

Arsenic (2.4.2, *Method A*): maximum 3 ppm.

Dissolve 0.33 g in *water R* and dilute to 25 ml with the same solvent.

Iron (2.4.9): maximum 50 ppm, determined on 0.20 g.

Heavy metals (2.4.8): maximum 20 ppm.

Dissolve 2.0 g in 10 ml of *buffer solution pH 3.5 R* and dilute to 20 ml with *water R*. 12 ml of the solution complies with test A. Prepare the reference solution using *lead standard solution (2 ppm Pb) R*.

Loss on drying (2.2.32): maximum 12.0 per cent, determined on 1.000 g by drying in an oven at 150 °C for 4 h.

ASSAY

Dissolve 0.200 g in *water R*. Carry out the complexometric titration of calcium (2.5.11).

1 ml of 0.1 M *sodium edetate* is equivalent to 4.008 mg of Ca.

01/2008:0981
corrected 6.0

CALCIUM HYDROGEN PHOSPHATE, ANHYDROUS

Calcii hydrogenophosphas anhydricus

CaHPO₄
[7757-93-9]

*M*_r 136.1

DEFINITION

Content: 98.0 per cent to 103.0 per cent.

CHARACTERS

Appearance: white or almost white, crystalline powder, or colourless crystals.

Solubility: practically insoluble in water and in ethanol (96 per cent). It dissolves in dilute hydrochloric acid and in dilute nitric acid.

IDENTIFICATION

- Dissolve with heating 0.1 g in 10 ml of *dilute hydrochloric acid R*. Add 2.5 ml of *dilute ammonia R1*, shake, and add 5 ml of a 35 g/l solution of *ammonium oxalate R*. A white precipitate is produced.
- Dissolve 0.1 g in 5 ml of *dilute nitric acid R*, add 2 ml of *ammonium molybdate solution R* and heat at 70 °C for 2 min. A yellow precipitate is produced.
- It complies with the limits of the assay.

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*.

Acid-insoluble substances: maximum 0.2 per cent.

Dissolve 5.0 g in 40 ml of *water R*, add 10 ml of *hydrochloric acid R* and heat to boiling for 5 min. Cool, then collect the insoluble substances using ashless filter paper. Wash with *water R* until turbidity is no longer produced when *silver nitrate solution R2* is added. Ignite at 600 ± 50 °C. The residue weighs not more than 10 mg.

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: maximum 0.25 per cent.

Test solution. Dissolve 0.20 g in a mixture of 20 ml of *water R* and 13 ml of *dilute nitric acid R*, dilute to 100 ml with *water R* and filter if necessary. Use 50 ml of this solution.

Reference solution. To 0.70 ml of 0.01 M *hydrochloric acid*, add 6 ml of *dilute nitric acid R* and dilute to 50 ml with *water R*.

Add 1 ml of *silver nitrate solution R2* to the test solution and to the reference solution and mix. After standing for 5 min protected from light, any opalescence in the test solution is not more intense than that in the reference solution.

Fluorides: maximum 100 ppm.

Potentiometry (2.2.36, *Method II*).

Chelating solution. Dissolve 45 g of *cyclohexylenedinitrilo-tetra-acetic acid R* in 75 ml of *sodium hydroxide solution R* and dilute to 250 ml with *water R*.

Test solution. Dissolve 1.000 g in 4 ml of *hydrochloric acid R1*, add 20 ml of chelating solution, 2.7 ml of *glacial acetic acid R* and 2.8 g of *sodium chloride R*, adjust to pH 5-6 with *sodium hydroxide solution R* and dilute to 50.0 ml with *water R*.

Reference solution. Dissolve 4.42 g of *sodium fluoride R*, previously dried at 300 °C for 12 h, in *water R* and dilute to 1000.0 ml with the same solvent. Dilute 50.0 ml of this solution to 500.0 ml with *total-ionic-strength-adjustment buffer R* (200 ppm F).

Indicator electrode: fluoride-selective.

Reference electrode: silver-silver chloride.

Carry out the measurement on 20.0 ml of the test solution. Add at least 3 times 0.10 ml of the reference solution and carry out the measurement after each addition. Calculate the concentration of fluorides using the calibration curve.

Sulphates: maximum 0.5 per cent.

Test solution. Dissolve 0.5 g in a mixture of 5 ml of *water R* and 5 ml of *dilute hydrochloric acid R* and dilute to 100 ml with *water R*. Filter if necessary. To 20 ml of this solution, add 1 ml of *dilute hydrochloric acid R* and dilute to 50 ml with *water R*.

Reference solution. To 1.0 ml of 0.005 M *sulphuric acid*, add 1 ml of *dilute hydrochloric acid R* and dilute to 50 ml with *water R*. Filter if necessary.

To the test solution and to the reference solution, add 2 ml of a 120 g/l solution of *barium chloride R* and allow to stand for 10 min. Any opalescence in the test solution is not more intense than that in the reference solution.

Arsenic (2.4.2, *Method A*): maximum 10 ppm, determined on 2 ml of solution S.

Barium. To 0.5 g, add 10 ml of *water R* and heat to boiling. While stirring, add 1 ml of *hydrochloric acid R* dropwise. Allow to cool and filter if necessary. Add 2 ml of a 10 g/l solution of *dipotassium sulphate R* and allow to stand for 10 min. No turbidity is produced.

Iron (2.4.9): maximum 400 ppm.

Dilute 0.5 ml of solution S to 10 ml with *water R*.

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 test A. Prepare the reference solution using *lead standard solution (1 ppm Pb) R*.

Loss on ignition: 6.6 per cent to 8.5 per cent, determined on 1.000 g to constant mass at 800-825 °C.

