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Magnesium Hydroxide Paste

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<https://www.uspnf.com/rb-magnesium-hydroxide-paste-20211119>.

DEFINITION

Magnesium Hydroxide Paste is an aqueous paste of Magnesium Hydroxide. It contains NLT 93.0% and NMT 107.0% of the labeled amount of magnesium hydroxide $[\text{Mg}(\text{OH})_2]$, the labeled amount being NLT 28.0% and NMT 70.0% of magnesium hydroxide.

IDENTIFICATION

Change to read:

- **A. IDENTIFICATION TESTS—GENERAL (191),** [Chemical Identification Tests, Magnesium](#)

Sample solution: 100 mg/mL ▲ of Paste▲ (RB 1-Dec-2022) in 3 N [hydrochloric acid](#)

Acceptance criteria: Meets the requirements

Delete the following:

- ▲ **B.** The retention time of the magnesium peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the Assay.▲ (RB 1-Dec-2022)

ASSAY

Change to read:

- **PROCEDURE**

▲ **Sample:** A portion of Paste equivalent to about 250 mg of magnesium hydroxide

Titrimetric system

Mode: Direct titration

Titrant: [0.05 M edetate disodium VS](#)

Endpoint detection: Visual

Analysis: Transfer the *Sample* to a 100-mL volumetric flask. Dissolve in 10 mL of 3 N [hydrochloric acid](#) and dilute with [water](#) to volume. Filter, if necessary, and transfer 25.0 mL of the filtrate to a beaker containing 75 mL of [water](#). Adjust the reaction of the solution to a pH of 7 (using pH indicator paper; see [Reagents, Indicators, and Solutions—Indicator and Test Papers](#)) with 1 N [sodium hydroxide](#), add 5 mL of [ammonia–ammonium chloride buffer TS](#) and 0.15 mL of [eriochrome black TS](#), and titrate with the *Titrant* to a blue endpoint. Each milliliter of 0.05 M edetate disodium is equivalent to 2.916 mg of magnesium hydroxide $[\text{Mg}(\text{OH})_2]$.▲ (RB 1-Dec-2022)

Acceptance criteria: 93.0%–107.0%

IMPURITIES

- **SOLUBLE ALKALIES**

Diluted paste solution: Accurately weigh a portion of Paste, equivalent to about 7.75 g of magnesium hydroxide, and mix with 75.0 mL of [water](#).

Sample solution: Pass about 25 mL of the *Diluted paste solution* through a suitable filter, discard the first 5 mL of the filtrate, and collect the rest of the filtrate.

[NOTE—Keep the remaining *Diluted paste solution* for use in the test for *Carbonate and Acid-Insoluble Matter*.]

Titrimetric system

Mode: Direct titration

Titrant: 0.10 N [sulfuric acid](#)

Endpoint detection: Visual

Analysis: Dilute 5 mL of the clear *Sample solution* with 40 mL of [water](#). Add 1 drop of [methyl red TS](#), and titrate with *Titrant* to the production of a persistent pink color.

[NOTE—Keep the remaining *Sample solution* for use in the test for *Soluble Salts*.]

Acceptance criteria: NMT 1.0 mL of *Titrant* is required.

- **SOLUBLE SALTS**

Analysis: To 5.0 mL of the clear *Sample solution* from the test for *Soluble Alkalies* add 3 drops of [sulfuric acid](#), evaporate on a steam bath to dryness, and ignite gently to constant weight.

Acceptance criteria: NMT 12 mg of residue remains.

• **CARBONATE AND ACID-INSOLUBLE MATTER**

Analysis: To 1 mL of the *Diluted paste solution* from the test for *Soluble Alkalies* add 2 mL of 3 N [hydrochloric acid](#).

Acceptance criteria: NMT a slight effervescence occurs, and the solution is NMT slightly turbid.

Change to read:

• **LIMIT OF CALCIUM**

▲[NOTE—A commercially available atomic absorption standard solution for calcium may be used where preparation of a calcium standard stock solution is described below. Concentrations of the *Standard solutions* and the *Sample solution* may be modified to fit the linear or working range of the instrument.]

Dilute hydrochloric acid: Dilute 100 mL of [hydrochloric acid](#) with [water](#) to 1000 mL.

Lanthanum solution: 50 mg/mL of lanthanum prepared as follows. To 58.65 g of [lanthanum oxide](#) add 400 mL of [water](#), and add, gradually with stirring, 250 mL of [hydrochloric acid](#). Stir until dissolved, and dilute with [water](#) to 1000 mL.

