Tests

**Solution S.** Dissolve 2.5 g in 20 ml of dilute hydrochloric acid R, filter if necessary and add dilute ammonia R1 until a precipitate is formed. Add just sufficient dilute hydrochloric acid R to dissolve the precipitate and dilute to 50 ml with distilled water R.

**Carbonates.** Shake 0.5 g with 5 ml of carbon dioxide-free water R and add 1 ml of hydrochloric acid R. No effervescence is produced.

**Chlorides (2.4.4):** maximum 330 ppm.

Dissolve 0.5 g in a mixture of 1 ml of nitric acid R and 10 ml of water R and dilute to 50 ml with water R. 15 ml of the solution complies with the limit test for chlorides.

**Fluorides (2.4.5):** maximum 100 ppm.

0.5 g complies with the limit test for fluorides.

**Sulphates (2.4.13):** maximum 0.5 per cent.

To 1 ml of solution S add 2 ml of dilute hydrochloric acid R and dilute to 25 ml with distilled water R. 15 ml of the solution complies with the limit test for sulphates.

**Arsenic (2.4.2):** maximum 10 ppm.

2 ml of solution S complies with limit test A.

**Barium.** To 10 ml of solution S add 0.5 ml of dilute sulphuric acid R. After 15 min, any opalescence in the solution is not more intense than that in a mixture of 10 ml of solution S and 0.5 ml of distilled water R.

**Iron (2.4.9):** maximum 400 ppm.

0.5 ml of solution S diluted to 10 ml with water R complies with the limit test for iron.

**Heavy metals (2.4.8):** maximum 40 ppm.

Dilute 10 ml of solution S to 20 ml with water R. 12 ml of the solution complies with limit test A. Prepare the standard using lead standard solution (1 ppm Pb) R.

**ASSAY**

Dissolve 0.30 g in a mixture of 1 ml of hydrochloric acid R1 and 5 ml of water R. Add 25.0 ml of 0.1 M sodium edetate and dilute to 200 ml with water R. Neutralise with concentrated ammonia R, add 10 ml of ammonium chloride buffer solution pH 10.0 R and about 50 mg of mordant black 11 tritrate R. Titrate the excess of sodium edetate with 0.1 M zinc sulphate until the colour changes from blue to violet. Carry out a blank titration.

1 ml of 0.1 M sodium edetate is equivalent to 17.21 mg of CaHPO₄·2H₂O.

CHARACTERS

A white or almost white, fine powder, practically insoluble in water.

Identification

A. To 0.80 g in a mortar, add 10 ml of water R and 0.5 ml of phenolphthalein solution R and mix. The suspension turns red. On addition of 17.5 ml of 1 M hydrochloric acid, the suspension becomes colourless without effervescing. The red colour occurs again when the mixture is triturated for 1 min. On addition of a further 6 ml of 1 M hydrochloric acid and triturating, the solution becomes colourless.

B. Dissolve about 0.1 g in dilute hydrochloric acid R and dilute to 10 ml with water R. 5 ml of the solution give reaction (b) of calcium (2.3.1).

Tests

**Matter insoluble in hydrochloric acid.** Dissolve 2.0 g in 30 ml of hydrochloric acid R. Boil the solution and filter. Wash the residue with hot water R. The residue weighs not more than 10 mg (0.5 per cent).

**Carbonates.** Not more than 5.0 per cent of CaCO₃.

Add 5.0 ml of 1 M hydrochloric acid to the titrated solution obtained under Assay and titrate with 1 M sodium hydroxide using 0.5 ml of methyl orange solution R as indicator.

1 ml of 1 M hydrochloric acid is equivalent to 50.05 mg of CaCO₃.

**Chlorides (2.4.4).** Dissolve 0.30 g in a mixture of 2 ml of nitric acid R and 10 ml of water R and dilute to 30 ml with water R. 15 ml of the solution complies with the limit test for chlorides (330 ppm).

**Sulphates (2.4.13).** Dissolve 0.15 g in a mixture of 5 ml of dilute hydrochloric acid R and 10 ml of distilled water R and dilute to 60 ml with distilled water R. 15 ml of the solution complies with the limit test for sulphates (0.4 per cent).

**Arsenic (2.4.2).** Dissolve 0.50 g in 5 ml of brominated hydrochloric acid R and dilute to 50 ml with water R. 25 ml of the solution complies with the limit test A for arsenic (4 ppm).

**Magnesium and alkali metals.** Dissolve 1.0 g in a mixture of 10 ml of hydrochloric acid R and 40 ml of water R. Boil and add 50 ml of a 63 g/l solution of oxalic acid R. Neutralise with ammonia R and dilute to 200 ml with water R. Allow to stand for 1 h and filter through a suitable filter. To 100 ml of the filtrate, add 0.5 ml of sulphuric acid R. Cautiously evaporate to dryness and ignite. The residue weighs not more than 20 mg (4.0 per cent calculated as sulphates).

**Heavy metals (2.4.8).** Dissolve 1.0 g in 10 ml of hydrochloric acid R1 and evaporate to dryness on a water-bath. Dissolve the residue in 20 ml of water R and filter. 12 ml of the filtrate complies with limit test A for heavy metals (20 ppm). Prepare the standard using lead standard solution (1 ppm Pb) R.

**ASSAY**

To 1.500 g in a mortar, add 20 ml to 30 ml of water R and 0.5 ml of phenolphthalein solution R. Titrate with 1 M hydrochloric acid by triturating the substance until the red colour disappears. The final solution is used in the tests for carbonates.

1 ml of 1 M hydrochloric acid is equivalent to 37.05 mg of Ca(OH)₂.