

## B. GENERAL TESTS

### Ash and Acid-Insoluble Ash Limit Tests

#### 1. Ash

The Ash Limit Tests are designed to measure the amount of the residual substances when a sample is ignited under the conditions specified in the individual monograph.

**Procedure** Ignite a crucible of platinum, quartz, or porcelain at 500 to 550 for 1 hour, allow to cool in a desiccator, and weigh it accurately. Unless otherwise specified, place 2 to 4 g of the sample in the crucible and weigh it accurately.

Take off or slide the lid of the crucible if necessary, heat the crucible gently first, then raise the temperature gradually, ignite at 500 to 550 for not less than 4 hours to incinerate until it is free from charred matter, cool in a desiccator, and weigh accurately. Incinerate the residue to constant weight, cool in a desiccator, and weigh accurately.

If charred matter still remains and a constant weight cannot be obtained with the above procedure, add hot water to the matter, filter it through a filter paper for quantitative analysis, and ignite the insoluble residue on the filter paper together with the filter paper at 500 to 550 until it is free from charred matter. Add the filtrate to the residue, evaporate to dryness, and ignite at 500 to 550, cool in a desiccator, and weigh accurately. If charred matter still remains, add small amount of ethanol, break up the ash with a glass rod, wash the glass rod with small amount of ethanol, and evaporate carefully the ethanol. Then, proceed as directed previously and weigh accurately.

#### 2. Acid-insoluble ash

The Acid-Insoluble Ash Limit Test is designed to measure the amount of ash insoluble to diluted hydrochloric acid.

**Procedure** Add carefully 25 ml of dilute hydrochloric acid to the ash (obtained as directed under the Ash Limit Test), boil gently for 5 minutes, collect the insoluble matter on a filter paper for quantitative analysis, wash thoroughly with hot water, and dry the residue together with the filter paper. Ignite it for 3 hours in a crucible of platinum, quartz, or porcelain, which has been prepared as directed in the Ash Limit Test and whose weight is already known. Cool it in a desiccator (silica gel) and weigh accurately. If the measured amount is larger than the specified value, ignite until a constant weight is obtained.