



## **Chapter B**

# **Plant material preparation and determination of weight percent ash**

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# Plant material preparation and determination of weight percent ash

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## Principle

The physical preparation of plant material generally consists of washing, drying, milling, and dry ashing an aliquot, or subsplit of the sample (Peacock, 1992). Whenever ashing is required by an analytical technique, a calculation of weight percent ash is reported. The results are converted back to a dry weight basis for comparison with other analytical techniques. Some analytical techniques, such as hydride generation atomic absorption spectroscopy and instrumental neutron activation analysis, do not require plant ash. For others, such as inductively coupled argon plasma atomic emission spectroscopy, plant ashing is a prerequisite.

## Interferences

The interferences most commonly encountered are: (1) dust from the sampling site which may coat stems and leaves; (2) loss of volatile elements at ashing temperature; and (3) incomplete ashing of some material species at the prescribed temperature. Most contamination of samples by dust can be eliminated by washing them in deionized water. The ashing temperature of 500°C was chosen because it is the temperature at which most plant materials will lose their organic components. It is maintained for 13 hours to maximize loss of organic material. Volatile elements (i.e., Se, As, Hg, P) are determined in unashed subsplits of the sample. Material that does not ash completely at 500°C is allowed to remain in the furnace for a second attempt. If the sample is still not ashed completely (as observed by weight, texture, color, and appearance), a notation is made on the Request For Analysis form, the submitter contacted for advice, and/or material forwarded to Sample Control.

## Scope

The average time required for preparation and percent ash calculation is 5 days, based on a suite of 40 samples. The minimum measurement of ash content is 0.01 percent. If the sample has been washed or washing is not requested and the sample has been milled, ashing and percent ash calculation can be done in 2 days.

## Apparatus

Laboratory equipment consists of the following:

- Thomas/Wiley Mill Standard Model 4, with 2 mm screen
- Fisher Scientific Isotemp muffle furnace 750 series, model 126
- Christy-Norris pulverizer, 8"
- Laboratory drying ovens, 0-200°C, 8-10 cu-ft capacity
- Spex 8000 Mixer/Mill

- Box fans (4.5") mounted to ring stands
- Vitreosil evaporating dishes (fused silica, 3.75" id)
- Coors evaporating dishes (porcelain, 3.0" id)
- Glass or plastic beakers, 4-L capacity
- Rotary mixer holding at least 36-pint sample containers
- Mettler AC100 electronic balance
- Scientech 3300 electronic balance

A supply of 0.5-oz polycons (pillboxes), 5-mm solid borosilicate beads and waxed weighing paper is also needed.

## Reagents

Acetone, C<sub>3</sub>H<sub>6</sub>O, laboratory grade, 1 pint (useful as a cleaning aid)

## Safety Precautions

As with all machinery, care must be taken while grinding or ashing the sample materials. The Wiley Mill has stationary and rotating razor-edged knives that can easily remove fingertips. The Christy/Norris Mill becomes very hot after just a short period of operation. Both mills must be cleaned between samples. This is usually done with compressed air (40 psi) and a toothbrush. Before cleaning, the mill should be at rest. Occasionally, a sample will leave a resinous buildup on the spinning cutting head, door, or screen. It can often be removed with the careful use of a razor knife and acetone. Due to dust, noise in excess of 80 dB, and heat hazards, the worker should be fully protected with a lab coat or smock, leather work gloves, dust mask, safety glasses, and hearing protectors. Acetone must be handled with care to avoid fumes and possible fire hazards. All work must be done in a dust hood having a face velocity of at least 150 linear feet per minute. A muffle furnace also creates a potential shock and burn hazard. Avoid contact with heating elements and never load or unload a hot furnace (>100°C). Review the *CHP* and *MSDS* for further information.

## Procedure

### Washing

A plant sample received for preparation, with a request for washing, undergoes a washing process to eliminate contamination from adhering particles such as dust. There are two methods for this: (1) "beaker soak" hand washing in tap or deionized water, and the most common (2) "colander rinse" with tap or deionized water.

