

# Elemental Analysis Manual

## for Food and Related Products

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## 2.2 Analytical Sample to Analytical Portion

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### GLOSSARY

This section provides information to assist the analyst on physically preparing the analytical sample prior to taking an analytical portion for analysis. Obtaining representative analytical portions is critical for accurate and reproducible results. EAM methods assume the analyst has made the analytical sample homogeneous with respect to the size of the analytical portion. Homogenized foods will vary greatly in texture and viscosity and may separate into liquid and solid components. Analytical samples that have been stored after being homogenized usually require re-homogenization before acquiring an analytical portion. The analyst must carefully observe the physical characteristics of the homogenate and choose an appropriate means of obtaining a representative analytical portion (*e.g.*, pouring directly from container possibly during active mixing, using a 1, 5 or 10 mL pipette or using a spatula).

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*Note: If non-homogeneity is known or suspected then replicate analytical portions should be analyzed and the results assessed.*

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### 2.2.1. LABORATORY HOMOGENIZATION EQUIPMENT

- [Food] Blenders<sup>1</sup>

A blender is a kitchen appliance used to blend ingredients or puree food. The term typically refers to a stationary, upright electrical device, which is to be distinguished from a hand-powered or electric mixer that may be used for similar purposes.

A typical blender is built around a vessel for the ingredients to be blended. At the top of the vessel is a cap to prevent ingredients from escaping when the blender is switched on. At the bottom is a blade assembly, typically removable for cleaning purposes. The bottom seal is most likely watertight. The vessel rests upon a base containing a motor (for turning the blade assembly) with controls on its surface. Most modern blenders offer a number of possible speeds.



- Food Processors<sup>2</sup>

A food processor is a kitchen appliance used to facilitate various repetitive tasks in the process of preparation of food. Today, the term usually refers to an electric-motor-driven appliance, although there are some manual devices also referred to as "food processors".

Food processors are similar to blenders in many ways. The primary difference is that food processors use swappable blades and disks (attachments) instead of a fixed blade. In addition, their bowls are wider and shorter, a more appropriate shape for the solid or semi-solid foods usually worked in a food processor. Usually little or no liquid is required in the operation of the food processor unlike a blender, which requires some amount of liquid to move the particles around its blade.



- Laboratory Homogenizers<sup>3</sup>

Laboratory homogenizers are high-speed, high-shear mixers that reduce samples to uniform-sized particles through maceration, cutting, and blending. They are used to process liquids, slurries, or granular substances. Product specifications for laboratory homogenizers include media viscosity, capacity, feed rate, motor speed, motor power, pressure range, and operating temperature. There are three basic types of laboratory homogenizers: fluidized bed, rotor-stator, and ultrasonic. Fluidized-bed homogenizers are durable vessels that fluidize the complete product bed. Rotor-stator homogenizers are single-shaft mixers with an impeller that rotates in close proximity to a stationary housing. Ultrasonic or vibrational homogenizers apply ultrasonic waves in a mixed medium to produce a steep gradient of acoustical pressure and, therefore, fluid movement and a very-fine level of mixing action. Homogenizers that produce high shear and ultrasonic waves can disrupt cellular structure of some foods leading to a better homogenization of the material.



- Cryogenic Mills

Cryogenic laboratory mills cool samples to cryogenic temperatures and pulverize them by magnetically shuttling a steel impactor back and forth against two stationary end plugs. The vial is immersed in liquid nitrogen throughout the grinding cycle.



- Mortar Grinders

Mortar grinders force the samples against two hard surfaces (i.e., mortar and pestle) to grind the sample by a combination of pressure and friction. A choice of materials in contact with the sample is available including agate, zirconium oxide, stainless steel, tungsten carbide and porcelain.



- Rotor Mills

Rotor mills use a stainless steel beater rotating at a high speed (3000-28000 rpm) to cut up the sample by impact and shearing.



