

Digestion and Analysis of Food Samples

Abstract

Accurate trace metals determination in foods is critical for both labelling purposes and consumer safety. The main source of food-related contamination comes from environmental sources, e.g., soil and water, but contamination can also occur as the food is being processed. The big four: arsenic, lead, cadmium, and mercury, are the largest concern due to their adverse impacts on human health. However, many other metals may be required for the purposes of accurate nutritional labelling. In most cases, a combination of toxic and nutritive elements is simultaneously analyzed by either Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) or Inductively Coupled Plasma Mass Spectrometry (ICP-MS).

Prior to analysis, sample preparation must be completed. Foods vary greatly in composition of fat, protein, and carbohydrates, complicating their sample prep. During acid digestion, carbohydrates will digest quickly and can create exothermic reaction conditions, while proteins and fats require higher temperatures to completely digest. One common practice to deal with these potentially large differences is to batch samples that are similar in nature; the disadvantage to batching similar samples is the analysis bottleneck that is potentially created.

CEM recently pioneered a major advancement in microwave digestion technology. Combining a Single-Mode Cavity™ (SMC) with rapid vortexing, the BLADE™ automated microwave digestion system can digest these difficult samples and many more quickly, completely, and without requiring similar samples in a single batch. The BLADE automatically loads each sample into the microwave cavity for acid digestion and cooling. Once completed, it is returned to its original rack position and the next sample is processed. Each vessel can contain a different material and acid combination, providing ultimate flexibility for laboratory work flow.

Introduction

Each year, an estimated 48 million people in the United States get sick and 3,000 die from foodborne diseases.¹ In an effort to curb those statistics, in December of 2010, Congress passed the Food Safety Modernization Act (FSMA) which was signed into law in January of 2011. The act overhauled regulations regarding food production and gave the Food and Drug Administration (FDA) more authority to oversee and enforce supply chains.

In order to help ensure foods are safe and comply with these new regulations, the FDA has published analytical methods that manufacturers or importers can use in their laboratories. One of the methods includes the FDA Elemental Analysis Manual (EAM) EAM 4.7 for ICP-MS.² This analytical method in particular outlines the sample preparation process using microwave-assisted acid decomposition.

The purpose of this study was to develop microwave digestion procedures on a variety of food products from the food pyramid, as well as Standard Reference Materials (SRMs) using the CEM BLADE microwave digestion system. A modified FDA EAM 4.7 methodology was used for the acid digestion step. Samples were analyzed for metals determination of major constituents as well as trace metal impurities of the prepared samples.

Materials and Methods

Samples were chosen, based on their composition, to include a high protein sample (beef jerky), a starch or carbohydrate (rice), along with two foods high in fats and oils (mayonnaise and olive oil). To add depth to the sample list, turmeric was included as an example of metals analysis in spices for both toxicity and geographic origin. Three NIST Standard Reference Materials (SRMs) were also digested, in order to validate methodology. These included Meat Homogenate (NIST SRM 1546a), Whole Milk Powder (NIST SRM 1549a) and Slurried Spinach (NIST SRM 2385).

Instrumentation

The BLADE automated microwave digestion system was used to prepare a variety of food samples (see **Figure 1** on page 2). The BLADE is a high-performance system that combines speed, simplicity, and automation to process samples sequentially. Samples were weighed into the 60 mL quartz vessel with a stir bar and acid was added. The vessel was sealed with a snap-on cap, simplifying vessel assembly. The vessels were then loaded into one of two digestion racks. The system automatically loaded the vessel into the cavity where the digestion was performed and the vessel cooled, after which, it was returned to its original rack position. Once complete, the next sample was automatically processed.

Every sample was under individual temperature and pressure control, which was documented and stored. The built-in camera allows for visual monitoring of the digestion process, a unique means to confirm digestion for method development, or simple troubleshooting. Since samples are processed sequentially, any combination of sample and acid types can be placed into the rack for digestion. Samples were analyzed on an Agilent 7850 ICP-MS (Agilent Technologies, Santa Clara, CA).



