

Total Ash

Purpose: To determine, as ash, the residue remaining after ignition.

A. Apparatus:

1. Flat-bottomed dish or crucible, preferably platinum or silica with minimum capacity of 50 mL.
2. Electric muffle furnace provided with pyrometer for indicating temperature and automatic thermoregulator to maintain $600 \pm 20^{\circ}\text{C}$ or equivalent microwave muffle furnace.
3. Desiccator containing desiccant such as anhydrous calcium sulphate or silica gel with indicator.
4. Fume hood or equivalent venting system.
5. Analytical balance, sensitivity 0.0001g (0.1 mg).

B. Reagents:

1. Distilled water.
2. Ashless filter paper (Whatman 42).
3. Nitric acid (HNO_3), ACS grade.

C. Preparation of sample:

Prepare samples as described in Method 1.0.

D. Procedure:

1. Ignite the flat-bottomed dish or crucible to a dull redness, cool to room temperature in a desiccator and weigh to the nearest 0.0001g (0.1 mg).
2. Weigh accurately 2 to 3 g of well mixed sample in the dish or crucible to the nearest 0.1 mg. (Note 1)

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3. Place the dish or crucible in the entrance of the open muffle furnace until the sample is well carbonized. The sample must not catch fire. (The sample may be placed under an infrared lamp until carbonized.) (Note 2)
4. Place the carbonized sample in the furnace at $600^{\circ} \pm 20^{\circ}\text{C}$. Incinerate for two hours, until light gray ash is obtained or to constant weight.
5. If carbon remains, leach ash with hot water, filter through ashless filter paper, wash paper thoroughly, and transfer paper and contents to the original crucible. Dry and ignite in the muffle furnace at $600^{\circ} \pm 20^{\circ}\text{C}$ until the ash is white. Cool dish with filtrate to dryness on steam bath and heat in muffle furnace for 30 minutes.
6. When carbon-free ash is obtained in steps D4 or 5 above, place the crucible in a desiccator, cool and weigh to nearest 0.0001g (0.1 mg).

E. Calculation:

$$\text{Ash \%} = \frac{\text{Wt. of ash (g)}}{\text{Wt. of sample (g)}} \times 100$$

F. Statistics:

TBD

G. Notes:

1. Ground mustard or mustard flour. Follow the above procedure through step D4. Leach the ash with hot water, filter, and wash thoroughly with hot distilled water. Transfer the paper and contents to the original crucible, dry, and heat in the muffle furnace for 30 minutes. Remove the crucible, allow to cool, add 5-10 drops of HNO_3 , evaporate to dryness, and heat in the furnace for 30 minutes. Repeat the nitric acid and heating treatment until the residue is white. Proceed as in step D5 beginning with "Cool dish with filtrate..."
2. Alternate procedure for step D3: Heat the crucible over a Bunsen burner, in a hood, until the sample is well-charred, taking care that it does not ignite.

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H. Reference:

AOAC Official Methods of Analysis (1995) 43.1.05; (941.12).
JAOAC 24, 667 (1941).