## 2.2.2. HOMOGENIZATION PROCEDURES

### 2.2.2.1. General Procedures

Examples of homogenization equipment used for specific types of foods are listed in 2.2 Table 1.

**2.2 Table 1. Suggested Procedure for Preparing Analytical Sample<sup>a</sup>**

Food Product	Suggested Procedure
Apple (red), raw (with peel)	Food Processor
Applesauce, bottled	Blender
Apricot, raw	Blender
Asparagus, fresh/frozen, boiled	Food Processor
Avocado, raw	Food Processor
Bagel, plain, toasted	Food Processor
Banana, raw	Food Processor
Beans, kidney, dry, boiled	Food Processor
Beef chow mein, from Chinese carry-out	Food Processor
Beef stew, homemade	Blender
Beef stroganoff with noodles, homemade	Food Processor
Beef, ground, regular, pan-cooked	Food Processor
Beef, meatloaf, homemade	Food Processor
Beef, roast, chuck, oven-roasted	Food Processor
Beef, steak, loin, pan-cooked	Food Processor
Beer	Mix by Hand
Beets, fresh/frozen, boiled	Food Processor
Beverage, carbonated, cola, low-calorie	Mix by Hand
Beverage, carbonated, cola, regular	Mix by Hand
Beverage, carbonated, fruit-flavored, regular	Mix by Hand
Beverage, fruit drink (10% juice), canned or bottled	Blender

Food Product	Suggested Procedure
BF, applesauce	Blender
BF, bananas	Blender
BF, beef and broth/gravy	Blender
BF, carrots	Blender
BF, chicken and broth/gravy	Blender
BF, chicken noodle dinner	Blender
BF, corn, creamed	Blender
BF, fruit dessert/pudding	Blender
BF, green beans	Blender
BF, infant formula, milk-based, high iron, RTF	Blender
BF, Infant formula, milk-based, low iron, RTF	Blender
BF, Infant formula, soy-based, RTF	Blender
BF, juice, apple	Mix by Hand
BF, juice, orange	Mix by Hand
BF, macaroni, tomato and beef	Blender
BF, mixed vegetables	Blender
BF, peaches	Blender
BF, pears	Blender
BF, peas	Blender
BF, rice cereal with apples	Blender
BF, rice infant cereal, instant, prepared with milk	Blender
BF, spinach, creamed	Blender
BF, split peas with vegetables & ham/bacon	Blender
BF, squash	Blender
BF, sweet potatoes	Blender
BF, teething biscuits	Food Processor
BF, turkey and rice	Blender
BF, vanilla custard/pudding	Blender
BF, vegetables and beef	Blender
BF, vegetables and chicken	Blender
BF, vegetables and ham	Blender
Bologna (beef/pork)	Food Processor
Bread, biscuits, refrigerated-type, baked	Food Processor
Bread, cornbread, homemade	Food Processor
Bread, cracked wheat	Food Processor
Bread, muffin, fruit or plain	Food Processor
Bread, rye	Food Processor
Bread, white roll	Food Processor
Bread, white, enriched	Food Processor
Bread, whole wheat	Food Processor
Broccoli, fresh/frozen, boiled	Food Processor
Brownie	Food Processor
Brussels sprouts, fresh/frozen, boiled	Food Processor
Butter, regular (salted)	Food Processor
Cabbage, fresh, boiled	Food Processor
Cake, chocolate snack with chocolate icing	Food Processor
Cake, chocolate with icing	Food Processor

