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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 SiO₂.

Acceptance criteria: NLT 35.0% of silicon dioxide (SiO₂) 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 $\text{Pb}(\text{NO}_3)_2$ 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



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