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Magnesium Aluminometasilicate

DEFINITION

Magnesium Aluminometasilicate is a synthetic material that exists in two forms, Type I-A and Type I-B, having different pH requirements. The required contents for both forms are the same: NLT 29.1% and NMT 35.5% of aluminum oxide (Al_2O_3), NLT 11.4% and NMT 14.0% of magnesium oxide (MgO), and NLT 29.2% and NMT 35.6% of silicon dioxide (SiO_2), calculated on the dried basis.

IDENTIFICATION

- **A. Identification Tests—General, *Aluminum* ⟨191⟩**

  Sample solution: Transfer 0.5 g of Magnesium Aluminometasilicate to a suitable container, add 5 mL of a sulfuric acid solution (1 in 3), and heat until white fumes are observed. Cool, add 20 mL of water, and filter. Neutralize the filtrate with ammonia TS, and retain for use in *Identification* test *B*. Collect the precipitate, and dissolve in 3 N hydrochloric acid.

  Acceptance criteria: The *Sample solution* meets the requirements.

- **B. Identification Tests—General, *Magnesium* ⟨191⟩**

  Sample solution: Use the filtrate retained from *Identification* test *A*.

  Acceptance criteria: The *Sample solution* meets the requirements.

- **C.**

  Analysis: Prepare a bead by fusing a few crystals of sodium ammonium phosphate on a platinum loop in the flame of a Bunsen burner. Place the hot, transparent bead in contact with Magnesium Aluminometasilicate, and again fuse.

  Acceptance criteria: Silica floats about in the bead producing, upon cooling, an opaque bead with a web-like structure.

ASSAY

- **Aluminum Oxide**

  Edetate disodium titrant: Prepare a solution with a concentration of 18.6 g/L of edetate disodium in water and standardize as follows. Weigh 2 g of aluminum wire, transfer to a 1000-mL volumetric flask, and add 50 mL of a mixture of hydrochloric acid and water (1:1). Swirl the flask to ensure contact of the aluminum and the acid, and allow the reaction to proceed until all of the aluminum has dissolved. Dilute with water to volume. Pipet 10 mL of this solution into a 250-mL beaker and add, in the order named and with continuous stirring, 25.0 mL of *Edetate disodium titrant* and 20 mL of acetic acid–ammonium acetate buffer TS, and boil gently for 5 min. Cool, and add 50 mL of alcohol and 2 mL of dithizone TS. Titrate with 0.05 M zinc sulfate VS to a bright rose-pink color. Perform a blank determination, substituting 10 mL of water for the aluminum solution, and make any necessary correction.
  Calculate the molarity of the solution taken:

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

  W = weight of aluminum in the portion of solution taken (mg)

  A_r = atomic weight of aluminum, 26.98

  V = volume of *Edetate disodium titrant* consumed (mL)

  Sample solution: Transfer 1.25 g of Magnesium Aluminometasilicate to a conical flask, add 10 mL of 3 N hydrochloric acid and 50 mL of water, and heat on a water bath for 15 min. To this solution add 8 mL of hydrochloric acid, and heat on a water bath for 10 min. After cooling, transfer the solution to a 250-mL volumetric flask, rinse the conical flask with water, and add the washings to the volumetric flask. Dilute with water to volume. Centrifuge, and use the supernatant as the *Sample solution*. Retain a portion for use in the *Assay* for *Magnesium Oxide*.

  Analysis: Transfer 20.0 mL of the *Sample solution* to a beaker and add 20.0 mL of *Edetate disodium titrant*. To this solution add 15 mL of acetic acid–ammonium acetate buffer TS and 20 mL of water, and boil for 5 min. After cooling, add 50 mL of alcohol and 2 mL of dithizone TS, and titrate with 0.05 M zinc sulfate VS until the color of the solution changes from green-violet to rose-pink. Perform a blank determination. Each mL of 0.05 M *Edetate disodium titrant* is equivalent to 2.5490 mg of Al_2O_3.

  Acceptance criteria: 29.1%–35.5% of aluminum oxide (Al_2O_3) on the dried basis

- **Magnesium Oxide**

  Sample solution: Use the *Sample solution* prepared for use in the *Assay* for *Aluminum Oxide*.

Analysis: Transfer 50.0 mL of the *Sample solution* to a suitable container, add 50 mL of water and 25 mL of a trolamine solution (1 in 2), and shake well. Add 25 mL of ammonia–ammonium chloride buffer TS and 0.04 g of eriochrome black T trituration as the indicator. Titrate with 0.05 M edetate disodium VS until the red-purple color changes to blue and persists for 30 s. Each mL of 0.05 M edetate disodium VS is equivalent to 2.0152 mg of MgO.