Figure 1. BLADE System with Quartz Vessels

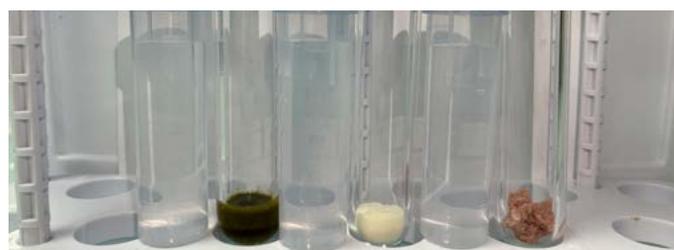


Figure 2. Samples Pre and Post Digestion (After Dilution).
L to R: SRM 2385 Slurried Spinach, SRM 1549a Whole Milk Powder, SRM 1546a Meat Homogenate



Figure 3. Samples Pre and Post Digestion (After Dilution).
L to R: Instant Rice, Beef Jerky, Olive Oil, Mayo, Ground Turmeric

Sample Preparation

Approximately 0.5 gram of each sample was weighed into a quartz BLADE vessel. An acid mixture of 4 mL of HNO₃, 0.5 mL of HCl and 0.5 mL H₂O₂ was added to each vessel, which was then sealed with the simple snap-on cap. The BLADE allowed for a reduction of the acid volume, which provides easier dilutions, lower blank values, and cost savings on reagents. To simplify the process, the same program was used for every sample. The digestion parameters are shown below in **Table 1**. All samples were digested in triplicate and were clear, colorless, and particle free upon dilution to 100 mL, with the exception of the turmeric. The spice turmeric is derived from the root of the plant and, as such, will uptake minerals including silicates from the soil in which it is grown. As a result, a small amount of white particulate remained in the digested sample. Silicates require hydrofluoric acid (HF) in order to break those bonds and provide a complete dissolution. A filtration step may be completed prior to analysis if HF is not available. The samples, both pre and post digestion are shown in **Figure 2** and **Figure 3**.

Table 1. Digestion Parameters of Various Types of Food

Sample	Acid (s)	Temp. Required	Ramp Time (min)	Hold Time (min)
SRM 1546a	4 mL HNO ₃ + 0.5 mL H ₂ O ₂ + 0.5 mL HCl	230 °C	4:00	4:00
SRM 1549a	4 mL HNO ₃ + 0.5 mL H ₂ O ₂ + 0.5 mL HCl	230 °C	4:00	4:00
SRM 2385	4 mL HNO ₃ + 0.5 mL H ₂ O ₂ + 0.5 mL HCl	230 °C	4:00	4:00
Beef Jerky	4 mL HNO ₃ + 0.5 mL H ₂ O ₂ + 0.5 mL HCl	230 °C	4:00	4:00
Olive Oil	4 mL HNO ₃ + 0.5 mL H ₂ O ₂ + 0.5 mL HCl	230 °C	4:00	4:00
Turmeric	4 mL HNO ₃ + 0.5 mL H ₂ O ₂ + 0.5 mL HCl	230 °C	4:00	4:00
Rice	4 mL HNO ₃ + 0.5 mL H ₂ O ₂ + 0.5 mL HCl	230 °C	4:00	4:00
Mayonnaise	4 mL HNO ₃ + 0.5 mL H ₂ O ₂ + 0.5 mL HCl	230 °C	4:00	4:00

Analysis

The metals of interest were selected based upon the certified values of the SRMs, as well as industry concerns regarding trace metal contaminants. Samples were analyzed on an Agilent 7850 ICP-MS with conditions listed in **Table 2**. The conditions listed were used to analyze all elements except Ca, Fe, and Se which were analyzed using “H2” tuning mode. The following elements were used as internal standards: Li, Sc, Ge, In, Rh, and Tb. No gas dilution techniques were used for this study.