Food Product	Suggested Procedure
Cake, yellow, with white icing, prepared from mixes	Food Processor
Candy, caramel	Separately weigh an equal amount of deionized water and candy. Heat water to boiling in a 2-quart stainless steel bowl. Add candy with continuous stirring using large stainless steel spoon. After candy has melted, pour into appropriate containers. [MCF = 0.5]
Candy, hard [Hard candy: Based on sugars cooked to the hard-crack stage, including suckers (known as boiled sweets in British English), lollipops, jawbreakers (or gobstoppers), lemon drops, peppermint drops and disks, candy canes, rock candy, etc.] <sup>4</sup>	Separately weigh an equal amount of deionized water and candy. Heat water to boiling in a 2-quart stainless steel bowl. Add candy with continuous stirring using large stainless steel spoon. After candy has melted or dissolved, pour into appropriate containers. [MCF = 0.5]
Candy, milk chocolate bar, plain	Food Processor. Avoid inducing elevated temperatures that cause phase separation.
Cantaloupe, raw/frozen	Blender
Carrot, fresh, peeled, boiled	Food Processor
Cauliflower, fresh/frozen, boiled	Food Processor
Celery, raw	Food Processor
Cereal, corn flakes	Food Processor
Cereal, cream of wheat (farina), enriched, cooked	Food Processor
Cereal, crisped rice	Food Processor
Cereal, fruit-flavored, presweetened	Food Processor
Cereal, oat ring	Food Processor
Cereal, oatmeal, plain, cooked	Food Processor
Cereal, raisin bran	Food Processor
Cereal, shredded wheat	Food Processor
Cheese, American, processed	Food Processor
Cheese, cheddar, natural (sharp/mild)	Food Processor
Cherries, sweet, raw	Blender
Chicken breast, oven-roasted (skin removed)	Food Processor
Chicken nuggets, fast-food	Food Processor
Chicken potpie, frozen, heated	Food Processor
Chicken, fried, fast-food	Food Processor
Chicken, fried - homemade	Food Processor
Chili con carne with beans, homemade	Food Processor
Coleslaw with dressing, homemade	Food Processor
Collards, fresh/frozen, boiled	Food Processor
Cookies, Chocolate chip	Food Processor
Cookies, sugar	Food Processor
Cookies, sandwich with crème filling	Food Processor
Corn, cream style, canned	Blender
Corn, fresh/frozen, boiled	Food Processor
Corn, hominy grits, enriched, cooked	Food Processor
Corn/tortilla chips	Food Processor
Cottage cheese, 4% milk fat	Blender
Crackers, butter-type	Food Processor

Food Product	Suggested Procedure
Crackers, graham	Food Processor
Crackers, saltine	Food Processor
Cream cheese	Food Processor
Cream substitute, non-dairy, liquid/frozen	Blender
Cream, Half & half	Blender
Cucumber, peeled, raw	Food Processor
Cucumber, pickles, Dill	Food Processor
Cucumber, pickles, sweet	Food Processor
Doughnut, cake-type	Food Processor
Egg, cheese, and ham on English muffin, fast-food	Food Processor
Eggplant, fresh, peeled, boiled	Food Processor
Eggs, boiled	Food Processor
Eggs, fried	Food Processor
Eggs, scrambled with oil	Food Processor
English muffin, plain, toasted	Food Processor
Fish sandwich on bun, fast-food	Food Processor
Fish sticks or patty, frozen, oven-cooked	Food Processor
Frankfurter (beef/pork), boiled	Food Processor
Frankfurter on bun, fast-food	Food Processor
French fries, frozen, heated	Food Processor
Frozen meal - Salisbury steak, heated	Food Processor
Frozen meal - turkey, heated	Food Processor
Fruit cocktail, canned in light syrup	Blender
Granola with raisins	Food Processor
Grapefruit, raw	Blender
Grapes, red/green, raw	Food Processor
Green beans, fresh/frozen, boiled	Food Processor
Ham, cured (not canned), baked	Food Processor
Hamburger, quarter-pound on bun, fast-food	Food Processor
Hamburger, quarter-pound with cheese on bun, fast-food	Food Processor
Honey	Mix by Hand
Ice cream, light, vanilla	Blender
Ice cream, regular, vanilla	Blender
Jelly, any flavor	Blender
Juice, apple, bottled	Mix by Hand
Juice, grape, frozen concentrate, reconstituted	Blender
Juice, grapefruit, bottled	Blender
Juice, orange, frozen concentrate, reconstituted	Blender
Juice, prune, bottled	Mix by Hand
Juice, tomato, bottled	Mix by Hand
Lamb chop, pan-cooked with oil	Food Processor
Lasagna with meat, homemade	Food Processor
Lemonade, frozen concentrate, reconstituted	Blender
Lettuce, iceberg, raw	Food Processor
Lima beans, immature, frozen, boiled	Food Processor
Liver (beef/calf), pan-cooked with oil	Food Processor
Luncheon meat (ham)	Food Processor
Luncheon meat, salami, (not hard)	Food Processor
Macaroni and cheese, prepared from box mix	Food Processor
Macaroni, boiled	Food Processor
Margarine, regular (salted)	Food Processor
Martini	Mix by Hand

