

Digestion and Analysis of Mixed Vitamin Samples with Minimal Acid Volume

Figure 1: Digested Samples



Abstract

Increasing public and regulatory concerns over the safety of vitamin supplements mean manufacturers are under greater pressure than ever to ensure good manufacturing and testing processes. CEM Corporation has pioneered many of the food testing techniques in common use today. The new iWave contactless temperature measurement technology provides a revolutionary advance in reaction monitoring, allowing extremely accurate and responsive temperature measurement of multiple samples at the same time.

We used the MARS 6 system with iWave temperature control to prepare six vitamin samples in a single batch for acid digestion. Metals analysis was performed using ICP-OES technology.

Introduction

It is estimated that more than half of the adult population regularly take vitamin supplements, with annual sales of \$14.3 billion a year. However the sector is fraught with media reports of false or fraudulent medicinal claims, and there is already significant scrutiny and questioning from consumers. Public concerns about the safety of dietary supplements in general were heightened after quality issues (predominantly heavy metal contamination) were revealed in a quarter of 2000 dietary supplements from more than 300 manufacturers.

The FDA has issued requirements and expectations for good manufacturing practice to ensure the quality and safety of dietary supplements. In addition when the new U.S. Pharmacopeia (USP) chapters <232>/<233> are implemented in January of 2018, stricter requirements will be put in place to limit the presence of heavy metals in drug products.

Instrumentation

Vitamin samples were digested in the MARS 6 using iWave in-situ temperature measurement. iWave is a novel technology advance that utilizes Light Emitting Technology™ to measure the temperature of the actual sample solution inside the vessel and does not require an internal probe.

Samples were prepared using CEM 55 mL MARSXpress vessels. It is a simple to use three piece vessel design. Using vent and reseal technology the vessel can easily prepare samples such as vitamins. Up to forty samples can be digested in a single run. Although the MARSXpress 55 mL vessels were used for this analysis, these could be substituted with the 75 mL MARSXpress or MARSXpress Plus vessels.

An Agilent 700 Series ICP-OES was used to analyze the samples. All Samples were diluted to a final volume of 50 mLs prior to analysis.

Procedure and Method

0.25 grams of six types of vitamin were weighed into 55 mL PFA MARSXpress liners. 9 mL of HNO₃ and 1 mL HCl was added. The vessels were capped and then inserted in the 40 position turntable and placed in the MARS 6 for digestion. The One Touch Pharmaceutical method was used. The One Touch Technology automatically counts the number of vessels and recognizes the vessel type. It then chooses the optimized conditions for the acid digestion.

Samples:

- Pantothenic Acid
- Ascorbic Acid
- Magnesium Oxide
- Manganese Carbonate
- Chromium AA Chelate
- Selenium Chelate

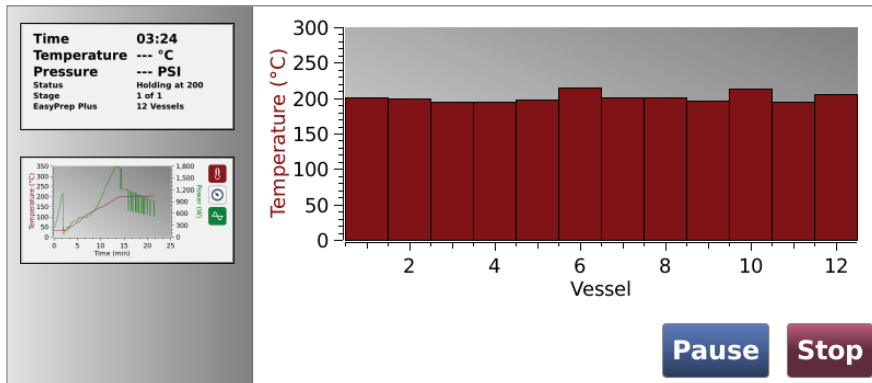
Acid mixture:

9 mL HNO₃
1 mL HCl

Results and Discussion

Use of the MARS 6 plus iWave allowed efficient processing of six different vitamin samples for elemental analysis. As illustrated in **Figure 2** the system automatically adjusts the power to compensate for the varied samples, so that by the end of the ramp, all samples are digesting similarly. The overall time to process these samples, including cool down, was 45 minutes. As illustrated in **Figure 1** most solutions are clear upon dilution with deionized water. However certain transition elements will provide for a colored aqueous solution. In our case, the transition elements Cr and Mn, present in the Chromium AA Chelate and Manganese Carbonate supplements, caused slight coloration of the digested samples.

Figure 2: Bar Graph of Vitamins



iWave technology provides precise control of the temperature of all samples during the digestion process.

ICP Results

The results shown in **Figure 3** are an average of the duplicate runs. The samples were spiked with 1 mL of a 100 ppm solution of As, Cd, Fe, Pb, Hg, and Se, making the final spike a 2 ppm recovery upon dilution to 50 mL. Results are good for each element including the volatile element As.

Figure 3: Mixed Vitamin Sample Results

Vitamins	Pb 182.143	As 188.980	Cd 226.502	Fe 238.204
Calapan	1.5867	1.9818	2.0705	2.0187
Ascorbic Acid	1.7946	1.8786	2.1786	2.1026
Magnesium Oxide	2.0617	2.1477	2.1715	2.0018
Manganese Carbonate	2.1039	2.0883	2.3416	2.1379
Chromium Chelate	1.8556	1.9305	2.1879	2.1443
Selenium Chelate	2.0715	2.1836	2.2564	2.2338
True Value	2.000	2.000	2.000	2.000
Average	1.9123	2.0351	2.2011	2.1065
% Recovery	95.6	102	110	105
STD DEV	0.2037	0.1232	0.0910	0.0864
% RSD	10.652	6.052	4.135	4.101