

Elemental Analysis Manual

for Food and Related Products

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2.3 Analytical Portion to Analytical Solution

Version 1.0 (June 2008)

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2.3.1 MICROWAVE DIGESTION

GLOSSARY

This section provides information to assist the analyst on preparing the analytical solution. The analytical method may contain more specific instructions that must be followed.

2.3.1. MICROWAVE DIGESTION

Microwave digestions are performed using commercial equipment specifically made for performing acid digestions.

Analytical Portion for Closed Vessels

Pressure built up by digestion products governs maximum analytical portion mass.

Typical analytical portions will range from 0.5 to 2 g. High-fat foods require a lower mass while a larger mass can be used with high-water content foods. Often maximum analytical portion may be determined using a sample's known energy (*i.e.*, caloric) content as an indicator of pressure

produced during digestion. For some products (*e.g.*, juice concentrates) the energy content may not be known. Maximum energy release permitted for 600 and 800 psi digestion vessels (90 mL capacity) was empirically determined as 3 and 6 kcal, respectively. This maximum vessel energy prevents a digestion from reaching the vessel's maximum operating pressure before digestion is complete. The energy of an analytical portion must not exceed these values for safe operation. An analytical portion should not exceed 5 g even if calculation indicates that a larger portion could be taken. This maximum mass will prevent excess dilution of the nitric acid used for digestion and ensure a complete digestion. Use 1 g reagent water for method blanks (MBKs). For samples of unknown composition, a maximum of 0.5 g dry weight should be analyzed for safety concerns. If maximum pressure attained for this unknown is less than the vessel limit then a greater mass may be analyzed. In many cases the maximum analytical portion mass will not be necessary or should not be used, *e.g.*, the high level of salt in some food may interfere with an analysis.

A food's energy content (usually provided as kcal/100 g) is available from many sources¹⁻². In addition, a food's energy content may be estimated from the calories and serving size provided on a food's label.

An example using a sports nutrition bar is given below and 2.3 Table 1 provides typical analytical portion masses for selected foods when using 800 psi digestion vessels.

Example: The nutrition label lists a serving size as the entire bar with a mass of 50 g and an energy value of 210 Calories (Consumer label "Calories" are kilocalories). Calculate the kcal/g:

$$\text{kcal/g} = \frac{210 \text{ kcal}}{50 \text{ g}} = 4.2 \text{ kcal/g}$$

Maximum mass for an 800 psi digestion vessel (6 kcal energy maximum) is calculated using the following equation:

$$\text{Maximum analytical portion(g)} = \frac{\text{Vessel Max. Energy (kcal)}}{\text{Food Energy (kcal/g)}} = \frac{6 \text{ kcal}}{4.2 \text{ kcal/g}} = 1.4 \text{ g}$$

Therefore, 1.4 g or less of this sports nutrition bar could be digested in an 800 psi microwave vessel.

Although these calculations will reveal the maximum aliquot that can be safely decomposed in a vessel, generally, about 1 g is recommended for an analytical portion. Approaching the maximum amount adds a risk of causing matrix effects during the determinative step.

2.3 Table 1. Typical Analytical Portion Mass for Selected Foods

<i>When Using 800 psi Microwave Digestion Vessels</i>			
Food	Portion (g)	Food	Portion (g)
American cheese	1.2	Lettuce, Iceberg	8
Beef liver	2.4	Nuts, mixed	0.8
Peaches, canned	8	Peanut butter	0.8
Spaghetti, canned	5	Raisin bran cereal	1.6
Dill pickles	4.0 ^a	Tomato catsup	3.0 ^a
Spinach	8	Yellow mustard	3.0 ^a
Fruit cocktail, canned	8	White bread	1.8
^a Recommended portion is less than the calorie limit of the digestion vessel due to the interference produced by the high-salt content.			

Analytical Portion for Venting Vessels

Limit vent and reseal type vessels (approximately 90 mL capacity) to a maximum of 9 kcal or 0.8 g dry weight unless the equipment manufacturer recommends a lower limit.

Digestion Procedure

If possible, place analytical balance in clean hood (Class 100) or on clean bench to minimize contamination while weighing analytical portions. Transfer analytical portion with a pipette, spatula or by pouring and into a tared, clean digestion vessel liner. Avoid placing analytical portions on walls of digestion vessels. Determine mass of analytical portion to an accuracy of 0.001 g. Move vessel liner to an exhausting clean hood and wash down any material on walls and wet sample with 1 to 2 mL reagent water. Do not add more than 2 mL of water. Pipette 7.0 mL or weigh 10.0 g of high purity nitric acid (sp gr 1.41 g/mL) into vessel liner. Weighing acid using a top loading balance and Teflon[®] FEP wash bottle is suggested. Some foods, especially those high in sugar, will react with nitric acid within several minutes. If foaming or reaction with the acid is observed, let the vessels sit uncovered in a Class 100 clean hood for 20 minutes or until reaction subsides. If a clean hood is unavailable, cap vessels but loosen the pressure relief nut (with the safety membrane) to allow pressure to escape. Seal vessels, tighten pressure relief nuts and run microwave digestion program as prescribed by the analytical method. After vessels have cooled to less than 50 °C remove to an exhausting clean hood and vent excess pressure slowly. Open vessels and add 2 mL high purity 30% hydrogen peroxide. Reseal vessels (tighten pressure relief nuts if equipped) and run the second stage of the microwave decomposition program. After vessels have cooled to less than 50 °C remove them to an exhausting clean hood and vent excess pressure slowly. Quantitatively transfer and dilute digestion solution with reagent water as prescribed by the analytical method. This analytical solution should be transferred to a plastic bottle or a capped polypropylene centrifuge tube for storage.