Acceptance criteria: 11.4%–14.0% of magnesium oxide (MgO) on the dried basis

- **Silicon Dioxide**

Sample: 1 g

Analysis: To the *Sample* add 30 mL of 3 N hydrochloric acid, and evaporate on a water bath to dryness. Moisten the residue with hydrochloric acid, and evaporate again on a water bath to dryness. To the residue add 8 mL of hydrochloric acid and 25 mL of hot water, and stir. Allow to stand, and then decant the supernatant through an ashless filter paper. To the residue in the container add 10 mL of hot water, stir, and decant the supernatant through the filter paper. Wash the residue in the container with three additional 10-mL portions of hot water, stir, and decant as described above. Treat the residue in the container with 50 mL of water, and heat on a water bath for 15 min. Filter, and rinse the residue on the filter paper with hot water until no precipitate is obtained when 1 mL of silver nitrate TS is added to 5 mL of the washing. Transfer the filter paper and its contents to a tared platinum crucible, heat to dryness, incinerate, and continue to heat at 800 ± 25° for 1 h. Cool, and weigh. Moisten the residue with 6 mL of hydrofluoric acid, evaporate to dryness, and ignite for 5 min. Cool, and weigh. The loss in weight represents the weight of silicon dioxide (SiO_2).

Acceptance criteria: 29.2%–35.6% of silicon dioxide (SiO_2) on the dried basis

IMPURITIES

- **Chloride and Sulfate,** *Chloride* ⟨221⟩

Sample: A 20-mL portion of the diluted filtrate retained from the test for *Soluble Salts*

Control: 0.75 mL of 0.020 N hydrochloric acid

Acceptance criteria: NMT 0.053%; the *Sample* shows no more chloride than corresponds to the *Control*.

- **Chloride and Sulfate,** *Sulfate* ⟨221⟩

Sample: A 2-mL portion of the diluted filtrate retained from the test for *Soluble Salts*

Control: 0.5 mL of 0.020 N sulfuric acid

Acceptance criteria: NMT 0.480%; the *Sample* shows no more sulfate than corresponds to the *Control*.

Change to read:

- ▲**Arsenic** ⟨211⟩**,** *Procedures, Procedure 1*▲ (CN 1-Jun-2023)**:** NMT 3 µg/g

Change to read:

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

Sample solution: To 0.11 g of Magnesium Aluminometasilicate add 8 mL of 2 N nitric acid, boil for 1 min, and cool. Dilute with water to 100 mL, and centrifuge. Dilute 30 mL of the supernatant with water to 45 mL.

Acceptance criteria: NMT 0.03%

SPECIFIC TESTS

- **Acid-Consuming Capacity**

Sample solution: Transfer 0.2 g of Magnesium Aluminometasilicate to a glass-stoppered flask, and add 100.0 mL of 0.1 N hydrochloric acid VS. Stopper the flask tightly, shake at 37 ± 2° for 1 h, and filter. Use the filtrate.

Analysis: Transfer 50.0 mL of the *Sample solution* to a beaker, and while stirring, titrate the excess hydrochloric acid with 0.1 N sodium hydroxide VS to attain a pH of 3.5. Perform a blank determination.

Acceptance criteria: NLT 210 mL of 0.1 N hydrochloric acid is consumed per g of Magnesium Aluminometasilicate, calculated on the dried basis.

- **pH** ⟨791⟩

Sample: 2 g

Analysis: Add 50 mL of water to the *Sample*. While stirring, immerse the pH electrodes in the suspension, and after 2 min, record the pH.

Acceptance criteria

Type I-A: 6.5–8.5

Type I-B: 8.5–10.5

- **Loss on Drying** ⟨731⟩

Analysis: Dry at 110° for 7 h.

Acceptance criteria: NMT 20.0%

- **Soluble Salts**

Sample: 10.0 g

Analysis: Transfer the *Sample* to a suitable container, add 150 mL of water, and boil gently for 15 min, with shaking. After cooling, dilute with water to 150 mL, and centrifuge. Dilute 75 mL of the clear filtrate with water to 100 mL, and retain the diluted filtrate for use in the tests for *Alkalinity*, *Chloride*, and *Sulfate*. Evaporate 25 mL of the diluted filtrate on a water bath, and heat at 700° for 2 h.

Acceptance criteria: NMT 0.020 g (NMT 1.6%)

• **Alkalinity**

Sample: A 20-mL portion of the diluted filtrate retained from the test for *Soluble Salts*

Analysis: Add 2 drops of phenolphthalein TS to the *Sample*.

Acceptance criteria: If a pink color is produced, NMT 0.50 mL of 0.1 N hydrochloric acid is required to discharge it.

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers, and prevent exposure to excessive heat.

• **Labeling:** Label it to indicate whether it is Type I-A or Type I-B.

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

Topic/Question	Contact	Expert Committee
MAGNESIUM ALUMINOMETASILICATE	Documentary Standards Support	SE2020 Simple Excipients

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