Food Product	Suggested Procedure
Mayonnaise, regular, bottled	Food Processor
Milk shake, chocolate, fast-food	Blender
Milk, chocolate, low fat, fluid	Blender
Milk, evaporated, canned	Blender
Milk, low fat (2%), fluid	Blender
Milk, skim, fluid	Blender
Milk, whole, fluid	Blender
Mixed vegetables, frozen, boiled	Food Processor
Mushrooms, raw	Food Processor
Mustard, yellow	Mix by Hand
Noodles, egg, enriched, boiled	Food Processor
Nuts, mixed, no peanuts, dry roasted	Blender
Oil, olive/safflower	Mix by Hand
Okra, fresh/frozen, boiled	Food Processor
Olives, black	Food Processor
Onion, mature, raw	Food Processor
Orange (navel/Valencia), raw	Blender
Pancakes from mix	Food Processor
Peach, canned in light/medium syrup	Blender
Peach, raw/frozen	Blender
Peanut butter, creamy	Food Processor
Peanuts, dry roasted, salted	Food Processor
Pear, canned in light syrup	Blender
Pear, raw (with peel)	Food Processor
Peas, green, frozen, boiled	Food Processor
Peas, mature, dry, boiled	Food Processor
Pepper, sweet, green, raw	Food Processor
Peppers, green, stuffed, homemade	Food Processor
Pie, apple, fresh/frozen	Food Processor
Pie, pumpkin, fresh/frozen	Food Processor
Pineapple juice, frozen concentrate, reconstituted	Blender
Pineapple, canned in juice	Blender
Pinto beans, dry, boiled	Food Processor
Pizza, cheese and pepperoni, regular crust, from pizza carry-out	Food Processor
Pizza, cheese, regular crust, from carry-out	Food Processor
Plums, raw	Food Processor
Popcorn, popped in oil	Food Processor
Popsicle, fruit-flavored	Blender
Pork and beans, canned	Blender
Pork bacon, oven-cooked	Food Processor
Pork chop, pan-cooked with oil	Food Processor
Pork roast, loin, oven-roasted	Food Processor
Pork sausage (link/patty), oven-cooked	Food Processor
Potato chips	Food Processor
Potato, baked (with peel)	Food Processor
Potato, boiled (without peel)	Food Processor
Potato, french-fried, fast-food	Food Processor
Potatoes, mashed, from flakes	Food Processor
Potatoes, scalloped, homemade	Food Processor
Pretzels, hard, salted	Food Processor
Prunes, dried	Food Processor



