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Ground Limestone

DEFINITION

Ground Limestone is a fine, white to off-white, microcrystalline powder mainly consisting of calcium carbonate. It is obtained by crushing, grinding, and classifying naturally occurring limestone, benefited by flotation and/or air classification. After drying at 200° for 4 h, it contains NLT 94.0% and NMT 100.5% of calcium carbonate (CaCO₃).

IDENTIFICATION

- **A. IDENTIFICATION TESTS—GENERAL, *Calcium* (191).**

Analysis: Add acetic acid to the Ground Limestone, and boil the resulting solution.

Acceptance criteria: The solution produces effervescence (presence of carbonate) after the addition of acetic acid. After boiling, it meets the requirements in the chapter.

- **B. ACID-INSOLUBLE SUBSTANCES:** It meets the requirements in the test for *Acid-Insoluble Substances*.

ASSAY

PROCEDURE

Sample: 200 mg of Ground Limestone, previously dried at 200° for 4 h

Blank: Proceed as directed in the *Analysis*, except omit the test specimen.

Titrimetric system

(See *Titrimetry* (541).)

Mode: Direct titration

Titrant: 0.05 M edetate disodium VS

Endpoint detection: Visual

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, and titrate with the *Titrant* until the solution is a distinct blue color. Perform a *Blank* determination.

Calculate the percentage of calcium carbonate (CaCO₃) 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 = actual molarity of the *Titrant* (mM/mL)

F = equivalency factor, 100.1 mg/mM

W = *Sample* weight (mg)

Acceptance criteria: 94.0%–100.5%

IMPURITIES

Change to read:

- **▲ *ARSENIC* (211), *Procedures, Procedure 1* ▲** (CN 1-JUN-2023)

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

Analysis: Proceed as directed in the chapter, except omit the addition of 20 mL of 7 N sulfuric acid.

Acceptance criteria: It meets the requirements in the chapter (NMT 3 ppm).

Change to read:

- **▲ *LEAD* (251), *Procedures, Procedure 1* ▲** (CN 1-JUN-2023)

Test preparation: Mix 1.0 g with 5 mL of water, 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 MAGNESIUM AND ALKALI SALTS**

Sample: 1.0 g

Analysis: Mix the *Sample* with 40 mL of water. Carefully add 5 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. Ignite the residue to constant weight.

Acceptance criteria: NMT 3.5%. The weight of the residue is NMT 17.5 mg.

• **LIMIT OF FLUORIDE**

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

Buffer solution: 294 mg/mL of sodium citrate dihydrate in water

Standard stock solution: 1.1052 mg/mL of [USP Sodium Fluoride RS](#) in water

Standard solution: Transfer 20.0 mL of *Standard stock solution* to a 100-mL volumetric flask containing 50.0 mL of *Buffer solution*, dilute with water to volume, and mix. Each mL of *Standard solution* contains 100 µg of fluoride ion.

Sample solution: Transfer 2.0 g of Ground Limestone 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 *Buffer solution* and sufficient water to make 100 mL.

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 \(791\)](#)).

Analysis

Samples: *Standard solution* and *Sample solution*

Standard response line: Transfer 50.0 mL of *Buffer solution* 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 *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.

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 portion of Ground Limestone taken:

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

C = concentration of fluoride ion, obtained from the *Standard response line*, in the *Sample solution* (µg/mL)

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

W = weight of Ground Limestone taken to prepare the *Sample solution* (g)

Acceptance criteria: NMT 50 ppm

SPECIFIC TESTS

• **ACID-INSOLUBLE SUBSTANCES**

Sample: 5.0 g

Analysis: Mix the *Sample* with 25 mL of water. Add 25 mL of hydrochloric acid, dropwise, with agitation, until it ceases to cause effervescence. 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: 0.2%–2.5%. The weight of the residue is between 10 and 125 mg.

• **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
GROUND LIMESTONE	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](#)