

## **LABORATORY DRY MATTER AND ASH PROCEDURE**

### **Up-dated September 2013**

There are many modifications of the procedure for the laboratory determination of dry matter and ash. However, for the sake of uniformity, we have developed the following methodology. The extreme detail is offered as a means of troubleshooting analytical problems and as a means of reducing the inherent variation presented by some samples. It must be remembered that the rigor of methodology is limited by the technique used in sampling and sample preparation.

### **PERCENT LAB DRY MATTER**

#### **I. Personal Protective Equipment:**

- A. Lab coat
- B. Safety glasses/goggles

#### **II. Procedure**

- A. Clean ceramic crucibles (or small aluminum weighing dishes if not doing ash), which have been dried for 24 hours are removed from the oven with wire tongs.
  - 1. The crucibles are placed in a desiccator freshly charged with a drying agent.
  - 2. The lid of the desiccator is positioned and left slightly ajar.
  
- B. After 2 minutes, the lid is moved to seal the chamber.
  - 1. Failure to allow for the cooling of the crucibles before positioning the lid will result in the formation of a vacuum in the desiccator.
  - 2. However, the **lid must not be left ajar** long enough to effect the weight of the crucibles.
  
- C. Once sealed, the crucibles should be allowed to cool for at least 5 minutes before weighing on the balance. As a rule of thumb, the crucibles should not be allowed to remain in the desiccator for more than 20 minutes. Before obtaining the tare weight of the crucibles, check the level and zero the balance.
  - 1. The balance is leveled with the adjustable legs and the center point bubble meter.
  - 2. To ensure that the atmosphere of the balance chamber will not affect the weight, the chamber should be charged with a container of fresh drying agent and allowed to equilibrate. Once the chamber is dried it is imperative that the doors of the balance remain closed at all times.
  - 3. When all is ready, the crucible may be transferred to the balance pan with tongs. The exact weight to the nearest 0.0001 of a gram is recorded.
- D. Zero the balance and without removing the crucible, add 1-gram sample. Record to the nearest 0.001 of a gram.
  - 1. The sample must be uniform and may be added to the crucible with a spatula. This process is repeated for a duplicate sample.
  
- E. The crucibles are returned to the oven set at 105 °C for 12-24 hours. It is helpful but not necessary that the oven be charged with a drying agent.
  
- F. At the end of the drying period, the crucibles are returned to the desiccator, cooled, and weighed in the same manner as previously described.

- G. The weight is recorded, and the dry sample is determined as the difference between the gross dry weight and crucible weight.
- H. The percent dry matter is expressed as the ratio of the dry sample weight to the wet sample weight times 100.
1. The duplicate samples are averaged and the two should not vary by more than 5.0% error calculated from the difference of the two samples. If the duplicates vary by more than 5.0%, the procedure should be repeated.

**Note:** If the sample is not to be used for an ash determination, the crucible may be replaced in this procedure with an aluminum dish and can discard the sample after weighing back. However, the continuous transferring of the aluminum dishes with tongs may require extreme caution and more time. It is necessary to use some mechanical means to move the aluminum dishes. As with the crucibles, manual contact may drastically affect the weights.

## II. Calculation

$$\frac{\text{Dry Sample Weight}}{\text{Wet Sample Weight}}$$

**Note:** To recharge the desiccant, ash at 200°C for two to three hours.

## PERCENT LAB ASH

### I. Principle

The crucible and dry sample remaining after the dry matter determination may be used for the ash determination. However, if dry matter has not been determined, an ash sample must be prepared following the same procedure outlined for dry matter preparation. Before proceeding, the crucible and dry sample weights must be known.

### II. Procedure

- A. The crucibles are transferred to a cool muffle furnace and the furnace is preset at 600 °C. Between the determination of the gross dry weight and the ashing process, the crucibles may be moved manually.
  1. The furnace is closed and turned on.
- B. Once the furnace has reached the preset temperature, allow the sample to ash for six hours. In some cases, the ashing time must be extended up to 12 hours.
- C. At the end of the ashing period, the furnace is turned off (on the small ash oven) and allowed to cool. **Note:** Large ash oven shuts off automatically. It takes about 20-22 hours for the oven to cool.
  1. Oven doors should not be opened until temperature is below 200 °C to avoid cracking and warping the firebrick oven linings.
- D. The samples are removed and placed in a desiccator with tongs, cooled and weighed following the procedure outlined above.
- E. This weight is the gross ashed weight and the sample ash is determined by the

difference between this value and the crucible wt.

1. The percent ash is expressed as the ratio of the sample ash weight to the dry sample weight times 100.

### III. Calculation

A.  $X 100 = \% DM$

B.  $\frac{\text{Ash Sample Weight}}{\text{Dry Sample Weight}}$

100- % ash = % organic matter

### COMMENTS

Care must be taken in the operation of the furnaces. The furnaces area is supplied with special long tongs and asbestos gloves. Please help us keep this equipment and area in order. Please notify the appropriate people of malfunctions and breakage.

Use the drying ovens for "samples only". Placing wet lab ware in these ovens will cause errors in dry matter determination in process. The oven control dial is not a temperature selector. It is a linear scale, which must be calibrated. Therefore, a thermometer must be in position on the upper vent to monitor the internal temperature. **No material** should be placed on the metal floor of the oven, as the heating elements and oven performance may be affected.

$X 100 = \% Ash$