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Magnesium Hydroxide Paste

To view the Notice from the Expert Committee that posted in conjunction with this accelerated revision, please click 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 [$Mg(OH)_2$], 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 [$Mg(OH)_2$].▲ (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.]

Sample solution: Nominally 5.0 mg/mL of magnesium hydroxide prepared as follows. Accurately weigh an amount of Paste equivalent to 0.25 g of magnesium hydroxide. Cautiously add 3.0 mL of nitric acid, and mix until the sample is dissolved. Accurately transfer this solution to a 50-mL volumetric flask, and dilute with water to volume.

[NOTE—The concentrations specified in *Standard solution A*, *Standard solution B*, and *Sample solution* are recommended if an ICP–MS instrument is used. If an inductively coupled plasma–optical emission spectroscopy (ICP–OES) instrument is used, the concentrations may be modified to adapt to the working range of the instrument.]

Instrumental conditions

(See *Plasma Spectrochemistry ⟨730⟩*.)

Mode: Quadrupole ICP–MS or ICP–OES

ICP–MS analytical isotopes: 206, 207, and 208 amu for lead; 205 amu for thallium internal standard. [NOTE—The instrument should read all isotopes for lead (206, 207, and 208 amu) and the thallium internal standard (205 amu), and should report the total lead content using the sum of all three isotopes.]

ICP–OES analytical wavelength: 220.353 nm. [NOTE—To minimize matrix interference when using ICP–OES, it is recommended that the method of standard additions be used.]

System suitability

[NOTE—Instrument performance must be verified to conform to the manufacturer's specifications for resolution and sensitivity. Before analyzing samples, the instrument must pass a suitable performance check (see *Plasma Spectrochemistry ⟨730⟩, Qualification of Plasma Spectrophotometers, Performance Qualification*).]

Samples: *Blank solution*, *Standard solution A*, and *Standard solution B*

Suitability requirements

Linearity: Construct a linear calibration curve using the responses from the *Blank solution*, *Standard solution A*, and *Standard solution B*. The correlation coefficient is NLT 0.999.

Analysis: Aspirate the *Sample solution*, at least in duplicate, and calculate the amount of lead using the calibration curve obtained from the *System suitability*. Report the average reading as the lead content of the sample.

Calculate the content of lead in the portion of Paste taken.

Acceptance criteria: NMT 0.00015% (1.5 ppm), based on the content of magnesium hydroxide in the Paste, as determined in the *Assay*

SPECIFIC TESTS

• **MICROBIAL ENUMERATION TESTS ⟨61⟩** and **TESTS FOR SPECIFIED MICROORGANISMS ⟨62⟩:** The total aerobic microbial count does not exceed 4×10^2 cfu/g, and it meets the requirements of the test for absence of *Escherichia coli*.

ADDITIONAL REQUIREMENTS

• **PACKAGING AND STORAGE:** Preserve in tight containers.

Delete the following:

▲• **USP REFERENCE STANDARDS ⟨11⟩**

USP Calcium Carbonate RS

USP Magnesium Hydroxide RS▲ (RB 1-Dec-2022)

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

Topic/Question	Contact	Expert Committee
MAGNESIUM HYDROXIDE PASTE	Documentary Standards Support	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

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