11 chapter

Ash Content Determination

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S.S. Nielsen, *Food Analysis Laboratory Manual*, Food Science Text Series,
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11.1 INTRODUCTION

11.1.1 Background

Ash refers to the inorganic residue remaining after either ignition or complete oxidation of organic matter in a food sample. The inorganic residue consists mainly of the minerals present in the food sample. Determining the ash content is part of the proximate analysis for nutritional evaluation. Also, ashing is the first step in the preparation of a sample for specific elemental analysis. Two major types of ashing procedures are commonly used, dry ashing and wet ashing. Dry ashing is heating food at elevated temperatures (500–600 °C) in a muffle furnace. Water and volatiles will evaporate, and organic matter will burn in the presence of oxygen and convert to CO_2 and oxides of N_2. In contrast, wet ashing is based on oxidizing organic matter using acids and oxidizing agents or their combination. Minerals are thus solubilized without oxidation. Food with high-moisture content, such as vegetables, is often dried prior to ashing. Food with high-fat content, such as meat, may need to be dried and their fat extracted prior to ashing. The ash content can be expressed on a wet basis or a dry basis.

11.1.2 Reading Assignment

Harris, G.K., and Marshall, M. R. 2017. Ash analysis. Ch. 16, in *Food Analysis*, 5th ed. S.S. Nielsen (Ed.), Springer, New York.

11.1.3 Objective

Determine the ash content of a variety of food products by the dry ashing technique and express on a wet weight basis and dry weight basis.

11.1.4 Principle of Method

Organic materials are incinerated at elevated temperatures (550 °C) in a muffle furnace, and inorganic matter (ash) remains. Ash content is measured by weight of inorganic matter remaining.

11.1.5 Chemical

	CAS No.	*Hazards*
Hydrochloric acid (HCl)	7647-01-0	Corrosive

11.1.6 Hazards, Cautions, and Waste Disposal

Concentrated hydrochloric acid is corrosive; avoid breathing vapors and contact with skin and clothes. The muffle furnace is extremely hot. Use gloves and tongs when handling crucibles. The crucibles have been dried and stored in desiccators prior to weighing. They will pick up moisture by sitting on the counter, so remove them from the desiccators only just before use. Open desiccators slowly to avoid damage and danger from broken glass.

11.1.7 Supplies

- Ashed crucibles (numbered) (prewashed with 0.2 *N* HCl, heated in a muffle furnace at 550 °C for 24 h, and stored in a desiccator prior to use)
- Variety of food products, e.g., Cheddar cheese, Parmesan cheese, pasteurized processed cheese, dry baby cereal (rice), whole wheat flour, all-purpose flour, quinoa, and dry breakfast cereal

11.1.8 Equipment

- Analytical balance
- Desiccator
- Electric muffle furnace

11.2 PROCEDURE

Note: Food products such as cheese will need to be dried before ashing (i.e., also determine the moisture content). For dry food products such as those listed above, drying is not needed before ashing. However, moisture content must be determined to calculate ash content on a dry weight basis. Follow standard procedures such as those described in the moisture determination experiment to obtain the moisture content of all samples to be ashed.

1. Remove ashed crucibles from the desiccator and record weight and number of crucible in the table.
2. Accurately weigh ca. 2 g of sample (note that cheese samples are pre-dried and placed in desiccators) into the crucible, and record weight on the spreadsheet. Prepare triplicate samples for each type of food product analyzed.
3. Place crucibles in muffle oven at 550 °C for 24 h.
4. Turn off the muffle furnace and allow it to cool (might take a few hours).
5. Remove crucibles from the muffle furnace and place into a desiccator to cool (note that this may need to be done by a teaching assistant). Return the following day to weigh the ashed sample and record weight of crucible plus ashed sample in the table.

11.3 DATA AND CALCULATIONS

$$\text{Weight of ash} = (\text{weight of crucible and ash}) - \text{weight of crucible}$$

$$\%\,\text{Ash} = (\text{weight of ash} / \text{original sample weight}) \times 100$$

Report the average ash %, standard deviation, and coefficient of variation for the food product analyzed. Also calculate average ash % on a dry basis, using the average % moisture value determined in the moisture analysis experiment.

Note: For cheese samples, the % ash obtained is on a dry weight basis (dwb), since samples to be ashed need to be pre-dried. Also, calculate % ash on a wet weight basis (wwb), using the moisture % obtained in the moisture analysis experiment.

Converting wet basis to dry basis:

$$\%\,\text{ash on dry basis} = \%\,\text{ash on wet basis} \times 100 / (100 - \%\,\text{moisture content})$$

Converting dry basis to wet basis:

$$\%\,\text{ash on wet basis} = \%\,\text{ash on dry basis} \times (100 - \%\,\text{moisture content}) / 100$$

Rep	*Crucible number*	*Crucible wt. (g)*	*Crucible + un-ashed sample (g)*	*Un-ashed sample (g)*	*Crucible + ashed sample (g)*	*Ash (g)*	*Moisture (%) (previously determined)*	*Ash (% wwb)*	*Ash (% dwb)*
Sample A									
1									
2									
3									
								$\bar{X}$ =	$\bar{X}$ =
								SD =	SD =
								CV =	CV =
Sample B									
1									
2									
3									
								$\bar{X}$ =	$\bar{X}$ =
								SD =	SD =
								CV =	CV =

11.4 QUESTIONS

1. For this laboratory we used a dry ashing technique, in what instance would you want to use a wet ashing technique?
2. What are the advantages and disadvantages to using a dry ashing technique?
3. What are the disadvantages of using a wet ashing technique?
4. Why was it necessary to pre-acid wash, pre-ash, and dry in a desiccator the crucibles prior to use?
5. Were your results comparable to USDA reported values? Explain any discrepancies.

RESOURCES

Harris GK, Marshall MR (2017) Ash analysis, Ch. 16. In: Nielsen SS (ed) Food Analysis, 5th edn. Springer, New York