

# Microwave Digestion of Mixed Dietary Supplements for Trace Metals Analysis

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## Abstract

Confirmation of toxic heavy metals in dietary supplements is becoming a very common occurrence. The source of these metals is most typically from plant based protein as the metals are taken up into the plants from the soil they are grown in. The need for a faster way to process the samples for analysis is essential to ensure the safety of these products. The MARS 6 offers a high-throughput microwave digestion solution for the industry. Up to forty samples can be simultaneously processed. Using new iWave temperature control it is possible to run a variety of samples in a single batch which allows for labs to maximize their throughput.

A MARS 6 microwave digestion system with iWave temperature control and 75 mL MARSXpress vessels was used to prepare a variety of supplements in a single batch. Metals analysis was performed using ICP-OES technology.

## Introduction

The global dietary supplements market is valued at just over \$130 billion and rising at near double-digit growth annually. The majority of adults in the US take one or more dietary supplements daily, including vitamins, minerals, botanicals, and many other products. Vitamin supplements account for nearly half of the market value with the rest broken into many other nutraceutical products such as sport drinks, herbal supplements, and even foods such as yogurt, cereal and energy bars, and frozen baked goods.

In an effort to maintain the trust of consumers, members of the dietary supplement industry are supporting the efforts of independent organizations, such as the USP, to create and validate methods and certify their products.

## Instrumentation

Dietary supplement samples were digested in the CEM MARS 6, Figure 1, using iWave in-situ temperature measurement. iWave is a novel technology advancement that utilizes light emitting technology to measure the temperature of the actual sample and solution inside the vessels and does not require an internal probe. The ability to accurately control each vessel makes it possible to run multiple sample types in a single batch.

Samples were prepared in CEM 75 mL MARSXpress vessels. This is a simple to use 3 piece vessel with vent and reseal technology so that it safely vents excess gaseous pressure within the vessel and then reseals itself to maintain elemental integrity. The 75 mL vessel is large enough to safely digest 0.5 g and larger samples which is necessary to reach low limits of detection of some contaminants while maintaining a homogenous sample.

An Agilent 5110 ICP-OES was used to analyze the samples.



Figure 1. MARS 6 with 75 mL MARSXpress Vessels

## Procedure and Method

Samples ranging from 0.5 to 2.0 grams were weighed into 75 mL PFA MARSXpress liners. Nine mL of HNO<sub>3</sub> and 0.5 mL of both HCl and a 50 ppm spike of 8 elements were added. Finally, 10 mL of deionized water was added to each sample. The water helped passivate the reactions of the large and mixed samples. The vessels were sealed and inserted into a 40 position turntable and placed into the MARS 6 for digestion. A custom method was created with a ramp time of 20 minutes to 210 °C and a hold time of 15 minutes. Table 1 illustrates the sample types as well as sample sizes used for the sample preparation.

Figure 2. Samples Digested in Single Batch



Table 1. Samples and Weights

Sample Type	Sample Size
Whey Protein	1.0 g
Whey Protein	2.0 g
Ensure	0.5 g
Ensure	1.0 g
Ensure	1.5 g
Ensure	2.0 g
Nature Valley Bar	1.0 g
Power Bar	1.0 g
Rice Flour	1.0 g

### Method Parameters

Vessel: 75 mL PFA MARSXpress

Reagents:  
 9 mL HNO<sub>3</sub>  
 0.5 mL HCl  
 0.5 mL 50 ppm spike  
 10 mL DI H<sub>2</sub>O

Temperature: 210 °C

Ramp: 20 min

Hold: 15 min

## Results and Discussion

The samples represent a the full spectrum of dietary supplements from high carbohydrate to high protein. As expected, some samples heated much more rapidly than others when power was applied and the digestion process was initiated. In addition, the varied sample sizes had a big impact on heating. The accuracy of the iWave allowed successful control the batch of samples. Note in Figure 3 that the 2 g whey powder heated very quickly. iWave sensed this and immediately reduced power to prevent an exothermic reaction from occurring. The addition of water played a critical role in helping to keep the reaction subsided. Once that sample was under control, precise power was delivered to complete the digestion of all of the samples. During the hold phase, all samples were at similar temperature is illustrated in Figure 4. The MARS 6 with 75 mL MARSXpress vessels proved to be a great tool for acid digestion of mixed dietary supplements in a single batch.

