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Calcium Carbonate

$CaCO_3$ 100.09

Carbonic acid, calcium salt (1:1);

Calcium carbonate (1:1) CAS RN®: 471-34-1.

DEFINITION

Calcium Carbonate, dried at 200° for 4 h, contains calcium equivalent to NLT 98.0% and NMT 100.5% of calcium carbonate ($CaCO_3$).

IDENTIFICATION

• **A. Identification Tests—General, *Calcium*⟨191⟩:** The addition of acetic acid to it produces effervescence (presence of carbonate), and the resulting solution, after boiling, meets the requirements of the tests.

ASSAY

• **Titrimetry ⟨541⟩**

Sample: 200 mg of Calcium Carbonate, previously dried at 200° for 4 h

Blank: 100 mL of water and 15 mL of 1 N sodium hydroxide

Titrimetric system

(See *Titrimetry ⟨541⟩*.)

Mode: Direct titration

Titrant: 0.05 M edetate disodium VS

Indicator: 300 mg of hydroxy naphthol blue

Endpoint detection: Visual, change to distinct blue

Analysis: Transfer the *Sample* to a 250-mL beaker. Moisten thoroughly with a few mL of water, and add, dropwise, sufficient 3 N hydrochloric acid to dissolve. Add 100 mL of water, 15 mL of 1 N sodium hydroxide, and 300 mg of hydroxy naphthol blue. Titrate with the *Titrant*. Calculate the percentage of calcium carbonate ($CaCO_3$) in the *Sample* taken:

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

V = *Sample* titrant volume (mL)

B = *Blank* titrant volume (mL)

M = titrant molarity (mmol/mL)

F = equivalency factor, 100.09 mg/mmol

W = weight of the *Sample* (mg)

Acceptance criteria: 98.0%–100.5% on the dried basis

IMPURITIES

• **Acid-Insoluble Substances**

Sample: 5.0 g

Analysis: Mix the *Sample* with 10 mL of water, and add hydrochloric acid, dropwise, with agitation, until it ceases to cause effervescence, then add water to make the mixture measure 200 mL, and filter. Wash the insoluble residue with water until the last washing shows no chloride, and ignite and weigh the residue.

Acceptance criteria: NMT 0.2%; the weight of the residue does not exceed 10 mg.

Change to read:

• ▲**Arsenic ⟨211⟩, *Procedures, Procedure 1*▲** (CN 1-Jun-2023)

Sample solution: Slowly dissolve 1.0 g in 15 mL of hydrochloric acid, and dilute with water to 55 mL.

Analysis: Omit the addition of 20 mL of 7 N sulfuric acid specified in ▲*Arsenic ⟨211⟩, Procedures, Procedure 1*▲ (CN 1-Jun-2023).

Acceptance criteria: NMT 3 ppm

• **Barium:** A platinum wire, dipped in the filtrate obtained in the test for *Acid-Insoluble Substances* and held in a nonluminous flame, does not impart a green color.

Change to read:

• ▲**Iron ⟨241⟩, *Procedures, Procedure 1*** ▲(CN 1-Jun-2023)

Sample solution: 40 mg in 5 mL of 2 N hydrochloric acid. Transfer to a beaker with the aid of water, and dilute with water to 10 mL.

Standard solution: Transfer 4.0 mL of the *Standard Iron Solution*, prepared as directed in ▲*Iron ⟨241⟩, Procedures, Procedure 1*▲ (CN 1-Jun-2023), to a beaker, and dilute with water to 10 mL.

Instrumental conditions

(See *Ultraviolet-Visible Spectroscopy ⟨857⟩*.)

Analytical wavelength: 530 nm

Blank: Water

Analysis: Separately to the *Sample solution* and *Standard solution* add 2 mL of citric acid solution (1 in 5) and 2 drops of thioglycolic acid, adjust with ammonia TS to a pH of 9.5 ± 0.1, dilute with water to 20 mL, and allow to stand for 5 min. Dilute with water to 50 mL. Concomitantly determine the absorbances of the solutions from the *Sample solution* and the *Standard solution*.

Acceptance criteria: NMT 0.1%; the absorbance of the solution from the *Sample solution* does not exceed that of the *Standard solution*.

Change to read:

• ▲**Lead ⟨251⟩, *Procedures, Procedure 1*** ▲(CN 1-Jun-2023)

Sample solution: 1.0 g in 5 mL of water

Analysis: To the *Sample solution* slowly add 8 mL of 3 N hydrochloric acid, evaporate on a steam bath to dryness, and dissolve the residue in 5 mL of water.