ASSAY

Dissolve 0.4 g in 12 ml of *dilute hydrochloric acid R* and dilute to 200 ml with *water R*. To 20.0 ml of this solution add 25.0 ml of 0.02 M sodium edetate, 50 ml of *water R*, 5 ml of *ammonium chloride buffer solution pH 10.7 R* and about 25 mg of *mordant black 11 triturate R*. Titrate the excess of sodium edetate with 0.02 M *zinc sulphate*. Carry out a blank titration.

1 ml of 0.02 M sodium edetate is equivalent to 2.72 mg of CaHPO_4 .

01/2008:0116
corrected 6.0

CALCIUM HYDROGEN PHOSPHATE DIHYDRATE

Calcii hydrogenophosphas dihydricus

$\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$
[7789-77-7]

M_r 172.1

DEFINITION

Content: 98.0 per cent to 105.0 per cent.

CHARACTERS

Appearance: white or almost white, crystalline powder.

Solubility: practically insoluble in cold water and in ethanol (96 per cent). It dissolves in dilute hydrochloric acid and in dilute nitric acid.

IDENTIFICATION

- Dissolve with heating 0.1 g in 10 ml of *dilute hydrochloric acid R*. Add 2.5 ml of *dilute ammonia R1*, shake, and add 5 ml of a 35 g/l solution of *ammonium oxalate R*. A white precipitate is produced.
- Dissolve 0.1 g in 5 ml of *dilute nitric acid R*, add 2 ml of *ammonium molybdate solution R* and heat at 70 °C for 2 min. A yellow precipitate is produced.
- It complies with the limits of the assay.

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*.

Acid-insoluble substances: maximum 0.2 per cent.

Dissolve 5.0 g in 40 ml of *water R*, add 10 ml of *hydrochloric acid R* and heat to boiling for 5 min. Cool, then collect the insoluble substances using ashless filter paper. Wash with *water R* until turbidity is no longer produced when *silver nitrate solution R2* is added to the filtrate. Ignite at 600 ± 50 °C. The residue weighs not more than 10 mg.

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: maximum 0.25 per cent.

Test solution. Dissolve 0.20 g in a mixture of 20 ml of *water R* and 13 ml of *dilute nitric acid R*, dilute to 100 ml with *water R* and filter if necessary. Use 50 ml of this solution.

Reference solution. To 0.70 ml of 0.01 M *hydrochloric acid*, add 6 ml of *dilute nitric acid R* and dilute to 50 ml with *water R*.

Add 1 ml of *silver nitrate solution R2* to the test solution and to the reference solution and mix. After standing for 5 min protected from light, any opalescence in the test solution is not more intense than that in the reference solution.

Fluorides: maximum 100 ppm.

Potentiometry (2.2.36, *Method II*).

Chelating solution. Dissolve 45 g of *cyclohexylenedinitrilotetra-acetic acid R* in 75 ml of *sodium hydroxide solution R* and dilute to 250 ml with *water R*.

Test solution. Dissolve 1.000 g in 4 ml of *hydrochloric acid R1*, add 20 ml of chelating solution, 2.7 ml of *glacial acetic acid R* and 2.8 g of *sodium chloride R*, adjust to pH 5-6 with *sodium hydroxide solution R* and dilute to 50.0 ml with *water R*.

Reference solution. Dissolve 4.42 g of *sodium fluoride R*, previously dried at 300 °C for 12 h, in *water R* and dilute to 1000.0 ml with the same solvent. Dilute 50.0 ml of this solution to 500.0 ml with *total-ionic-strength-adjustment buffer R* (200 ppm F).

Indicator electrode: fluoride-selective.

Reference electrode: silver-silver chloride.

Carry out the measurement on 20.0 ml of the test solution. Add at least 3 times 0.10 ml of the reference solution and carry out the measurement after each addition. Calculate the concentration of fluorides using the calibration curve.

Sulphates: maximum 0.5 per cent.

Test solution. Dissolve 0.5 g in a mixture of 5 ml of *water R* and 5 ml of *dilute hydrochloric acid R* and dilute to 100 ml with *water R*. Filter if necessary. To 20 ml of this solution, add 1 ml of *dilute hydrochloric acid R* and dilute to 50 ml with *water R*.

Reference solution. To 1.0 ml of 0.005 M *sulphuric acid*, add 1 ml of *dilute hydrochloric acid R* and dilute to 50 ml with *water R*. Filter if necessary.

To the test solution and to the reference solution, add 2 ml of a 120 g/l solution of *barium chloride R* and allow to stand for 10 min. Any opalescence in the test solution is not more intense than that in the reference solution.

Arsenic (2.4.2, *Method A*): maximum 10 ppm, determined on 2 ml of solution S.

Barium. To 0.5 g, add 10 ml of *water R* and heat to boiling. While stirring, add 1 ml of *hydrochloric acid R* dropwise. Allow to cool and filter if necessary. Add 2 ml of a 10 g/l solution of *dipotassium sulphate R* and allow to stand for 10 min. No turbidity is produced.

Iron (2.4.9): maximum 400 ppm.

Dilute 0.5 ml of solution S to 10 ml with *water R*.

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 test A. Prepare the reference solution using *lead standard solution (1 ppm Pb) R*.

Loss on ignition: 24.5 per cent to 26.5 per cent, determined on 1.000 g by ignition to constant mass at 800-825 °C.

ASSAY

Dissolve 0.4 g in 12 ml of *dilute hydrochloric acid R* and dilute to 200 ml with *water R*. To 20.0 ml of this solution add 25.0 ml of 0.02 M sodium edetate, 50 ml of *water R*, 5 ml of *ammonium chloride buffer solution pH 10.7 R* and