Status: Currently Official on 14-Feb-2025
Official Date: Official as of 01-Mar-2019
Document Type: NF Monographs
DocId: GUID-58FB8205-2E75-4B19-86D8-CE49B262EC97_4_en-US
DOI: https://doi.org/10.31003/USPNF_M12120_04_01
DOI Ref: zr7x9

Calcium Silicate

DEFINITION

Calcium Silicate, crystalline or amorphous, is a compound of calcium oxide and silicon dioxide. It contains NLT 4.0% of calcium oxide and NLT 35.0% of silicon dioxide.

IDENTIFICATION

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

Sample solution: Mix 0.5 g with 10 mL of 3 N hydrochloric acid. Filter, and neutralize the filtrate to litmus paper with 6 N ammonium hydroxide.

Acceptance criteria: The neutralized filtrate meets the requirements.

- **B.**

Analysis: Prepare a bead by fusing a few crystals of sodium ammonium phosphate on a platinum loop in the flame of a gas burner. Place the hot, transparent bead in contact with the specimen of Calcium Silicate, and again fuse.

Acceptance criteria: Silica floats about in the bead, producing, upon cooling, an opaque bead having a web-like structure.

ASSAY

- **Silicon Dioxide**

Sample: Calcium Silicate in the appropriate amount (see *Table 1*)

Table 1

Sample Weight (mg)	Calcium Oxide Content (%)
400	>25
600	11–25
1000	4–10

Analysis: Transfer the *Sample* to a beaker, add 5 mL of water and 10 mL of perchloric acid, and heat until dense white fumes of perchloric acid are evolved. Cover the beaker with a watch glass, and continue to heat for 2 h. Allow to cool, add 30 mL of water, filter, and wash the precipitate with 200 mL of hot water. [Note—Retain the combined filtrate and washings for use in the assay for *Calcium Oxide*.]

Transfer the filter paper and its contents to a platinum crucible, heat slowly to dryness, then heat sufficiently to char the filter paper. Ignite at about 900°–1000° to constant weight. Moisten the residue with 5 drops of perchloric acid, add 15 mL of hydrofluoric acid, heat cautiously on a hot plate until all of the acid is driven off, and ignite at a temperature NLT 1000° to constant weight. Cool in a desiccator, and weigh: the loss in weight represents the weight of SiO_2.

Acceptance criteria: NLT 35.0% of silicon dioxide (SiO_2) and 90.0%–110.0% of the content stated in the labeling, or within the range of percentages stated in the labeling

- **Calcium Oxide**

Sample solution: Use the combined filtrate and washings retained from the assay for *Silicon Dioxide*.

Titrimetric system

(See *Titrimetry* ⟨541⟩.)

Mode: Direct titration

Titrant: 0.05 M edetate disodium VS

Endpoint detection: Visual

Analysis: Neutralize the *Sample solution* to litmus with 1 N sodium hydroxide. Add, while stirring, 10 mL of 0.05 M edetate disodium VS from a 50-mL buret. Add 15 mL of 1 N sodium hydroxide and 300 mg of hydroxy naphthol blue, and continue the titration to a blue endpoint.

Calculate *Z*, the percentage of calcium oxide (CaO) in the sample taken:

$$Z = [(V \times M \times F)/W] \times 100$$

V = volume of *Titrant* consumed by the *Sample solution* (mL)

M = actual molarity of the *Titrant* (mmol/mL)

F = equivalency factor, 56.08 mg/mmol

W = weight of the sample (mg)

Calculate the percentage of the labeled amount of calcium oxide (CaO):

$$\text{Result} = (Z/L) \times 100$$

Z = percentage of CaO in the sample taken as calculated above

L = labeled amount of CaO

Acceptance criteria: NLT 4.0% of calcium oxide (CaO) and 90.0%–110.0% of the content stated in the labeling, or within the range of percentages stated in the labeling

IMPURITIES

- **Loss on Ignition ⟨733⟩:** Transfer 1 g to a suitable tared crucible, dry at 105° for 2 h, and ignite at 900° to constant weight: it loses NMT 20.0% of its weight.

