Microwave Digestion of Large Mixed Food Samples with Varied Sample Weights

Abstract

Microwave digestion has been the method of choice for preparing samples for elemental analysis in food for over two decades. The ability to digest and analyze a food sample large enough to be considered representative and homogeneous has posed a problem due to the pressure limits of the digestion vessel. CEM recently introduced a new high performance vessel designed to work with larger sample sizes. iPrep's patented dual seal technology will hold much higher temperatures and pressures than typical digestion vessels. Using this vessel and iWave advanced temperature control, mixed food samples with large and varied sample weights were digested in a single run.

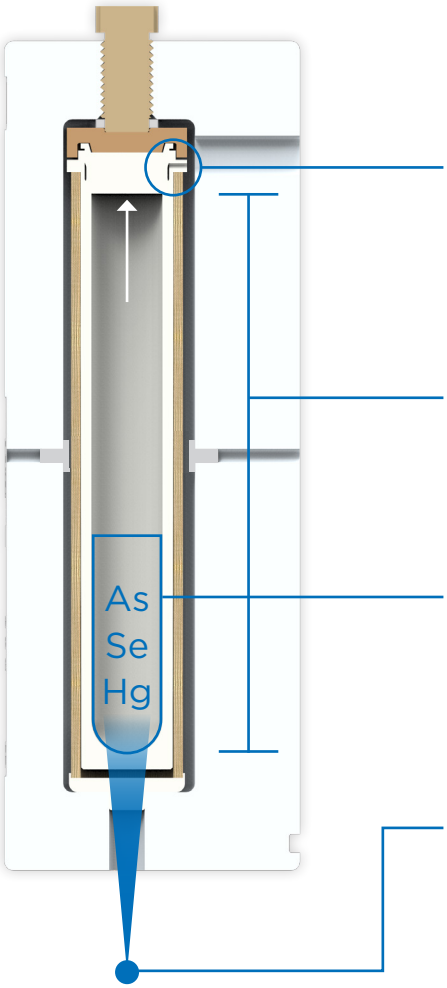
Introduction

The ability to analyze heavy metals in food materials is essential for food safety and consumer health. Arsenic and other heavy metals such as cadmium, lead, and mercury are considered toxic and have adverse health effects in human metabolism. These toxins are only tolerated at extremely low concentrations and excesses are associated with many adverse health effects. They may injure the kidney and cause symptoms of chronic toxicity, including impaired organ function and tumors. Exposure to lead has been associated with reduced IQ and general learning disability. In addition to safety, food analysis is also important to ensure product quality and to determine country of origin as well as possible counterfeiting. This application note will focus on the use of iPrep vessels to prepare a variety of food types using much larger sample sizes than previously possible. These samples will include foods high in sugars, proteins and fat. The food materials, with varied sample sizes, will be digested in a single run in order to illustrate the versatility of the preparation.

Instrumentation

Four unique samples were prepared in a single batch in a CEM MARS 6 microwave digestion system equipped with iWave technology. iWave is a novel Light Emitting Technology™ that is able to measure the temperature of the actual sample solution inside the vessel and does not require an internal probe. Samples were digested using CEM iPrep vessels. The patented dual-seal design (**Figure 1**) provides for higher temperatures and fine control of the vent and reseal process necessary for these sample types. iPrep is a simple to use three piece vessel which used a hand torque device that only requires 21 inch pounds of force to seal the vessel.

Figure 1: iPrep Vessel Diagram



Dual-Seal Design

The high temperature and pressure conditions afforded by this seal and vessel design provide for the complete digestion of difficult organics, such as PET, bunker oil, organic dyes, toner, thermoplastics, and many other difficult to digest materials.

2X Capacity

In addition, its large 110 mL volume allows for larger sample sizes, as compared to other high performance vessels.

Elemental Integrity

The dual-seal function provides for unmatched control of the gaseous byproducts from digestions such as CO_2 and NO_x fumes. These are precisely vented outside the vessel, while maintaining the full integrity of all elements. Even volatile analytes such as As, Se, and Hg.

iWave Light Emitting Technology (LET)

iWave utilizes the emission of light from the sample (solution) to rapidly and accurately measure temperature. It reads the temperature of the solution itself, instead of reading the temperature of the vessel. This makes iWave the most accurate temperature sensor on the market.

