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Magnesium Aluminosilicate

DEFINITION

Magnesium Aluminosilicate is a synthesized material that contains NLT 20.5% and NMT 27.7% of magnesium oxide (MgO), NLT 27.0% and NMT 34.3% of aluminum oxide (Al_2O_3), and NLT 14.4% and NMT 21.7% of silicon dioxide (SiO_2), calculated on the dried basis.

IDENTIFICATION

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

  Sample: 0.5 g

  Analysis: Transfer the *Sample* 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: Meets the requirements

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

  Sample solution: The filtrate retained from *Identification* test *A*

  Acceptance criteria: 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 Aluminosilicate, and again fuse.

  Acceptance criteria: The silica floats about in the bead, producing, upon cooling, an opaque bead with a weblike 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 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. 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 (g)

  A_r = atomic weight of aluminum, 26.98 g/mol

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

  Sample solution: Transfer 1.25 g of Magnesium Aluminosilicate 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*.

  [Note—Retain a portion of the *Sample solution* for use in the *Assay* for *Magnesium Oxide*.]

  Blank: 10 mL of 3 N hydrochloric acid and 50 mL of water

  Titrimetric system

  (See *Titrimetry* ⟨541⟩.)

  Mode: Residual titration

  Titrant: *Edetate disodium titrant*

  Back-titrant: 0.05 M zinc sulfate VS

  Endpoint detection: Visual

Analysis: Transfer 20.0 mL of the *Sample solution* to a beaker, and add 20.0 mL of *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 the *Back-titrant* until the color of the solution changes from green-violet to rose-pink. Perform a blank determination, and make the necessary correction. Each mL of 0.05 M *Edetate disodium titrant* is equivalent to 2.5490 mg of aluminum oxide (Al_2O_3).

Acceptance criteria: 27.0%–34.3% on the dried basis

- **Magnesium Oxide**

Sample solution: Use the portion retained from the *Sample solution* prepared in the *Assay* for *Aluminum Oxide*.

Titrimetric system

(See *Titrimetry* ⟨541⟩.)

Mode: Direct titration

Titrant: 0.05 M edetate disodium VS

Endpoint detection: Visual

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 (500 mg/mL), and shake well. Add 25 mL of ammonia–ammonium chloride buffer TS and 0.04 g of eriochrome black TS trituration as the indicator. Titrate with *Titrant* 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 magnesium oxide (MgO).

Acceptance criteria: 20.5%–27.7% 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 again evaporate 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, 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 SiO_2.

Acceptance criteria: 14.4%–21.7% on the dried basis

IMPURITIES

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

Analysis: A 20-mL portion of the diluted filtrate retained from the test for *Soluble Salts* shows no more chloride than corresponds to 0.75 mL of 0.020 N hydrochloric acid.

Acceptance criteria: NMT 0.053%

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

Analysis: A 2-mL portion of the diluted filtrate retained from the test for *Soluble Salts* shows no more sulfate than corresponds to 0.5 mL of 0.020 N sulfuric acid.

Acceptance criteria: NMT 0.480%

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: 0.11 g

Analysis: To the *Sample* 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 Aluminosilicate 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 filtrate from the *Sample solution* to a beaker, and while stirring, titrate the excess hydrochloric acid with 0.1 N sodium hydroxide VS to a pH of 3.5. Perform a blank determination, and make any necessary corrections.

Acceptance criteria: NLT 250 mL of 0.1 N hydrochloric acid is consumed per g of Magnesium Aluminosilicate, 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: 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 *Chloride*, *Sulfate*, and *Alkalinity*. Evaporate 25 mL of the diluted filtrate on a water bath, and heat at 700° for 2 h.

  Acceptance criteria: NMT 1.6%; the residue weighs NMT 0.020 g.

- **Alkalinity**

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

  Analysis: Add 2 drops of phenolphthalein TS to the *Sample*, containing 1 g of Magnesium Aluminosilicate.

  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.

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

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

Chromatographic Database Information: Chromatographic Database

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