

Analysis of Ash Contents of Foods

A. Introduction

Ash is the inorganic residue from the incineration of organic matter. The ash content is determined from the loss of weight which occurs during complete oxidation of the sample at a high temperature (usually 500 to 600°C) through volatilization of organic materials. The ash obtained is not necessarily of exactly the same composition as the mineral components present in the original food because there may be losses via volatilization or some interaction between constituents.

For complete ashing, the heating is continued until the resultant ash is uniform in color, white or gray, occasionally green or reddish, and free from particles of unburned carbon and fused lumps. Ashing may be performed by incineration over an open flame, in a muffle furnace, in a closed system in the presence of oxygen, or by wet combustion in the presence of sulfuric acid, nitric acid, and perchloric acid alone or in mixtures.

The determination of ash content is of value in the analysis of food for various reasons. The ash content can be regarded as a general measure of quality in certain foods such as tea, flour and edible gelatine, and often is a useful criterion in identifying the authenticity of a food. The ash analysis has been chiefly used for the determination of adulteration of certain foods. A high ash figure suggests the presence of an inorganic adulterant, and this condition is advisable to determine the acid-insoluble ash. The presence of large amounts of ash in finished products such as sugar, starch, gelatin, fruit acids, or pectin is objectionable. The ashing of vegetable and plant materials, particularly cut sections, is recognized as a useful

tool in determining the nature and distribution of mineral constituents of plants. The ash content is an index of the quality of feed stuffs used for poultry and cattle. The ash content serves as a reliable index of the metabolism of yeast.

Total ash content is a useful parameter of the nutritional value of many foods and feeds. It is helpful with many foods to quantify not only the total ash but also the ash soluble and insoluble in water, the alkalinity of the soluble ash and of the total ash, and the proportion of ash insoluble in acids. High levels of acid-insoluble ash indicate the presence of sand or dirt in sample.

B. Methods of Ashing

There are two major procedures of ashing, which are eventually utilized to determine mineral contents of the sample. These methods are dry ashing and wet ashing techniques: in dry ashing, the organic matter of the sample is oxidized by complete incineration at a high temperature in the presence of oxygen, where as, in wet ashing, the sample is oxidized with a mixture of concentrated strong acid. In addition to the two direct methods, there are indirect ashing techniques such as conductometric methods which can determine the total electrolyte content of foods. The nature of the ashing procedure is determined by the purpose for which the ash is prepared, the particular constituents that are to be determined, and the method of analysis to be used.

1. Dry Ashing

Dry ashing is the most standard method to determine ash content of a sample. The total ash content of a foodstuff is the inorganic residue remaining after the organic matter has been burnt away. In dry ashing, the sample is ignited at 550o-600oC to oxidize all organic materials without flame. The inorganic material which does not

volatilize at that temperature is called ash. The required equipments and general procedure of dry ashing method for total ash are described in the following:

a. Equipment and Apparatus

- (1) Muffle furnace (electric), thermostatically controlled.
- (2) Porcelain crucible, Coors #1 or platinum dish
- (3) Analytical balance, 0.1-mg sensitivity
- (4) Desiccator, charged with efficient desiccant
- (5) Tongs, muffle
- (6) Marking ink, permanent type for crucibles

Additional equipments for wet samples

- (1) Bunsen burner
- (2) Hot plate thermostatically controlled
- (3) Tripod, iron
- (4) Steam bath
- (5) Atmospheric oven
- (6) Wash bottle
- (7) Double deionized water