Procedure and Method

Pieces of pepperoni and cheese were sliced and weighed and added directly to the iPrep liner. The milk powder was weighed in to the liner as received. The meat loaf frozen dinner entrée was first blended, to ensure homogenaty, and then weighed into the iPrep liner. The sample types as well as the approximate weights are recorded in **Table 1** below. Ten mL of concentrated HNO_3 was added to each sample. The vessels were capped and assembled and then placed in the MARS 6 for digestion. The vessels were distributed evenly around the cavity in positions illustrated in **Table 1** below. The One Touch iPrep method Food was used. One Touch Technology automatically recognizes the vessel type and counts the number of vessels. It then calculates optimized conditions for the acid digestion. The maximum temperature achieved using this method is 210°C. Food samples in general do not require extremely high temperatures to achieve complete digestion.

Table 1: Samples and approximate weights

Sample	Vessel Positions	Sample Weight
Milk Powder	1, 7	1.0 g
Pepperoni Stick	2, 8	2.0 g
Cheddar Cheese Block	4, 10	2.0 g
Mixed Frozen Entrée	5, 11	5.0 g

Acid used: 10 mL concentrated HNO_3 in each vessel

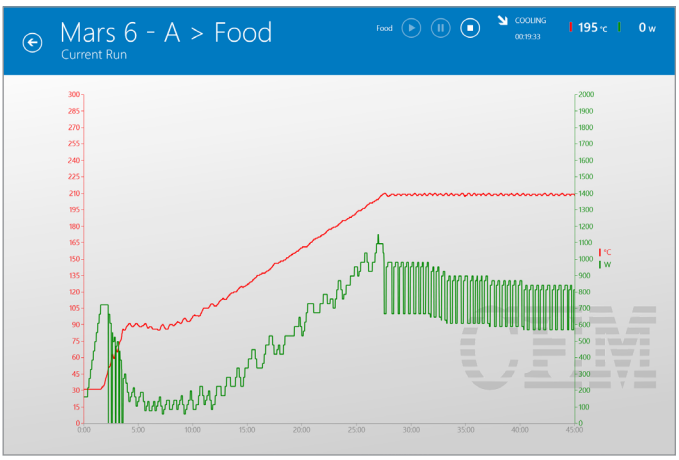
Results and Discussion

The MARS 6 with iPrep vessels and iWave temperature control simultaneously processed the four different food samples with varied sample weights. Each sample was run in duplicate for a total of eight samples in the digestion run. The milk powder samples in vessels 1 and 5 begin to digest first and achieve higher initial temperatures due to the high carbohydrate value. iWave detects this temperature surge and quickly reduces power as illustrated by the green power curve in **Figure 3** in order to keep the samples from going exothermic. Once the initial reaction subsides power is again increased. Towards the end of the digestion, the pepperoni sample is the highest in temperature as illustrated in **Figure 4**. This is due to the high fat content of the samples which require higher temperatures to initiate decomposition. Note that the duplicate samples are nearly identical in temperature for each sample type and that the milk powder samples are now the lowest in temperature having completely digested early in the run. The precise temperature control of iWave and the high pressure capacity of the iPrep vessel allowed for the total digestion of the mixed food and sample sizes as illustrated in **Figure 5**.

Figure 2: Bar Graph Early in Run

The milk powder samples digest quickly due to high carbohydrate content.

Figure 3: Temperature and Power Line Graph

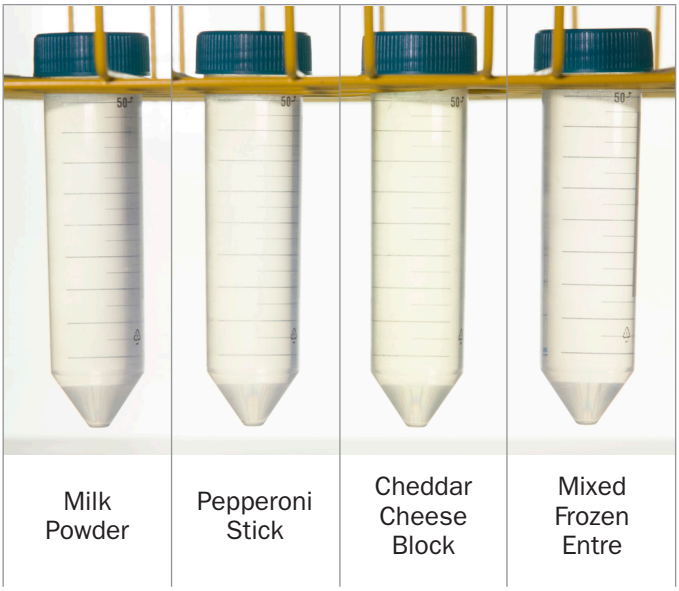


The iWave temperature control system immediately reduces power (green line) in order to avoid exothermic reaction of milk powder samples.

Figure 4: Bar Graph Late in Run

At the end of the run the high fat pepperoni sample is now the highest in temperature.

Figure 5: Photos of the Samples



Digested samples diluted to 50 mL.