Figure 3. Temperature Profile Early in Ramp

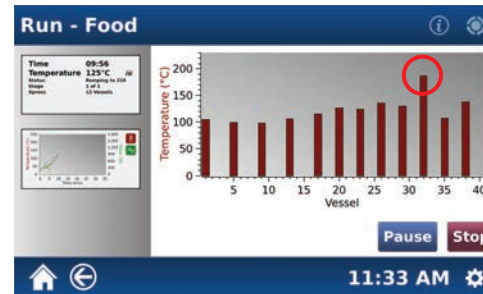
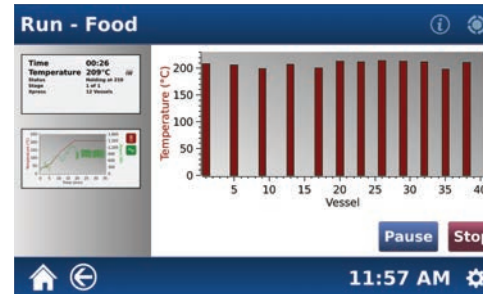


Figure 4. Temperature Profile During Hold



## ICP Results

The result shown in Table 2 represent spike recoveries from an 8 element spike applied to all samples.

Table 2. Spiked Recoveries

		Al	As	Fe	Hg	Pb	Sb	Se	Sn
Whey protein 1 g	Average	31.68	26.85	54.03	23.98	28.69	24.39	30.18	24.29
	% RSD	0.90	1.61	1.06	1.26	2.61	1.77	3.19	1.02
	% Recovery	126.72	107.40	216.10	95.91	114.77	97.58	120.70	97.15
Whey protein 2 g	Average	39.31	26.31	80.32	22.97	28.46	22.93	30.29	22.91
	% RSD	4.40	3.86	3.80	3.67	5.09	7.03	6.78	6.47
	% Recovery	157.24	105.26	321.30	91.88	113.86	91.70	121.15	91.65

Ensure 0.5 g	Average (ppm)	23.77	24.64	34.59	22.90	25.38	23.98	27.11	24.17
	% RSD	2.88	2.64	3.39	3.76	3.76	3.76	2.08	2.84
	% Recovery	95.07	98.56	138.35	91.59	101.50	95.93	108.44	96.68

Ensure 1.0 g	Average (ppm)	23.73	25.85	45.24	23.30	26.95	24.25	28.34	24.41
	% RSD	1.38	1.60	0.88	0.70	5.54	0.35	5.41	1.01
	% Recovery	94.92	103.40	180.95	93.21	107.81	97.01	113.34	97.63

Ensure 1.5 g	Average (ppm)	22.88	25.18	52.71	22.28	27.13	22.52	27.60	22.97
	% RSD	4.32	2.26	5.27	3.77	2.63	5.97	3.46	6.99
	% Recovery	91.51	100.74	210.83	89.13	108.54	90.08	110.41	91.88

Ensure 2.0 g	Average (ppm)	23.62	24.86	62.96	22.72	26.10	23.59	27.82	23.60
	% RSD	4.31	0.28	2.06	2.37	2.52	3.43	0.27	3.83
	% Recovery	94.47	99.46	251.83	90.89	104.39	94.35	111.27	94.38

Nature Valley 1 g	Average (ppm)	23.75	26.15	51.03	24.83	27.31	24.33	28.94	24.70
	% RSD	2.85	2.28	3.77	3.23	3.30	3.68	2.32	2.63
	% Recovery	90.57	104.13	99.99	96.52	87.39	94.46	110.31	93.98

Power Bar 1 g	Average (ppm)	31.03	25.47	96.38	23.72	27.35	23.96	28.52	24.41
	% RSD	4.42	2.20	3.77	2.72	2.88	4.03	2.34	4.62
	% Recovery	79.31	101.12	73.58	92.40	88.21	93.02	108.09	93.26

Rice flour 1 g	Average (ppm)	27.70	27.25	32.50	25.44	27.66	25.11	29.77	25.74
	% RSD	9.93	9.45	7.28	9.09	7.69	9.59	8.64	9.40
	% Recovery	96.75	108.07	104.14	99.49	99.12	98.48	113.27	100.07