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Calcium Stearate

Octadecanoic acid, calcium salt;
Calcium stearate
CAS RN®: 1592-23-0.

DEFINITION

Calcium Stearate is a compound of calcium with a mixture of solid organic acids obtained from sources of vegetable or animal origin and consists mainly of variable proportions of calcium stearate ($C_{36}H_{70}CaO_4$) and calcium palmitate ($C_{32}H_{62}CaO_4$). It contains NLT 6.4% and NMT 7.4% of calcium (Ca), calculated on the dried basis. The content of stearic acid in the fatty acid fraction is NLT 40.0% of the total content. The sum of stearic acid and palmitic acid in the fatty acid fraction is NLT 90.0% of the total content.

IDENTIFICATION

• **A. Identification Tests—General, *Calcium* ⟨191⟩**

Sample: 1 g

Analysis: Heat the *Sample* with a mixture of 25 mL of water and 5 mL of hydrochloric acid.

Acceptance criteria: Fatty acids are liberated and appear as an oily layer floating on the surface of the liquid. The water layer meets the requirements.

• **B.** The retention times of the major peaks of the *Sample solution* correspond to those of the *Standard solution*, as obtained in the *Assay* for *Content of Stearic Acid and Palmitic Acid*.

ASSAY

• **Content of Calcium**

Sample: 1.2 g

Titrimetric system

(See *Titrimetry* ⟨541⟩.)

Mode: Direct titration

Titrant: 0.05 M edetate disodium VS

Endpoint detection: Colorimetric

Analysis: Boil the *Sample* with 50 mL of 1 N sulfuric acid for about 3 h, using a watch glass cover to avoid splattering, or until the separated fatty acid layer is clear. Add water, if necessary, to maintain the original volume. [Note—Stirring may be helpful in obtaining a clear layer and decreasing extraction time.] Cool, filter, and wash the filter and the flask thoroughly with water until the last washing is not acid to litmus. Neutralize the filtrate with 1 N sodium hydroxide to litmus. While stirring, preferably with a magnetic stirrer, titrate with 0.05 M edetate disodium VS as follows. Add about 30 mL from a 50-mL buret, then add 15 mL of 1 N sodium hydroxide and 300 mg of hydroxy naphthol blue. Continue the titration to a blue endpoint. Each mL of 0.05 M edetate disodium is equivalent to 2.003 mg of calcium.

Acceptance criteria: 6.4%–7.4% on the dried basis

• **Content of Stearic Acid and Palmitic Acid**

Boron trifluoride–methanol solution: 140 g/L of boron trifluoride in methanol

Sample solution: Dissolve 100 mg of Calcium Stearate in a small conical flask fitted with a suitable reflux attachment with 5 mL of *Boron trifluoride–methanol solution*. Boil under reflux for 10 min. Add 4.0 mL of *n*-heptane through the condenser, and boil again under reflux for 10 min. Allow to cool. Add 20 mL of a saturated solution of sodium chloride. Shake, and allow the layers to separate. Remove about 2 mL of the organic layer, and dry it over 0.2 g of anhydrous sodium sulfate. Dilute 1.0 mL of this solution with *n*-heptane to 10.0 mL.

Standard solution: Prepare as directed in the *Sample solution*, using 50 mg of USP Stearic Acid RS and 50 mg of USP Palmitic Acid RS.

Chromatographic system

(See *Chromatography* ⟨621⟩, *System Suitability*.)

Mode: GC

Detector: Flame ionization

Column: 30-m × 0.32-mm fused silica; 0.5-µm layer of phase G16

Temperatures

Injection port: 220°

Detector: 260°

Column: See *Table 1*.

Table 1

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
70	—	70	2
70	5	240	5

Carrier gas: Helium, passed through a bed of molecular sieve for drying, if necessary

Flow rate: 2.4 mL/min

Injection volume: 1 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Resolution: NLT 5.0 between the methyl palmitate and methyl stearate peaks. [NOTE—The relative retention times for methyl palmitate and methyl stearate are about 0.9 and 1.0, respectively.]

Relative standard deviation: NMT 3.0% for the methyl stearate and methyl palmitate peaks; NMT 1.0% for the ratio of the peak areas of methyl palmitate to the peak areas of methyl stearate, from 6 replicate injections

Analysis: Calculate the percentage of stearic acid ($C_{18}H_{36}O_2$) in the fatty acid fraction of the sample taken:

$$\text{Result} = (r_U/r_T) \times 100$$

r_U = peak area due to methyl stearate

r_T = sum of all the peak areas, excluding the solvent peak

Calculate the percentage of palmitic acid ($C_{16}H_{32}O_2$) in the fatty acid fraction of the sample taken:

$$\text{Result} = (r_U/r_T) \times 100$$

r_U = peak area due to methyl palmitate

r_T = sum of all the peak areas, excluding the solvent peak

Acceptance criteria

Stearic acid: NLT 40.0%

Sum of stearic acid and palmitic acid: NLT 90.0%

SPECIFIC TESTS

• **Loss on Drying ⟨731⟩**

Analysis: Dry a sample at 105° to constant weight.

Acceptance criteria: NMT 4.0%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in well-closed containers.

• **Labeling:** Label it to indicate the content of stearic acid in the fatty acid fraction and to indicate the fatty acids used to produce calcium stearate are from sources of vegetable or animal origin.

• **USP Reference Standards ⟨11⟩**

USP Palmitic Acid RS

USP Stearic Acid RS

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
CALCIUM STEARATE	Documentary Standards Support	CE2020 Complex Excipients

Chromatographic Database Information: Chromatographic Database

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