Change to read:

- **Limit of Lead**

Palladium matrix modifier: Palladium nitrate (1% Pd)[1]

Magnesium matrix modifier: Magnesium nitrate (2% Mg)[2]

Nitric acid, 65% [7697-37-2]: Use a suitable grade with a content of NLT 65.0%.[3]

Nitric acid diluent: Transfer 42 mL of *Nitric acid, 65%* to a 1000-mL volumetric flask. Dilute with water to volume.

Matrix modifier solution: Transfer 1.0 mL of *Palladium matrix modifier* and 100 µL of *Magnesium matrix modifier* to a 20-mL volumetric flask. Dilute with water to volume, and mix.

Lead standard solution: A solution containing $Pb(NO_3)_2$ in 0.5 M nitric acid corresponding to 1000 mg of ▲lead/L▲ (ERR 1-Mar-2019) [4]

Standard solution: Transfer 100 µL of *Lead standard solution* to a 100-mL volumetric flask. Dilute with water to volume, and mix. This solution contains the equivalent of 1.0 µg/mL of lead.

Calibration solutions

Calibration solution A: Transfer 50 mL of *Nitric acid diluent* to a 100-mL volumetric flask, and dilute with water to volume (0 µg/mL of lead).

Calibration solution B: Transfer 1 mL of *Standard solution* and 50 mL of *Nitric acid diluent* to a 100-mL volumetric flask, and dilute with water to volume (0.01 µg/mL of lead).

Calibration solution C: Transfer 5 mL of *Standard solution* and 50 mL of *Nitric acid diluent* to a 100-mL volumetric flask, and dilute with water to volume (0.05 µg/mL of lead).

Sample solution: Weigh 2.0 g of Calcium Silicate into a 150-mL beaker. Mix with 50 mL of *Nitric acid diluent*. Cover with a watch glass, and boil for 20 min. Allow to cool. With the aid of a vacuum, pass through a glass filter,[5] and wash the filter several times with water. Transfer the filtrate into a 100-mL volumetric flask. Dilute with water to volume, and mix.

System suitability solution: Transfer 5 mL of *Standard solution* and 50 mL of *Nitric acid diluent* into a 100-mL volumetric flask. Dilute with water to volume, and mix. This solution contains the equivalent of 0.05 µg of lead/mL.

Instrumental conditions

(See *Atomic Absorption Spectroscopy ⟨852⟩*.)

Mode: Graphite furnace atomic absorption spectrophotometer

Analytical wavelength: 283.3 nm (lead emission line)

Lamp: Lead hollow-cathode lamp and an adequate means of Zeeman background correction

Carrier gas: Argon

Under typical conditions, the *Sample solution* and *Calibration solutions* volumes are 20 µL, the volume of the *Matrix modifier solution* is 10 µL, the injection temperature is 20°, and the oven conditions are as follows (see *Table 2*).

[Note—These conditions may be optimized for each instrument.]

Table 2

Step	Temperature (°)
Drying 1	110
Drying 2	130
Pyrrolysis	950

Step	Temperature (°)
Read	1800
Clean out	2450

Analysis

Samples: *Calibration solutions*, *Sample solution*, and *System suitability solution*

Plot the absorbance of each *Calibration solution* versus its content of lead, in µg/mL, and draw the best straight line fitting the three points. From this plot, determine the concentration, in µg/mL, of lead in the *Sample solution*: the correlation coefficient is NLT 0.99, and the recovery for the *System suitability solution* is 85%–115%.

Calculate the quantity, in ppm, of lead in the *Sample solution*.

$$\text{Result} = (C \times V)/W$$

C = concentration of the *Sample solution* (µg/mL)

V = volume of the *Sample solution* (mL)

W = weight of sample taken (g)

Acceptance criteria: NMT 10 ppm

- **Limit of Fluoride**

[Note—Store all solutions in polytef containers.]

Buffer solution: 294 mg/mL of sodium citrate

Ionic strength adjustment buffer: Transfer 42 mL of hydrochloric acid, 121 g of tris(hydroxymethyl)aminomethane, and 115 g of sodium tartrate to a 500-mL volumetric flask containing 250 mL of water. Stir to dissolve, and dilute with water to volume.

Standard stock solution: 221 µg/mL of USP Sodium Fluoride RS. This stock solution contains 100 µg/mL of fluoride ion.