Note: Analysts should be cognizant that the addition of concentrated nitric acid to some samples types may cause a strong exothermic reaction (especially those high in sugar). If the vessel is not immediately capped and sealed, a loss of nitric acid could occur through evaporation. Likewise, a portion of the sample can be lost through mechanical means when the sample reacts vigorously in an open vessel. For samples that behave in this manner, cap the vessels immediately and

allow the sample to complete this exothermic reaction stage. When convenient, leave the vessel over night to go through this exothermic stage and cool. The cooled vessels should be vented before the heating program is initiated. For vent and reseal type vessels (such as CEM Omni vessels), this may allow the use of slightly larger sample portions. For sealed vessels (such as CEM XP-1500 vessels) this step may prevent vessel blowout when dealing with samples that have unknown behavior. Membranes are not a dependable defense against vessel blowout caused by sudden high pressure during the heating cycle.

Note: After diluting to volume the analytical solutions should be clear and colorless to slightly yellow. Turbidity and/or a deep color usually indicate an incomplete digestion. Insoluble food ingredients may be present (e.g. titanium dioxide, silica). In cases of an incomplete digestion, determine if there was a malfunction of the microwave digestion system such as lack of safety membrane, safety membrane cap not tight, wrong oven program etc. A copy of the computer generated graph of the digestion run's temperature and pressure versus time is helpful towards diagnosing digestion problems. Correct problem and re-digest sample. If microwave digestion system did not malfunction then re-digest sample using a smaller (at least a factor of 2 less) analytical portion. Some foods (especially spinach) contain silica, which will not dissolve in this procedure. A very small amount of white silicate precipitate from these foods is to be expected and is not a problem. Centrifugation can separate these particles from the analytical solution. However, some matrices (i.e., certain dietary supplements) may yield a high proportion of undigested materials after the heating cycle. Additional treatments (e.g., hydrofluoric acid, smaller analytical portion) may be required for accurate results.

A typical microwave digestion program and a typical second stage microwave digestion program for peroxide oxidation are given in 2.3 Table 2.

2.3 Table 2. Microwave Digestion Programs

<i>Digestion Programs for CEM MARS 5 with 12-Position Carousel with Ramp to Temperature Feature</i>		
<i>Power is applied for the Ramp Time minutes or until Control Pressure or Control Temperature is met. If Control Pressure or Control Temperature are met before end of Ramp Time then program proceeds to Hold Time</i>		
	Digestion	Peroxide Oxidation
Maximum Power (Watts)	1200	1200
Control Pressure (psi) ^a	800	800
Ramp Time (min)	25	10
Hold Time (min)	10	5
Control Temperature (°C)	200	200
^a Only use with non-venting vessels.		

Microwave Digestion Vessel Cleaning

Digestion vessels are acid cleaned after each digestion. Vessels being used for the first time or after an incomplete digestion are cleaned with liquid laboratory-grade detergent and then subjected to the acid cleaning. Incomplete digestions are usually dark colored (yellow to brown), have a bad odor and may contain material that did not dissolve. The manufacturer of the microwave digestion equipment may provide additional information on cleaning vessels and other components of the equipment. Be careful not to use anything that can scratch the vessel walls. The Teflon material is relatively soft and can scratch easily.

Detergent cleaning—Disassemble vessels and soak for at least 2 hours in a solution of liquid laboratory-grade detergent and hot water. Thermowells should be wiped down with a paper towel and detergent solution. Rinse thermowells and vessel components with warm tap water and rinse thoroughly with reagent water. Allow to dry in a clean area; preferably dry in a Class 100 clean area.

Acid cleaning—Add 10 mL of nitric acid to each vessel and microwave them according to the Clean Program listed in 2.3 Table 3 or as suggested by the manufacturer. After vessels have cooled to less than 50 °C remove from oven and vent excess pressure slowly in a fume hood. Disassemble vessels, rinse off covers and liners with reagent water into a waste container. Thoroughly, rinse covers and liners with copious quantities of reagent water. Dry in a clean area; preferably dry in a Class 100 clean area. Outside surfaces of vessels may be dried with laboratory tissues. If vessels are not used after drying, store assembled in a Class 100 clean area or other appropriate contamination free environment.

2.3 Table 3. Microwave Digestion Clean Program

<i>Program for CEM MARS 5 with 12-Position Carousel with 12-Position Carousel with Ramp to Temperature Feature</i>	
Stage:	1
Maximum Power (Watts)	1200
Control Pressure (psi)	{Not Used}
Ramp Time (min)	10
Hold Time (min)	3
Control Temperature (°C)	200

REFERENCES

- (1) Souci, S. W., Fachmann, W., and Kraut, H. (1994) Food Composition and Nutrition Tables, 5th Ed., CRC Press, Boca Raton, FL
- (2) U.S. Department of Agriculture, Agricultural Research Service (2007) *USDA Nutrient Database for Standard Reference, Release 20*, Nutrient Data Laboratory Home Page, (Accessed March 31, 2008) ([link removed](#)). Note: Release numbers change as new versions are released.