Microwave Digestion and ICP-MS for Reliable Trace Elemental Analysis in Clams

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Introduction

Clams are widely consumed due to their nutritional benefits, serving as rich sources of essential elements like iron, zinc, and magnesium. However, as bottom-dwelling organisms, they can accumulate environmental contaminants, including toxic heavy metals such as arsenic, cadmium, lead, and mercury, which pose significant health risks. In addition, products containing clams are susceptible to further contamination as they are processed. Ensuring food safety requires thorough analysis of both beneficial and harmful elements. This study evaluates trace elemental concentrations in various clam species, assessing both their nutritive and toxic elements. Microwave digestion is a critical sample preparation technique for accurate and reproducible trace elemental analysis. Using ICP-MS analysis following microwave digestion, this method offers a rapid, efficient, and reliable approach to trace elemental analysis in fresh and processed clams, supporting both nutritional assessment and food safety regulations.

Materials and Methods

Samples (digested in triplicate using CEM BLADE™ and MARS™ 6):

- NIST SRM 1566b Oyster Tissue
- Countneck, minced countneck, littleneck, and steamer clams

Digestion Method

1. Weigh 1 g of sample or 0.25 g of SRM in a BLADE Quartz or MARSXpress™ Plus TFM vessel.
2. Add acid mixture 7 mL HNO_3 + 1 mL HCl of trace-level grade acids to vessel. Add an additional 1 mL water to the MARS 6 SRM samples.
3. Cap vessel and place in BLADE autosampler or MARSXpress turntable.



BLADE Digestion Parameters

Stage	1
Temperature	220 °C
Ramp Time	4:00 (mm:ss)
Hold Time	4:00 (mm:ss)
Vent Profile	Organic



MARS 6 Digestion Parameters

Method	Classic
Control Type	Ramp to Temp
Power	1600 W
Ramp Time	20:00 (mm:ss)
Temperature	200 °C
Hold Time	15:00 (mm:ss)



Clear and Colorless Digests

Most digests were clear and colorless. Some particles were present in steamer clam digests. Digests were analyzed using an Agilent 7850 ICP-MS.

Results

Table 1. Percent Recovery and Percent RSD for NIST SRM 1566b Using BLADE and MARS 6 (n=3)

NIST SRM 1566b Oyster Tissue [ppb]		Mg	Fe	Cu	Zn	Se	As	Cd	Hg	Pb
BLADE	% Recovery	108.5	91.70	96.96	104.2	96.74	98.69	97.71	91.99	90.03
	% RSD	0.71	0.84	0.91	1.45	3.85	0.51	0.93	3.49	0.31
MARS 6	% Recovery	107.2	98.58	93.84	106.1	104.7	98.97	99.94	101.0	93.58
	% RSD	1.19	5.32	1.57	1.14	3.85	1.24	0.16	1.38	1.02

Table 2. Average Concentration (ppb) of the Big Four Heavy Metals in Different Types of Clams (n=3)

		As	Cd	Hg	Pb
Countneck Clams	MARS 6	1190.	60.11	6.99	109.7
	BLADE	1229	59.52	5.89	106.2
Minced Clams (Countneck)	MARS 6	1039	28.86	4.34	16.74
	BLADE	955.9	27.76	4.32	14.59
Littleneck Clams	MARS 6	1091	127.9	36.95	112.6
	BLADE	1007	107.1	28.13	130.5
Steamer Clams	MARS 6	2807	71.47	11.59	473.9
	BLADE	2620	63.29	9.12	605.4

Table 3. Nutritive Elemental Concentrations (ppm) in Different Types of Clams Using BLADE and MARS 6 (n=3)

		Al	Cu	Fe	Mg	Se	Si	Zn
Countneck Clams	MARS 6	2.59	1.30	18.5	767	0.27	8.18	14.1
	BLADE	3.15	1.29	16.6	737	0.25	11.4	14.0
Minced Clams (Countneck)	MARS 6	1.21	0.21	11.2	332	0.24	8.65	14.3
	BLADE	1.84	0.15	9.88	302	0.22	11.5	12.5
Littleneck Clams	MARS 6	1.72	2.32	15.7	981	0.24	7.47	8.97
	BLADE	1.31	1.47	17.4	993	0.24	8.46	8.56
Steamer Clams	MARS 6	404	0.99	853	1010	0.36	151	8.40
	BLADE	427	0.80	793	974	0.31	283	7.44

Conclusions

Proper monitoring and analysis of elemental impurities and nutritive elements in foodstuff is imperative for ensuring consumer safety. Efficient sample preparation to generate homogenous solutions for analysis plays a vital role in this process. In this study, both the BLADE and MARS 6 microwave digestion systems were able to achieve strong recoveries and RSDs for the SRM, validating the method. In addition, both systems were able to reliably digest real samples in preparation for ICP-MS analysis, examining both toxic as well as nutritive elements. Regardless of laboratory needs (high-throughput batch or automated sequential), a robust and reliable sample preparation solution is available for food testing.

Scan code to access more information on microwave digestion.