When the "beaker soak" method is used, water must be constantly changed since the sample is actually moved from one beaker to another over the course of a few minutes. The sample is rinsed in a colander, and the beakers are rinsed and refilled for the next sample. All samples rinsed manually are transferred to a colander for drying. Drying temperatures are held under 40°C unless specified otherwise. Material having a resinous coating on stems or leaves is dried without heat to minimize the possibility of its loss through liquification. Samples are dried to brittleness; usually 24–48 hours.

## Milling

Dry samples can be put directly into the grinder. It has been found that the Wiley Mill is best for young, woody growth up to a thickness of 5 mm. The Christy/Norris Mill is used for all thicker materials such as twigs, roots, and branches up to about 13 mm in diameter, mosses and lichens are ground in a blender. Larger diameter material must be cut to prevent jamming of the mill. This is most easily accomplished with pruning shears or a band saw.

Samples are mixed using a rotary type of tumbling device. The top 2.5 cm of the pint container must remain unfilled to insure proper mixing. The holders are set to accommodate four 8.9-cm diameter sample containers in line. These sample holders form the circumference of the mixer. Samples are held in place by flat plates secured with wing nuts to long threaded bolts mounted in the holder frames. The mixer is driven by a gear reduction motor having an output of 45 rpm (39:1 ratio). A toothed timing belt is linked to a 1/3 hp, single phase, ac 1,725 rpm drive motor. The average weight of a plant sample after grinding is 200 g.

## Dry Ashing

Ashing requires only a portion of the sample, but enough to satisfy the analytical need and be representative of the entire sample. The amount of this "aliquot" is also determined by its density, estimated ash yield, and amount of sample available. Sample material is taken off the top of its container with a plastic teaspoon and weighed. An aliquot of 10 g is optimum for a 3.75 inch Vitreosil dish, although satisfactory results have been obtained from splits of 1 to 24 g. Using a spatula, the sample is spread evenly along the sides and bottom of each dish to enhance even heat distribution throughout the aliquot. The ashing vessels are arranged in each furnace three across, left to right, upper shelf first (nine per shelf). Any consistent arrangement is appropriate provided it is known which dish holds what sample. There should be some space between each dish and the furnace walls. Breakage can occur from tightly packed arrangements. Shelving material is stainless-steel mesh wrapped over a similar framework forming a flat tray.

Ashing proceeds with the door fully closed with the fan on to bring into the furnace the required oxygen. The furnaces are programmed to "ramp" up to the ashing temperature of 500°C over a period of 5 hours. Complete ashing is insured by maintaining this temperature for 13 hours. The furnaces are allowed to cool for 8 hours before sample dishes are removed. While cooling, the door should be slightly open but not swung away until the inside temperature dips below 200°C. Sample dishes should remain undisturbed until cooled to 100°C. Ashing vessels are removed using tongs and placed on a metal or insulated surface for further cooling. At least 20 min should be allowed for this. After sample removal, cooling of the furnaces is enhanced by box fans positioned in front of the interior. Due to limitations in the controller and programmer circuitry, the brick temperature must be reduced to 24°C or less before the next ashing cycle can begin. In all, furnace cooling requires about 10 hours to complete.

The ash is weighed and transferred to 0.5-oz pillboxes using weighing paper as funnels. The ash must then be mixed and reduced in volume as it tends to be highly charged with static. This is done through the use of a 5-mm solid-borosilicate bead (placed into the polycon prior to addition of the ash), and 10 to 60 s of shaking in a Spex 8000 mixer/mill. The ash is then ready for laboratory analysis.

## Calculation

Weight percent ash is determined for all ashed samples. It requires the measurement of the empty vessel, the combined weight of vessel and sample aliquot before ashing, and the weight of the cooled vessel and ashed sample aliquot. All are weighed and recorded to a minimum of four decimal places (0.0001 g). The net weight of the aliquot and resulting ash must be determined by difference, multiplying the result by 100. Rounding gives the reported values in three significant figures. The weight of the vessel is subtracted from both figures and the formula for calculating percent ash is:

$$\% \text{ ash} = \frac{\text{ashed sample wt (g)}}{\text{unashed sample wt (g)}} \times 100$$

## Assignment of Uncertainty

Reference materials are included in each batch of samples for control check use by the analyst.

## Bibliography

Peacock, Thomas R., 1992, The preparation of plant material and determination of weight percent ash: U.S. Geological Survey Open-File Report 92-345, 9 p.