Table 2. Instrument Conditions of the Agilent 7850 ICP-MS

Parameter	Value for [He] Mode
RF Power (W)	1550
Sampling Depth (mm)	8
Carrier Gas (L/min)	1.05
Dilution Gas	N/A
Helium Cell Gas (mL/min)	5.0
Energy Discrimination	5.0

Table 3. Percent Recoveries for Three NIST SRM Food Samples

2385 Slurried Spinach (N= 3)			1549a Whole Milk Powder (N= 3)			1546a Meat Homogenate (N= 3)		
Element	Cert Value mg/kg	% Recovery	Element	Cert Value mg/kg	% Recovery	Element	Cert Value mg/kg	% Recovery
Na	47	93.86	Na	3176	100.23	Na	9600	97.37
Mg	368	101.01	Mg	892	96.76	Mg	178.1	105.64
K	3650	98.47	K	11920	96.11	K	2490	97.67
Ca	624	86.96	Ca	8810	94.84	Ca	360	95.86
Mn	3810	100.08	Mn	0.184	89.37	Mn	0.286	95.69
Fe	17.1	94.58	Fe	n/a	n/a	Fe	10.17	88.11
Zn	8.37	105.06	Zn	33.8	99.85	Zn	17.88	103.44
Cu	0.9	87.37	Cu	n/a	n/a	Cu	0.605	92.8
			Se	0.242	90.74	Se	0.281	102.17
			Sr	2.14	96.13	Sr	0.305	104.9
			Ba	0.566	99.19	Ba	0.077	96.95
						Mo	0.016	94.54

Results and Discussion

The SRMs chosen represent three food materials with differing compositions, including a vegetable, a meat, and a dairy product. Recoveries ranged from 87 – 105% with the majority of recoveries between 95 and 100%, as shown in **Table 3**. The results validate that the sample preparation step provided an ideal matrix for ICP-MS analysis.

While none of the samples had high values of any of the big four metals (As, Pb, Cd, Hg), there are a few highlights of interest for rice, beef jerky, and turmeric. The rice sample exhibits the highest content of As, which has been reported in various studies in recent years, but it is not at a level that would cause concern. Zinc, an element commonly used as an animal feed supplement, was found to be the highest in concentration in the beef jerky sample.³ The turmeric showed a high concentration of Pb. Lead chromate is a common adulterant found in spices such as turmeric, which is becoming a growing food safety concern.⁴ To confirm the high levels found in this study, additional work will need to be completed.

Table 4. Metals Recoveries of Food Samples in Our Study

Sample	Instant Rice	Beef Jerky	Olive Oil	Mayo	Ground Turmeric
Element	mg/kg				
Mg	147.48998	545.81846	0.44490	10.52736	1825.13387
K	416.23561	6515.95372	4.62604	88.37060	31211.08095
Ca	76.53873	353.85796	0.80470	67.86973	1541.80060
Mn	8.48363	0.85182	0.04656	0.05351	148.73338
Fe	10.11218	36.33313	0.15371	1.98255	715.77627
Cu	1.84437	1.32996	0.01828	0.05260	4.09711
Zn	13.99955	63.68335	0.90525	1.92541	18.14652
As	0.12888	0.01021	0.00049	0.00098	0.09856
Se	0.22792	0.12325	0.00016	0.02480	0.03970
Mo	0.48272	0.04631	0.00065	0.03019	0.23136
Cd	0.01253	0.00116	0.00011	0.00011	0.03975
Hg	0.00416	0.00025	0.00013	0.00018	0.00138
Pb	0.00175	0.00603	0.00276	0.00136	0.30594

Conclusion

The BLADE successfully digested many types of food samples. With a single programmed method and reduced acid volume, the BLADE proved to simplify sample preparation methodology. The reduced acid concentration in the diluted samples was ideal for obtaining lower detection limits and extending the lifetime of hardware for the ICP-MS. The high temperature used in the procedure, coupled with rapid vortexing, permitted the complete destruction of the organic matrix. This, in turn provided a solution that yielded excellent recoveries for all of the elements certified for the three SRMs. With fast digestion times and automation, the BLADE allows for multi-tasking in the laboratory to help streamline workflow.

References

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