

**<281> RESIDUE ON IGNITION****Change to read:**

\* The *Residue on Ignition / Sulfated Ash* test utilizes a procedure to measure the amount of residual substance not volatilized from a sample when the sample is ignited in the presence of sulfuric acid according to the procedure described below. This test is usually used for determining the content of inorganic impurities in an organic substance. •<sub>2</sub>

**Change to read:**

\* **Procedure** — •<sub>2</sub> Weigh accurately 1 to 2 g of the substance, or the amount specified in the individual monograph, in a suitable crucible \* (silica, platinum, quartz, or porcelain) •<sub>2</sub> that previously has been ignited \* at  $600 \pm 50^{\circ}$  for 30 minutes, •<sub>2</sub> cooled \* in a desiccator (silica gel or other suitable desiccant), •<sub>2</sub> and weighed. \* Moisten the sample with a small amount (usually 1 mL) of sulfuric acid. •<sub>2</sub> Heat, gently at first, \* at a temperature as low as practicable •<sub>2</sub> until the substance is thoroughly charred, cool, then, unless otherwise directed in the individual monograph, moisten the residue with \* a small amount (usually 1 mL) •<sub>2</sub> of sulfuric acid, heat gently until white fumes are no longer evolved, and ignite at \*  $600 \pm 50^{\circ}$ , •<sub>2</sub> unless another temperature is specified in the individual monograph, until the carbon is consumed. \* Ensure that flames are not produced at any time during the procedure. •<sub>2</sub> Cool in a desiccator \* (silica gel or other suitable desiccant), •<sub>2</sub> weigh, and calculate the percentage of residue. \* Unless otherwise specified, if •<sub>2</sub> the amount of the residue so obtained exceeds the limit specified in the individual monograph, \* repeat the moistening with sulfuric acid, heating and ignition as before, •<sub>2</sub> until constant weight is attained or until the percentage of residue complies with the limit in the individual monograph.

Conduct the ignition in a well-ventilated hood, but protected from air currents, and at as low a temperature as is possible to effect the complete combustion of the carbon. A muffle furnace may be used, if desired, and its use is recommended for the final ignition at \*  $600 \pm 50^{\circ}$ . •<sub>2</sub>

Calibration of the muffle furnace may be carried out using an appropriate digital temperature meter and a working thermocouple probe calibrated against a standard thermocouple traceable to the National Institute of Standards and Technology.

Verify the accuracy of the measuring and controlling circuitry of the muffle furnace by checking the positions in the furnace at the control set point temperature of intended use. Select positions that reflect the eventual method of use with respect to location of the specimen under test. The tolerance is  $\pm 25^{\circ}$  at each position measured.

Sulphated Ash tests found in the European \* and Japanese •<sub>2</sub> Pharmacopoeias are considered equivalent to this test, except where noted.