % RPD = 0.4 %	Mean = 681 µg/kg Recovery = 101 % RPD = 0.5 %
	Pb (454 µg/kg, 291-618 µg/kg)	Mean = 442 µg/kg Recovery = 97 % RPD = 0.6 %	Mean = 437 µg/kg Recovery = 96 % RPD = 0.5 %

Table S6

Results obtained under two different digestion temperatures.

FAPAS QC material	Analyte (certified mass fraction, range of certified value)	Results of 210 °C digestion temperature (n = 3)	Results of 180 °C digestion temperature (n = 3)
T07533QC Vegetable oil	As (878 µg/kg, 592-1164 µg/kg)	Mean = 913 µg/kg Recovery = 104 % RSD = 3.2 %	Mean = 978 µg/kg Recovery = 111 % RSD = 1.4 %
	Cu (547 µg/kg, 356-739 µg/kg)	Mean = 526 µg/kg Recovery = 96 % RSD = 0.7 %	Mean = 533 µg/kg Recovery = 98 % RSD = 1.2 %
	Fe (797 µg/kg, 533-1060 µg/kg)	Mean = 792 µg/kg Recovery = 99% RSD = 2.6 %	Mean = 834 µg/kg Recovery = 105% RSD = 2.1 %
	Pb (307 µg/kg, 190-425 µg/kg)	Mean = 296 µg/kg Recovery = 96% RSD = 1.2 %	Mean = 293 µg/kg Recovery = 95% RSD = 0.3 %

Table S7

Results obtained under two different days (Intermediate precision study).

CRM	Analyte (certified mass fraction, range of certified value)	First day (June 2025) (n = 3)	Second day (August 2025) (n = 3)
Peanut butter NIST SRM 2387	Ca (411 mg/kg, 393-429 mg/kg)	Mean = 410 mg/kg Recovery = 100 % RSD = 0.4 %	Mean = 416 mg/kg Recovery = 101 % RSD = 0.2 %
	Between days Ca result (n = 6)	Mean = 413 mg/kg; Recovery = 101 % RSD = 0.8 %	
	Cu (4.93 mg/kg, 4.78-5.08 mg/kg)	Mean = 4.62 mg/kg Recovery = 94 % RSD = 0.7 %	Mean = 4.87 mg/kg Recovery = 99 % RSD = 1.1 %
	Between days Cu result (n = 6)	Mean = 4.75 mg/kg; Recovery = 96 % RSD = 3.0 %	
	Fe (16.4 mg/kg, 15.6-17.2 mg/kg)	Mean = 15.6 mg/kg Recovery = 95 % RSD = 1.1 %	Mean = 16.2 mg/kg Recovery = 98 % RSD = 3.0 %
	Between days Fe result (n = 6)	Mean = 15.9 mg/kg; Recovery = 97 % RSD = 2.8 %	
	Mn (16.0 mg/kg, 15.4-16.6 mg/kg)	Mean = 15.4 mg/kg Recovery = 96 % RSD = 1.6 %	Mean = 16.4 mg/kg Recovery = 102 % RSD = 1.0 %
	Between days Mn result (n = 6)	Mean = 15.9 mg/kg; Recovery = 99 % RSD = 3.5 %	
	Na (4890 mg/kg, 4750-5030 mg/kg)	Mean = 4922 mg/kg Recovery = 101 % RSD = 1.4 %	Mean = 5038 mg/kg Recovery = 103 % RSD = 0.8 %
	Between days Na result (n = 6)	Mean = 4980 mg/kg; Recovery = 102 % RSD = 1.6 %	
	Zn (26.3 mg/kg, 25.2-27.4 mg/kg)	Mean = 26.6 mg/kg Recovery = 101 % RSD = 1.3 %	Mean = 27.8 mg/kg Recovery = 106 % RSD = 0.7 %
	Between days Zn result (n = 6)	Mean = 27.2 mg/kg; Recovery = 103 % RSD = 2.7 %	