Sample solution: Transfer 2.0 g of Calcium Silicate to a 100-mL polytef beaker containing a magnetic stir bar. Add 20 mL of water and 2.0 mL of hydrochloric acid. Cover with a watch glass, and heat to a vigorous boil for 1 min, stirring continuously. Remove from heat, and cool. Transfer the cooled suspension to a 100-mL polytef beaker. Add 25 mL of *Buffer solution,* and adjust with ammonium hydroxide or hydrochloric acid to a pH of 5–6. Add 50 mL of *Ionic strength adjustment buffer* and water to make 100 mL of solution.

Electrode system: Use a fluoride-specific ion-indicating electrode and a suitable reference electrode connected to a pH meter capable of measuring potentials with a reproducibility of ±0.2 mV (see pH ⟨791⟩).

Standard response line: Obtain a standard response line with four standard solutions containing 0, 0.10, 0.20, and 0.40 µg/mL of fluoride as follows. Add 23 mL of water, 2 mL of hydrochloric acid, and 25 mL of *Buffer solution* to a 100-mL plastic beaker. Adjust with ammonium hydroxide to a pH of 5–6, and add *Ionic strength adjustment buffer* to obtain 100 mL of solution. Insert the electrode into the solution, stir for at least 15 min, and record the potential for the standard solution containing 0 µg/mL of fluoride. When the electrode has stabilized, add 100 µL of the *Standard stock solution* to the beaker, and stir. Allow the electrode to stabilize for 5 min, and measure the potential for the standard solution containing 0.10 µg/mL of fluoride. Similarly add another 100 and 200 µL of the *Standard stock solution* and record the potential for the standard solutions, containing 0.20 µg/mL of fluoride and 0.40 µg/mL of fluoride, respectively. After each addition, continue to stir for 5 min before recording the reading.

Analysis: Insert the calibrated electrode into the *Sample solution,* stir for 5 min, and record the measurement. From the measured potential of the *Sample solution* and the *Standard response line,* determine the concentration, C, in µg/mL, of fluoride ion in the *Sample solution.* Calculate the quantity, in ppm, of fluoride in Calcium Silicate:

$$\text{Result} = (C \times V)/W$$

C = concentration of fluoride ion in the *Sample solution* (µg/mL)

V = volume of the *Sample solution* (mL)

W = weight of Calcium Silicate in the *Sample solution* (g)

Acceptance criteria: NMT 50 ppm

SPECIFIC TESTS

- **pH ⟨791⟩:** 8.4–11.2, determined in a well-mixed aqueous suspension (1 in 20)
- **Ratio of Silicon Dioxide to Calcium Oxide:** Divide the percentage of silicon dioxide obtained in the test for *Silicon Dioxide* by the percentage of calcium oxide obtained in the test for *Calcium Oxide*.

Acceptance criteria: 0.5–20

- **Sum of CaO, SiO$_2$, and Loss on Ignition:** The sum of the percentages obtained in the three tests is NLT 90.0%.

ADDITIONAL REQUIREMENTS

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

- **Labeling:** The labeling states the claimed percentage or range of percentages for the content of calcium oxide and for the content of silicon dioxide.
- **USP Reference Standards ⟨11⟩**
  USP Sodium Fluoride RS

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[1] A suitable grade is available as catalog number RCMMPD10KN-50 from VWR, www.vwr.com.

[2] Available as catalog number RCMMMG20KN-50, from VWR, www.vwr.com.

[3] A suitable grade is available as catalog number 441-2 from EMD Chemicals, www.emdchemicals.com.

[4] A suitable grade is available as catalog number 1.19776 from EMD Chemicals, www.emdchemicals.com.

[5] Glass filter: Whatman GF/B glass microfiber filters, 1.0 µm, Whatman no. 1821-090, is suitable. Available as catalog number 28497-492, from VWR, www.vwr.com.

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Topic/Question	Contact	Expert Committee
CALCIUM SILICATE	Documentary Standards Support	SE2020 Simple Excipients

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 33(3)

Current DocID: GUID-58FB8205-2E75-4B19-86D8-CE49B262EC97_4_en-US
DOI: https://doi.org/10.31003/USPNF_M12120_04_01
DOI ref: zr7x9