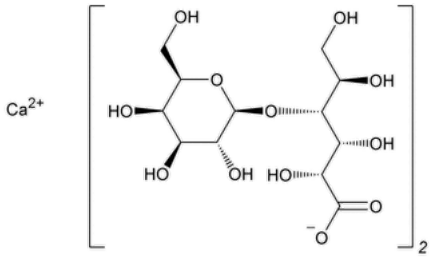
Status: Currently Official on 14-Feb-2025
Official Date: Official as of 01-May-2020
Document Type: USP Monographs
DocId: GUID-B19FE9F5-413D-4F71-B9B7-3AE2B115665D_4_en-US
DOI: https://doi.org/10.31003/USPNF_M11827_04_01
DOI Ref: jx2x0

Calcium Lactobionate



$C_{24}H_{42}CaO_{24} \cdot 2H_2O$ 790.68

D-Gluconic acid, 4-*O*-β-D-galactopyranosyl-, calcium salt (2:1), dihydrate;

Lactobionic acid, calcium salt (2:1), dihydrate;

Calcium lactobionate (1:2), dihydrate CAS RN®: 110638-68-1; UNII: 7D8YVA497F.

DEFINITION

Calcium Lactobionate contains NLT 96.0% and NMT 102.0% of calcium lactobionate ($C_{24}H_{42}CaO_{24} \cdot 2H_2O$).

IDENTIFICATION

• **A. Identification Tests—General, *Calcium*⟨191⟩:** A 20 mg/mL solution meets the requirements.

Change to read:

• **B.** ▲**Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: 197K**▲ (CN 1-May-2020)

• **C. Thin Layer Chromatography**

Standard solution: 10 mg/mL of USP Calcium Lactobionate RS in water

Sample solution: 10 mg/mL of Calcium Lactobionate in water

Chromatographic system

(See *Chromatography ⟨621⟩, Thin-Layer Chromatography*.)

Mode: TLC

Adsorbent: 0.25-mm layer of chromatographic silica gel

Application volume: 5 µL

Developing solvent system: Alcohol, ethyl acetate, ammonium hydroxide, and water (50:10:10:30)

Spray reagent: Dissolve 2.5 g of ammonium molybdate in 50 mL of 2 N sulfuric acid in a 100-mL volumetric flask. Add 1.0 g of ceric sulfate, swirl to dissolve, dilute with 2 N sulfuric acid to volume, and mix.

Analysis:

Samples: *Standard solution* and *Sample solution*

Develop the chromatogram until the solvent front has moved about three-fourths of the length of the plate. Remove the plate from the chamber, and dry at 100° for 20 min. Allow to cool, and spray with the *Spray reagent*. Heat the plate at 110° for about 10 min.

Acceptance criteria: The principal spot of the *Sample solution* corresponds in color, size, and R_F value to that of the *Standard solution*.

ASSAY

• **Procedure**

Sample: 800 mg of Calcium Lactobionate

Blank: 150 mL of water and 2 mL of 3 N hydrochloric acid

Titrimetric system

(See *Titrimetry ⟨541⟩*.)

Mode: Direct titration

Titrant: 0.05 M edetate disodium VS

Endpoint detection: Visual

Analysis: Dissolve the *Sample* in a mixture of water and 3 N hydrochloric acid (150:2). While stirring with a magnetic stirrer, add 15 mL of *Titrant* from the titration buret. Add 15 mL of 1 N sodium hydroxide and 300 mg of hydroxy naphthol blue, and continue the titration to a blue endpoint. Perform the *Blank* determination.

Calculate the percentage of calcium lactobionate ($C_{24}H_{42}CaO_{24} \cdot 2H_2O$) in the *Sample* taken:

$$\text{Result} = \{[(V_S - V_B) \times M \times F]/W\} \times 100$$

V_S = *Titrant* volume consumed by the *Sample* (mL)

V_B = *Titrant* volume consumed by the *Blank* (mL)

M = *Titrant* molarity (mM/mL)

F = equivalency factor, 790.7 mg/mmol

W = *Sample* weight (mg)

Acceptance criteria: 96.0%–102.0%

IMPURITIES

- **Halides**

Standard solution: 0.7 mL of 0.020 N hydrochloric acid

Sample: 1.2 g

Analysis: Proceed as directed for *Chloride and Sulfate ⟨221⟩, Chloride*.

Acceptance criteria: NMT 0.04%

- **Chloride and Sulfate,** *Sulfate⟨221⟩*

Standard solution: 1 mL of 0.020 N sulfuric acid

Sample: 2.0 g

Analysis: Dissolve the *Sample* in boiling water.

Acceptance criteria: NMT 0.05%

- **Reducing Substances**

Sample: 1.0 g of Calcium Lactobionate

Blank: 20 mL of water

Titrimetric system

(See *Titrimetry ⟨541⟩*.)

Mode: Residual titration

Titrant: 0.1 N iodine VS

Back-titrant: 0.1 N sodium thiosulfate VS

Endpoint detection: Visual

Analysis: Transfer the *Sample* to a 250-mL conical flask, dissolve in the *Blank*, and add 25 mL of alkaline cupric citrate TS. Cover the flask, boil gently for 5 min, accurately timed, and cool rapidly to room temperature. Add 25 mL of 0.6 N acetic acid, 10.0 mL of *Titrant*, and 10 mL of 3 N hydrochloric acid, and titrate with *Back-titrant*, adding 3 mL of starch TS as the endpoint is approached. Perform the *Blank* determination. Calculate the percentage of reducing substances (as dextrose) in the *Sample* taken:

$$\text{Result} = \{[(V_B - V_S) \times N \times F]/W\} \times 100$$

V_B = *Back-titrant* volume consumed by the *Blank* (mL)

V_S = *Back-titrant* volume consumed by the *Sample* (mL)

N = *Back-titrant* normality (mEq/mL)

F = equivalency factor, 27 mg/mEq

W = *Sample* weight (mg)

Acceptance criteria: NMT 1.0%

SPECIFIC TESTS

- **Optical Rotation,** *Specific Rotation⟨781S⟩*

Sample solution: 100 mg/mL

Acceptance criteria: +22.0° to +26.5°

- **pH ⟨791⟩**

Sample solution: 50 mg/mL

Acceptance criteria: 5.4–7.4

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in well-closed containers.
- **USP Reference Standards ⟨11⟩**

USP Calcium Lactobionate RS

Topic/Question	Contact	Expert Committee
CALCIUM LACTOBIONATE	Nagaphani Batchu Senior Scientist I, Documentary Standards	NBDS2020 Non-botanical Dietary Supplements

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 27(6)

Current DocID: GUID-B19FE9F5-413D-4F71-B9B7-3AE2B115665D_4_en-US
DOI: https://doi.org/10.31003/USPNF_M11827_04_01
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