b. Procedure

- (1) Place new or clean marked crucibles in a muffle furnace at 600°C for one hour. Turn off the furnace, transfer cooled crucibles from furnace to a desiccator, and cool to room temperature.
- (2) Weigh them as quickly as possible to prevent moisture absorption. Use metal tongs to move the crucibles after they are ashed or dried (crucible preparation).
- (3) Weigh accurately 2 g of dry foods or 10 g of wet samples into the prepared, pre-weighed crucible. If alkalinity of ash is to be determined, calculations are simplified if exactly these weights are used.
- (4) For wet sample, evaporate the wet sample to dryness on a hot plate or steam bath or in a atmospheric oven at 100°C for 1 h. Omit this step for dry samples.
- (5) Carbonize the samples under the hood. Place the sample crucibles on iron tripod over a Bunsen burner or hot plate and slowly char for about 30 min. Heat cautiously to prevent spattering, and add a few drops of olive oil to reduce spattering.
- (6) Place the charred crucibles in muffle furnace set at 550°C. Burn off the samples until they become completely free from carbon to be a light gray or white color ash.
- (7) If black carbon spots persist in the sample crucible due to incomplete oxidation of tough samples (i.e. animal tissues), turn off the furnace, add a few drops of concentrated nitric acid directly onto the unburnt spots, and switch on the furnace again. Raise the temperature to 600°C and leave 1-2 hrs depending upon the incompleteness of the ashing.

(8) Transfer the ashed sample crucible to a desiccator and cool to room temperature. When cooled, weigh the crucible as quickly as possible to prevent moisture absorption.

(9) Save the ash sample if mineral determinations are to be made.

c. Calculations:

$$\% \text{ ash} = \frac{\text{weight of residue}}{\text{weight of sample}} \times 100$$

2. Wet Ashing

Wet ashing is used primarily for the digestion of samples for determination of mineral elements. Wet oxidation method is preferable to the dry ashing procedures for the decomposition of organic matter prior to the determination of mineral constituents because they obviate difficulties resulting from losses of more volatile constituents during dry ashing and slow solution of the residue after ashing.

The use of a single acid is desirable, but usually not practical for the complete decomposition of organic material. Nitric acid alone is a good oxidant, but it usually boils away before the sample is completely oxidized. The use of perchloric acid with nitric acid or nitric-sulfuric acid mixtures has been suggested for the rapid decomposition of many organic compounds which are difficult to oxidize.

In wet ashing, the organic matter of the sample is oxidized with concentrated nitric and perchloric acids. The acids are partially removed by volatilization and the soluble mineral constituents remain dissolved in nitric acid. Any silica present is

dehydrated and made insoluble. The equipment, reagents, and general procedures of wet ashing methods are in the following:

a. Equipment

(1) Fume hood, constructed for safe exhaustion of perchloric acid and nitric acid fumes.

(2) Hot plate, thermostatically controlled.

(3) Volumetric flasks. 100 ml, 250 ml, pyrex or equivalent quality made of borosilicate glass.

(4) Watch glass

(5) Goggles (protective glasses)

(6) Glass rod, for sample stirring and transfer.

(7) Boiling beads

(8) Air-oven

(9) Steam bath

b. Reagents

(1) Nitric acid, 69 to 71% concentrated

(2) Digestion acid, add 1 volume perchloric acid (60 to 62% HClO_4) to 4 volumes nitric acid (69 to 71% HNO_3).

(3) Hydrochloric acid, approximately 2M. Add 1 volume hydrochloric acid (36% HCl) to 5 volumes deionized water.

c. Procedure

(1) Weigh accurately 1 or 2 g dry and ground sample into a 250 ml beaker, and add 20 ml digestion acid and 3 glass boiling beads under a fume hood. Cover the beaker with a watch glass. Wear the goggles.

(2) For liquid foods such as wine, juices and beverages an aliquot of the sample should first be dried in a hot-air oven or steam bath, then dry residue should be ashed with digestion acid.

(3) After addition of the digestion acid, allow the reaction to proceed in the hood at room temperature for 3-4 hr with occasional swirling using hands and glass rod, or leave overnight.

(4) Place the sample beaker on a hot plate maintained at the liquid in the beaker just simmers. When the initial reaction has subsided, increase the temperature of the hot plate to 180 to 200°C.

(5) Continue the digestion until the fluid in the beaker become clear and no visible particles are remained. If the solution darkens when the volume is reduced, remove the flask from the hot plate, add 1 or 2 ml nitric acid and continue the digestion with occasional swirling.

(6) Raise the temperature of the hot plate to 240°C and evaporate the digestion acid until dense white fumes are formed within the beaker.

(7) Remove the beaker from the hot plate. Transfer the solution quantitatively to a 100 ml volumetric flask with deionized distilled water.

