Barium sulfate (Barii sulfas)

Molecular formula. $BaSO_4$

Relative molecular mass. 233.4

Chemical name. Barium sulfate (1:1); CAS Reg. No. 7727-43-7.

Description. A white, heavy, fine powder; free from grittiness; odourless.

Solubility. Practically insoluble in water and in organic solvents; very slightly soluble in acids and in solutions of alkali hydroxides.

Category. Radiocontrast medium.

Storage. Barium sulfate should be kept in a well-closed container.

Additional information. Barium sulfate is inclined to caking.

Requirements

Identity tests

A. Boil 0.2 g in a solution of 5.0 g of sodium carbonate R dissolved in 5 mL of water for 5 minutes, then add 10 mL of water and filter (keep the precipitate for test B). To 5 mL of the filtrate add 5 mL of hydrochloric acid (~70 g/l) TS; this solution yields reaction A described under 2.1 General identification tests as characteristic of sulfates.

B. Wash the precipitate from test A with 3 successive small quantities of water. To the residue add 5 mL of hydrochloric acid (~70 g/l) TS, filter, and to the filtrate add 0.3 mL of sulfuric acid (~100 g/l) TS; a white precipitate is produced which is insoluble in sodium hydroxide (~80 g/l) TS.

Sedimentation. Place 5.0 g, previously sifted, in a glass-stoppered 50-mL graduated cylinder, having the 50-mL graduation mark 11 - 14 cm from the base. Add sufficient water to produce 50 mL, shake for 5 minutes and allow to stand for 15 minutes; the barium sulfate does not settle below the 15-mL graduation mark.

Heavy metals. For the preparation of the test solution boil 4 g with 6 mL of acetic acid (~60 g/l) PbTS and 44 mL of water for 10 minutes, filter, allow to cool and dilute to 50 mL with water. Determine the heavy metals content in 25 mL of the filtrate as described under 2.2.3 Limit test for heavy metals, according to Method A; not more than 10 µg/g.

Arsenic. Transfer 0.5 g to a long-necked combustion flask, add 30 mL of water and 2 mL of nitric acid (~1000 g/l) TS, insert a small funnel into the neck of the flasks and heat in an inclined position on a water-bath for 2 hours. Allow to cool, adjust to the original volume with water, and filter. Wash the residue three times with 5 mL of water, combine the filtrate and washings, add 1 mL of sulfuric acid (~1760 g/l) TS, and evaporate on a water-bath until white fumes are evolved. Dissolve the residue in 10 mL of sulfuric acid (~100 g/l) TS, add 10 mL of water, and proceed as described under 2.2.5 Limit test for arsenic; the arsenic content is not more than 2 µg/g.

Soluble barium salts. Boil 10 g with 20 mL of water and 30 mL of acetic acid (~60 g/l) TS for 5 minutes, filter, allow to cool and dilute to 50 mL with water. To a 10-mL portion of this solution add 1 mL of sulfuric acid (~100 g/l) TS and to a second 10-mL portion add 1 mL of water. When compared after 1 hour, the two solutions remain equally clear.

Phosphates. To 1.0 g add 3 mL of nitric acid (~130 g/l) TS and 7 mL of water and heat on a water-bath for 5 minutes. Filter and dilute the filtrate to 10 mL with water. Add 5 mL of ammonium molybdate/vanadate TS and allow to stand for 5 minutes; any yellow colour produced is not more intense than that of a reference solution prepared similarly using 10 mL of phosphate standard (5 µg/mL) TS.

Oxidizable sulfur compounds. Shake 1.0 g with 5 mL of water for 30 seconds and filter. To the filtrate add 0.1 mL of starch TS, 0.1 g of potassium iodide R, 1 mL of freshly prepared potassium iodate (3.6 mg/l) TS, and 1 mL of hydrochloric acid (1 mol/l) VS, and shake well; the colour produced is more intense than that of a solution prepared in a similar way, but omitting the potassium iodate.

Acid-soluble substances. Boil 5 g with 15 mL of acetic acid (~300 g/l) TS and 10 mL of water for 5 minutes. Filter, evaporate the filtrate to dryness on a water-bath, and dry to constant weight at 105°C; the residue weighs not more than 15 mg.

Loss on ignition. Ignite 1.0 g at 600 °C; it loses not more than 20 mg/g.

Acidity or alkalinity. Heat 5.0 g with 20 mL of carbon-dioxide-free water R on a water-bath for 5 minutes and filter. To 10 mL of the filtrate add 0.05 mL of bromothymol blue/ethanol TS; not more than 0.5 mL of hydrochloric acid (0.01 mol/l) VS or 0.5 mL of carbonate-free sodium hydroxide (0.01 mol/l) VS is required to obtain the midpoint of the indicator (green).