Blank solution: Transfer 4 mL of the *Lanthanum solution* and 10 mL of *Dilute hydrochloric acid* to a 200-mL volumetric flask, and dilute with [water](#) to volume.

Standard stock solution: 1.0 mg/mL of calcium prepared as follows. Transfer 249.7 mg of calcium carbonate, previously dried at 300° for 3 h and cooled in a desiccator for 2 h, to a 100-mL volumetric flask. Dissolve in a minimum amount of [hydrochloric acid](#), and dilute with [water](#) to volume.

Standard solution 1: 1.0 µg/mL of calcium prepared as follows. Transfer 1.0 mL of *Standard stock solution* to a 1000-mL volumetric flask containing 20 mL of the *Lanthanum solution* and 40 mL of *Dilute hydrochloric acid*, and dilute with [water](#) to volume.

Standard solution 2: 5.0 µg/mL of calcium prepared as follows. Transfer 5.0 mL of *Standard stock solution* to a 1000-mL volumetric flask containing 20 mL of the *Lanthanum solution* and 40 mL of *Dilute hydrochloric acid*, and dilute with [water](#) to volume.

Standard solution 3: 10.0 µg/mL of calcium prepared as follows. Transfer 10.0 mL of *Standard stock solution* to a 1000-mL volumetric flask containing 20 mL of the *Lanthanum solution* and 40 mL of *Dilute hydrochloric acid*, and dilute with [water](#) to volume.

Standard solution 4: 15.0 µg/mL of calcium prepared as follows. Transfer 15.0 mL of *Standard stock solution* to a 1000-mL volumetric flask containing 20 mL of the *Lanthanum solution* and 40 mL of *Dilute hydrochloric acid*, and dilute with [water](#) to volume.

Sample solution: Nominally 1.25 mg/mL of magnesium hydroxide prepared as follows. Transfer a portion of the Paste, equivalent to 250 mg of magnesium hydroxide, to a beaker, add 30 mL of *Dilute hydrochloric acid*, and stir until dissolved, heating if necessary. Transfer the solution so obtained to a 200-mL volumetric flask containing 4 mL of *Lanthanum solution*, and dilute with [water](#) to volume.

Instrumental conditions

(See [Atomic Absorption Spectroscopy \(852\)](#).)

Mode: Atomic absorption spectrophotometry

Analytical wavelength: 422.7 nm

Lamp: Calcium hollow-cathode

Flame: Nitrous oxide-acetylene

Analysis

Samples: *Blank solution*, *Standard solutions*, and *Sample solution*

Using the *Blank solution* as the blank, determine the concentration, C_s , in µg/mL, of calcium in the *Sample solution* using the calibration graph.

Calculate the percentage of calcium in the portion of Paste taken:

$$\text{Result} = [C_s \times (F/C_U)] \times 100$$

C_s = concentration of calcium in the *Sample solution* (µg/mL)

F = unit conversion factor, 0.001 mg/µg

C_U = nominal concentration of magnesium hydroxide in the *Sample solution* (mg/mL)

Acceptance criteria: NMT 1.5%▲ (RB 1-Dec-2022)

• **LIMIT OF LEAD**

[NOTE— Use water with a resistivity of NLT 18 megohm-cm to prepare the solutions.]

Internal standard solution: 20 µg/L of thallium in [water](#) [from commercially available, National Institute of Standards and Technology (NIST)-traceable standard solution for thallium]. [NOTE—Use the *Internal standard solution* only if an inductively coupled plasma-mass spectrometry (ICP-MS) instrument is used. The *Internal standard solution* is added inline via a mixing block between the sample probe and the spray chamber.]

Blank solution: 6.0% (v/v) [nitric acid](#) in [water](#)

Diluent: 2.0% (v/v) [nitric acid](#) in [water](#)

Standard stock solution: 100 µg/L of lead in *Diluent* (from commercially available, NIST-traceable standard solution for lead). [NOTE—Prepare the *Standard stock solution* fresh every 2 months.]

Standard solution A: 10 µg/L of lead in *Blank solution* from *Standard stock solution*

Standard solution B: 1.0 µg/L of lead in *Blank solution* from *Standard solution A*

[NOTE—Prepare *Standard solution A* and *Standard solution B* fresh weekly.]