(8) Stopper the flask, thoroughly mix the solution, and leave overnight. The water and acid insoluble silica will settle to the bottom and aliquots of the solution are drawn from the top, filtered, and stored in small polyethylene bottles for later analyses.

(9) The sample solution prepared can be used for the determination of the various macro and trace minerals by atomic absorption spectrophotometry or other methods.

Caution: Perchloric acid is a violent oxidant and explosive. Therefore, a special care should be exercised for all use of perchloric acid by using protective glasses and fume hood.

3. Conductometric Method

Conductometric methods are indirect methods for measuring the total electrolyte content of foods. Conductometric procedures provide a simple, rapid, and accurate means of determining the ash content of sugars. Foods containing high sugar (i.e. sucrose, syrup or molasses) are generally low in minerals and requires oxidation of a large amount of samples which have strong foaming carbohydrates.

The principle of conductometric methods is based on the fact that the mineral matter which constitutes the ash of the sugar dissociates in a solution, while the sucrose, a non-electrolyte, does not dissociate. Therefore, the conductance of the solution is an index of the concentration of the ions present, which is the mineral or ash content of the sample.

4. Comparison of Dry Ashing with Wet Ashing

There are quite a few advantages and disadvantages of drying ashing method in determination of ash and mineral compared with those of wet ashing method. Dry

ashing is the most commonly used procedure to determine the total mineral content of foods. Dry ashing procedure is used to measure water soluble, water insoluble, and acid insoluble ash. The advantages and disadvantages of the two direct ashing methods.

5. Soluble and Insoluble Ash

A finally prepared ash sample is composed of soluble and insoluble portions in water and/or acids. To determine water insoluble ash, the ash in the crucible (dish) is solubilized with about 25ml deionized water after weighing the total ash content. A watch glass is covered over the dish to avoid loss by spattering, and heated nearly to boiling. The ash solution is filtered through an ashless filter paper, and washed with equal volume of hot water. The filter paper and residue are placed again in the crucible, ignited and weighed. From that weight, the water insoluble ash is calculated and the water soluble ash is determined by the difference.

The acid insoluble ash can be also measured as the same way as the above procedure of water insoluble ash by replacing deionized water with 10% hydrochloric acid (sp. gr. 1.050) as solvent.

6. Alkalinity of Ash

The alkalinity of the ash is ascribed to the presence of the salts of such acids as citric, tartaric, or malic which are converted to the corresponding carbonates on incineration (11). Therefore, the alkalinity of the water-soluble ash from grape products is potassium carbonate derived from naturally occurring cream of tartar, whereas the alkalinity of the water-insoluble ash results from calcium and magnesium carbonates derived from calcium salts of fruit acids. It is known that

the ash of fruit and vegetable products is alkaline in reaction, while that of meat products and certain cereals is acid

The alkalinity of the ash can be used in determining the acid-base balance of the food, and in detecting adulteration of the food with minerals. The concentrations of the acid-forming elements (P, S, Cl) and the base-forming elements (K, Na, Ca, and Mg) directly influence the acid-base balance in a food material. The quantity of these elements in a given weight of food material is converted into milliliters of 1N acid or base, respectively, equivalent to the amount of acid or base element present. The general procedure and calculation for determination of alkalinity are as follows:

a. Procedure

- (1) Moisten an ashed sample with a small amount of deionized water, and quantitatively transfer the sample into a 400-ml beaker.
- (2) Add 50ml of 0.1N HCl to the beaker. Cover the sample beaker with a watch glass and boil gently for 5 min. Cool the beaker and rinse the watch glass with freshly boiled deionized water and drain rinsings back into the beaker.
- (3) Add 30 ml of 10% CaCl₂ solution, gently stir, cover with a watch glass and let it stand for 10 min.
- (4) Add 10 drops of 1% phenolphthalein indicator (1.0 g in 100 ml 95% ethyl alcohol), and titrate with 0.1N NaOH to a faint pink color persisting for 30 sec.

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b. Calculations:

$$\text{ml } 0.1\text{N HCl}/100 \text{ g} = 1000 \times 50 \times (\text{N of HCl}) - (\text{N of NaOH} \times \text{ml NaOH})$$

weight of sample