Acceptance criteria: NMT 3 ppm

• **Limit of Fluoride**

[Note—Prepare and store all solutions in plastic containers.]

Solution A: 294 mg/mL of sodium citrate dihydrate in water

Sample: 2.0 g

Standard stock solution: 1.11 mg/mL of USP Sodium Fluoride RS in water

Standard solution: Combine 20.0 mL of the *Standard stock solution* with 50.0 mL of *Solution A*, and dilute with water to 100.0 mL. [Note—Each mL of this solution contains 100 µg of fluoride ion]

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

Standard response line: Transfer 50.0 mL of *Solution A* and 4.0 mL of hydrochloric acid to a beaker, and add water to make 100 mL. Add a plastic-coated stirring bar, insert the electrodes into the solution, stir for 15 min, and read the potential, in mV. Continue stirring, and at 5-min intervals add 100, 100, 300, and 500 µL of the *Standard solution*, reading the potential 5 min after each addition. Plot the logarithms of the cumulative fluoride ion concentrations (0.1, 0.2, 0.5, and 1.0 µg/mL) versus potential, in mV.

Analysis: Transfer the *Sample* to a beaker containing a plastic-coated stirring bar, add 20 mL of water and 4.0 mL of hydrochloric acid, and stir until dissolved. Add 50.0 mL of *Solution A* and sufficient water to make 100 mL of test solution. Rinse and dry the electrodes, insert them into the *Sample solution*, stir for 5 min, and read the potential, in mV. From the measured potential and the *Standard response line*, determine the concentration, *C*, in µg/mL, of fluoride ion in the *Sample solution*. Calculate the content of fluoride in the specimen taken:

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

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

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

W = weight of *Sample* (g)

Acceptance criteria: NMT 50 ppm

• **Limit of Magnesium and Alkali Salts**

Sample: 1.0 g

Analysis: Mix the *Sample* with 35 mL of water. Carefully add 3 mL of hydrochloric acid, heat the solution, and boil for 1 min. Rapidly add 40 mL of oxalic acid TS, and stir vigorously until precipitation is well-established. Add immediately to the warm mixture 2 drops of methyl red TS and then 6 N ammonium hydroxide, dropwise, until the mixture is just alkaline. Cool to room temperature, transfer to a 100-mL graduated cylinder, dilute with water to 100 mL, mix, and allow to stand for 4 h or overnight. Filter, and to 50 mL of the clear filtrate in a platinum dish add 0.5 mL of sulfuric acid, and evaporate the mixture on a steam bath to a small volume. Carefully heat over a free flame to dryness, and continue heating to complete decomposition and volatilization of ammonium salts. Finally, ignite the residue to constant weight.

Acceptance criteria: NMT 1.0%; the weight of the residue is NMT 5 mg.

Change to read:

• ▲**Mercury ⟨261⟩, *Procedures, Procedure 2*** ▲(CN 1-Jun-2023)

Mercury stock solution and **Standard mercury solution:** Proceed as directed in *Mercury ⟨261⟩*.

Standard solution: Proceed as directed in *Mercury ⟨261⟩*, except use 3 mL of hydrochloric acid instead of 3 mL of sulfuric acid.

Sample stock solution: 4.0 g in a 100-mL beaker, and cautiously dissolve in 14 mL of 6 N hydrochloric acid

Sample solution: Proceed as directed in *Mercury* ⟨261⟩ using the *Sample stock solution*, except use 3 mL of hydrochloric acid instead of 3 mL of sulfuric acid.

Analysis

Samples: *Standard solution* and *Sample solution*

Proceed as directed in *Mercury* ⟨261⟩.

Acceptance criteria: NMT 0.5 ppm

SPECIFIC TESTS

• **Loss on Drying** ⟨731⟩**:** Dry a sample at 200° for 4 h: it loses NMT 2.0% of its weight.

ADDITIONAL REQUIREMENTS

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

• **USP Reference Standards** ⟨11⟩

USP Sodium Fluoride RS

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

Topic/Question	Contact	Expert Committee
CALCIUM CARBONATE	Nagaphani Batchu Senior Scientist I, Documentary Standards	NBDS2020 Non-botanical Dietary Supplements
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	NBDS2020 Non-botanical Dietary Supplements

Chromatographic Database Information: Chromatographic Database

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