



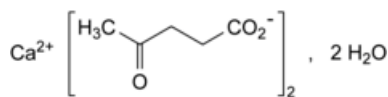
Edition: BP 2025 (Ph. Eur. 11.6 update)

Calcium Levulinate Dihydrate



General Notices

(Ph. Eur. monograph 1296)



C₁₀H₁₄CaO₆·2H₂O 306.3 5743-49-7

Action and use

Source of calcium.

Ph Eur

DEFINITION

Calcium di(4-oxopentanoate) dihydrate.

Content

98.0 per cent to 101.0 per cent (dried substance).

CHARACTERS

Appearance

White or almost white, crystalline powder.

Solubility

Freely soluble in water, very slightly soluble in ethanol (96 per cent), practically insoluble in methylene chloride.

IDENTIFICATION

First identification: A, D, E.

Second identification: B, C, D, E.

A. Infrared absorption spectrophotometry (2.2.24).

Comparison [calcium levulinate dihydrate CRS](#).

B. Thin-layer chromatography (2.2.27).

Test solution Dissolve 60 mg of the substance to be examined in [water R](#) and dilute to 1 mL with the same solvent.

Reference solution Dissolve 60 mg of [calcium levulinate dihydrate CRS](#) in [water R](#) and dilute to 1 mL with the same solvent.

Plate [TLC silica gel plate R](#).

Mobile phase [concentrated ammonia R](#), [ethyl acetate R](#), [water R](#), [ethanol \(96 per cent\) R](#) (10:10:30:50 V/V/V/V).

Application 10 µL.

Development Over a path of 10 cm.

Drying At 100-105 °C for 20 min and allow to cool.

Detection Spray with a 30 g/L solution of [potassium permanganate R](#). Dry in a current of warm air for about 5 min or until the spots become yellow. Examine in daylight.

Results The principal spot in the chromatogram obtained with the test solution is similar in position, colour and size to the principal spot in the chromatogram obtained with the reference solution.

C. To 1 mL of solution S (see Tests), add 20 mL of a 2.5 g/L solution of [dinitrophenylhydrazine R](#) in [dilute hydrochloric acid R](#). Allow to stand for 15 min. Filter, wash the precipitate with [water R](#). Dry the precipitate in an oven at 100-105 °C. The melting point (2.2.14) is 203 °C to 210 °C.

D. It gives reaction (b) of calcium (2.3.1).

E. Loss on drying (see Tests).

TESTS

Solution S

Dissolve 10.0 g in [carbon dioxide-free water R](#) prepared from [distilled water R](#) and dilute to 100.0 mL with the same solvent.

Appearance of solution

Solution S is clear (2.2.1) and not more intensely coloured than reference solution Y₆ (2.2.2, Method II).

pH (2.2.3)

6.8 to 7.8 for solution S.

Oxidisable substances

To 1 mL of solution S, add 10 mL of [water R](#), 1 mL of [dilute sulfuric acid R](#) and 0.25 mL of a 3.0 g/L solution of [potassium permanganate R](#). Mix. After 5 min, the violet colour of the mixture is still visible.

Sucrose and reducing sugars

To 5 mL of solution S add 2 mL of [hydrochloric acid R1](#) and dilute to 10 mL with [water R](#). Heat to boiling for 5 min and allow to cool. Add 10 mL of [sodium carbonate solution R](#). Allow to stand for 5 min, dilute to 25 mL with [water R](#) and filter. To 5 mL of the filtrate add 2 mL of [cupri-tartaric solution R](#) and heat to boiling for 1 min. No red precipitate is formed.

Chlorides (2.4.4)

Maximum 50 ppm.

Dilute 10 mL of solution S to 15 mL with [water R](#).

Sulfates ([2.4.13](#))

Maximum 200 ppm.

Dilute 7.5 mL of solution S to 15 mL with [distilled water R](#).

Magnesium and alkali metals

Maximum 1.0 per cent.

To 10 mL of solution S, add 80 mL of [water R](#), 10 mL of [ammonium chloride solution R](#) and 1 mL of [ammonia R](#). Heat to boiling. To the boiling solution, add dropwise 50 mL of warm [ammonium oxalate solution R](#). Allow to stand for 4 h, then dilute to 200 mL with [water R](#) and filter. To 100 mL of the filtrate, add 0.5 mL of [sulfuric acid R](#). Evaporate to dryness on a water-bath and ignite to constant mass at 600 ± 50 °C. The residue weighs a maximum of 5.0 mg.

Loss on drying ([2.2.32](#))

11.0 per cent to 12.5 per cent, determined on 0.200 g by drying at 105 °C.

Pyrogens ([2.6.8](#))

If intended for use in the manufacture of parenteral preparations without a further appropriate procedure for the removal of pyrogens, it complies with the test for pyrogens. Inject per kilogram of the rabbit's mass 4 mL of a solution containing per millilitre 50 mg of the substance to be examined.

ASSAY

Dissolve 0.240 g in 50 mL of [water R](#). Carry out the complexometric titration of calcium ([2.5.11](#)).

1 mL of [0.1 M sodium edetate](#) is equivalent to 27.03 mg of $C_{10}H_{14}CaO_6$.

STORAGE

Protected from light.

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