Food Product	Suggested Procedure
Radish, raw	Food Processor <hr/> Alternative: Food Processor with separately weigh equal amounts of deionized water and food product. [MCF = 0.5]
Raisins	Food Processor <hr/> Alternative: Food Processor with separately weigh equal amounts of deionized water and food product. [MCF = 0.5]
Rice, white, enriched, cooked	Food Processor
Salad dressing, French, regular	Mix by Hand
Salad dressing, Italian, low-calorie	Blender
Salmon, steaks/fillets, baked	Food Processor
Sauerkraut, canned	Food Processor
Sherbet, fruit-flavored	Blender
Shrimp, boiled	Food Processor
Soup, bean with bacon/pork, canned, condensed, prep with water	Blender
Soup, chicken noodle, canned, condensed, prep with water	Blender
Soup, clam chowder, New England, canned, condensed, prep with whole milk	Blender
Soup, mushroom, canned made with whole milk	Blender
Soup, tomato, canned, condensed, prep with water	Blender
Soup, vegetable beef, canned, condensed, prepared with water	Blender
Sour cream	Blender
Spaghetti with meat sauce, homemade	Food Processor
Spaghetti with tomato sauce, canned	Blender
Spinach, fresh/frozen, boiled	Food Processor
Squash, Summer, fresh/frozen, boiled	Food Processor
Squash, winter (Hubbard/acorn), fresh/frozen, boiled	Food Processor
Strawberries, raw/frozen	Food Processor
Sugar, white, granulated	Mix by Hand
Sweet potato, fresh, baked	Food Processor
Sweet roll/Danish pastry	Food Processor
Syrup, chocolate	Mix by Hand
Syrup, pancake	Mix by Hand
Taco/tostada with beef and cheese, from Mexican carry-out	Food Processor
Tomato catsup	Blender
Tomato sauce, plain, bottled	Blender
Tomato, raw	Food Processor
Tomato, stewed, canned	Blender
Tortilla, flour	Food Processor
Tuna noodle casserole, homemade	Food Processor
Tuna, canned in oil	Food Processor
Turkey breast, oven-roasted	Food Processor
Turnip, fresh/frozen, boiled	Food Processor
Veal cutlet, pan-cooked	Food Processor



Food Product	Suggested Procedure
Water, tap	Mix by Hand
Watermelon, raw/frozen	Blender
Whiskey	Mix by Hand
Wine, dry table, red/ white	Mix by Hand
Yogurt, low fat, fruit-flavored	Blender
Yogurt, plain, low fat	Blender
<sup>a</sup> Based on instructions used in FDA's Total Diet Study. BF: Baby Food.	

#### 2.2.2.2. Candy Procedures

Some types of candy products contain small particulates as sources of contaminants. Traditional homogenization procedures may not achieve a sufficiently homogenous analytical sample for the relatively small portions analyzed. This inhomogeneity causes greater measurement variability than expected and is difficult to discern from other sources of measurement variability. The high variability of the results, while reflecting the true inhomogeneous nature of the contaminant, may discredit the quality of the analysis. In an effort to overcome this problem, the analytical sample is blended with dilute nitric acid and heated to solubilize the contaminants<sup>5-6</sup>. The objective of the procedure given below is to produce a homogeneous analytical sample from which replicate analytical portions can be taken with the expectation of accurate and precise analytical results. Supporting information for the procedure is available<sup>7</sup>.

The potential application of this procedure includes varieties of hard and soft candy, powdered candy products, and fruit candy products, especially candies that have chili or other ingredients that may be the source of the contaminant of interest. However, because of the diversity in the physical nature of candy products this procedure may not be adequate for all confectionary products.

- *Laboratory Sample.* To reduce inherent within lot composition variability of candy products, a relatively large laboratory sample is collected for preparation of the analytical sample. A laboratory sample should consist of a maximum of 10, 2 lbs (0.91 kg) subsamples. The subsamples should be sealed retail units from the same manufacturing code. If the candy is packed in a sealed bag of at least 2 lbs, then the each of the 10 subsamples is a bag of candy. Otherwise, each subsample is the number of bags that provide at least 2 lbs of candy.
- *Analytical Sample.* The amount of the laboratory sample subjected to homogenization to prepare the analytical sample should be approximately 1 kg. Take equal portions of each of the 10 subsamples to obtain the 1 kg.
- *Equipment.* A food processor is required and laboratory homogenizer may be needed. The procedure described below uses portions that can be contained in about a 6-liter stainless steel bowl of a food processor. The volume of sample plus the volume added water and acid used to aid homogenization should be limited to approximately 2 liters to allow proper blending action. The analytical sample must be contained in a commercial food processing vessel or a beaker that may need to accommodate an equivalent weight of water or acid.
- *Method Blanks.* Prepare duplicate method blanks in an identical fashion as preparing the analytical sample.
- *Mass Correction Factor.* An analytical portion for analysis is obtained from the treated analytical sample preparations described below. In the analytical method's calculation to obtain the analyte concentration this analytical portion mass must be

