2.3 Sulfated ash

The test Method B is based on the internationally-harmonized texts developed by the Pharmacopoeial Discussion Group (PDG). Some editorial modifications have been made in order to be in line with the style used in The International Pharmacopoeia.

The sulfated ash test uses 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. The test is usually used for determining the content of inorganic impurities in an organic substance. Unless otherwise indicated in the individual monograph, use Method A.

Method A

Accurately weigh about 1 g of the substance, or the quantity specified in the monograph, into a suitable crucible (usually platinum) and moisten with sulfuric acid (~1760 g/l) TS. Heat gently to remove the excess of acid and ignite at about 800 °C until all the black particles have disappeared; again moisten with sulfuric acid (~1760 g/l) TS and reignite. Add a small amount of ammonium carbonate R and ignite to constant weight.

Method B

Ignite a suitable crucible (for example silica, platinum, quartz or porcelain) at 550 °C to 650 °C for 30 minutes, cool the crucible in a desiccator (silica gel or other suitable desiccant) and weigh it accurately. Take the amount of test sample specified in the individual monograph in the crucible and weigh the crucible accurately. Moisten the sample with a small amount (usually 1 mL) of sulfuric acid (~1760 g/l) TS, heat gently at a temperature as low as practicable until the sample is thoroughly charred. After cooling moisten the residue with a small amount (usually 1 mL) of sulfuric acid (~1760 g/l) TS, heat gently until white fumes are no longer evolved and ignite at 550 °C to 650 °C until the residue is completely incinerated. Ensure that flames are not produced at any time during the procedure. Cool the crucible in a desiccator (silica gel or other suitable desiccant), weigh accurately and calculate the percentage of residue.

Unless otherwise specified, if the amount of residue so obtained exceeds the limit specified in the individual monograph, repeat the moistening with sulfuric acid, heating and ignition as before, using a 30-minute ignition period, until two consecutive weighings of the residue do not differ by more than 0.5 mg or until the percentage of residue complies with the limit in the individual monograph.