corrected for the added solvent (water or nitric acid) using a mass correction factor (MCF) (*see* §3.4.6). To calculate MCF both the mass of the analytical sample and the mass of all added solvent(s) are required. Therefore, these masses must be determined and recorded during the homogenization procedures described below. Determine the mass of the materials at the end of any heat treatment cycle in order to account for the losses of liquid through vaporization.

### **Candy Product Types**

- Type I: Semi-solid or soft candy (*e.g.*, chocolate-based, fruit-based, including tamarind, tejocote, apple, etc.) with or without visible particulate ingredients.
- Type II: Hard candy (including suckers) with or without visible particulate ingredients either in the candy or on the surface of the candy.
- Type IIA: Hard candy (including suckers) with supplemental (but separate) ingredients (*e.g.*, salt, chili).
- Type III: Powdered sugar or flavored salt products with or without other particulate ingredients (*e.g.*, chili).

### **Homogenization of Candy Type I Products**

- (1) Water treatment—Add the analytical sample, approximately 1 kg, to the food processor bowl. Add a mass of water (ASTM Type I) approximately equal to the mass of the analytical sample except for chocolate-based products. Some chocolate-based products may have too much fat to blend with water. Chocolate should be blended first without water and then an equal mass of water added. At this stage, the mixture can be left over night to soften or partially dissolve. If the sample will not disperse by blending, heating may be required (*e.g.*, on a hot plate). Blend the sample in the food processor until a visually homogenous composite is obtained. This mixture needs to have paste-like consistency in order to disperse particles and to permanently suspend all ingredients. This paste-like composite must be stable (*i.e.*, no phase separation) until an analytical portion(s) is removed for acid treatment. If blending with water fails to form a paste-like consistency for Type I product, then treat the product as you would for a Type II product.
- (2) Concentrated nitric Acid treatment—Weigh duplicate portions equivalent to about 40 g of product into beakers. Nitric acid washed glass beakers, polypropylene (translucent) or polymethylpentene (transparent) beakers can be used (heat stable to ~150° C). To prevent deformation a water bath should be used to heat plastic beakers. Add concentrated nitric acid (Metals grade) (20 ml initially) and heat to approximately 100° C until the product has been largely solubilized. Be careful not to heat the mixture too rapidly for this may cause excessive foaming and loss of mixture. The beakers should be covered with watch glasses to minimize contamination. More nitric acid may be added to achieve a more complete dissolution.

### **Homogenization of Candy Types II, IIA, and III Products**

10% Nitric Acid Treatment—Add the analytical sample, approximately 1 kg, to a beaker. For analysis of supplementary packets from Type IIA candies (typically a mixture of salt and chili), combine the number of packets (separately from the candy) that would be equal to the numbers

of candy used to make the composite. Add an equal mass of 10% nitric acid. Heat the mixture (to approximately 100° C) until the sample mass is largely dissolved. This step may need to be left over night to maximize dissolution. The remaining particulate materials should be subjected to further homogenization with a laboratory homogenizer such as a laboratory homogenizer. This final mixture should be highly acidic and should be largely a solution with respect to the analyte. Many candy ingredients and some natural product may not completely dissolve after the solubilization procedure. Silicon dioxide, chili flakes, and titanium dioxide common additives will not dissolve in this type of procedure. However, lead metal and other metallic ingredients should be completely dissolved. This liquid should now be in a satisfactory state for removing the analytical portion(s). If particulates persist in the product then stir vigorously while removing the analytical portions.

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