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Magnesium Aluminum Silicate

DEFINITION

Magnesium Aluminum Silicate is a blend of colloidal montmorillonite and saponite that has been processed to remove grit and nonswellable ore components.

The requirements for viscosity and ratio of aluminum content to magnesium content differ for the several types of Magnesium Aluminum Silicate, as set forth in the table below.

Type	Viscosity (mPa · s)		Al Content/ Mg Content		Appearance
	Min.	Max.	Min.	Max.	
IA	225	600	0.5	1.2	Fine granules or flakes
IB	150	450	0.5	1.2	Microfine powder
IC	800	2200	0.5	1.2	Fine granules or flakes
IIA	100	300	1.4	2.8	Fine granules or flakes

IDENTIFICATION

• **A.**

Sample: 2 g

Analysis 1: Add the *Sample* in small portions to 100 mL of water with intense agitation. Allow to stand for 12 h to ensure complete hydration. Place 2 mL of the resulting mixture on a suitable glass slide, and allow to air-dry at room temperature to produce an oriented film. Place the slide in a vacuum desiccator over a free surface of ethylene glycol. Evacuate the desiccator, and close the stopcock so that the ethylene glycol saturates the desiccator chamber. Allow to stand for 12 h. Record the X-ray diffraction pattern (see *X-Ray Powder Diffraction* ⟨941⟩), and calculate the *d* values.

Acceptance criteria 1: The largest peak corresponds to a *d* value between 15.0 and 17.2 Å.

Analysis 2: Prepare a random powder specimen of Magnesium Aluminum Silicate, record the X-ray diffraction pattern, and determine the *d* values in the region between 1.48 and 1.54 Å.

Acceptance criteria 2: Peaks are found at 1.492–1.504 Å and at 1.510–1.540 Å.

• **B.** It meets the requirements of the test for *Viscosity* in *Specific Tests*.

• **C.** It meets the requirements for *Ratio of aluminum content to magnesium content* in the test for *Aluminum Content and Magnesium Content*.

• **D.** Its appearance corresponds to the description in the *table* in the *Definition*.

ASSAY

• **Aluminum Content and Magnesium Content**

Aluminum content

Aluminum standard stock solution: Dissolve 1.000 g of aluminum in a mixture of 10 mL of hydrochloric acid and 10 mL of water by gentle heating. Transfer the solution to a 1000-mL volumetric flask, dilute with water to volume, and mix. This solution contains the equivalent of 1 mg/mL of aluminum.

Aluminum standard solutions: Transfer 2-, 5-, and 10-mL aliquots of the *Aluminum standard stock solution* to separate 100-mL volumetric flasks containing 200 mg of sodium chloride, dilute each with water to volume, and mix.

Sample stock solution: Transfer 0.200 g of Magnesium Aluminum Silicate to a 25-mL platinum crucible containing 1.0 g of lithium metaborate, and mix. Using a muffle furnace or a suitable burner, heat slowly at first, and ignite at 1000°–1200° for 15 min. Cool, place the crucible in a 100-mL beaker containing 25 mL of dilute nitric acid (50 mg/mL), and add an additional 50 mL of the dilute acid, filling and submerging the upright crucible. Place a polyfluorocarbon-coated magnetic stirring bar into the crucible, and stir gently with a

magnetic stirrer to dissolve. Pour the contents into a 250-mL beaker, and remove the crucible. Warm the solution, transfer through a rapid-flow filter paper with the aid of water into a 200-mL volumetric flask, dilute with water to volume, and mix.

Sample solution: Pipet 20 mL of the *Sample stock solution* into a 100-mL volumetric flask. Add 20 mL of a solution of sodium chloride (10 mg/mL), dilute with water to volume, and mix.

Instrumental conditions

(See *Atomic Absorption Spectroscopy* ⟨852⟩.)

Mode: Atomic absorption spectrophotometer equipped with a single-slot burner

Analytical wavelength: 309 nm

Lamp: Aluminum hollow-cathode

Flame: Oxidizing acetylene–air–nitrous oxide

Analysis

Samples: *Aluminum standard solutions* and *Sample solution*

Determine the absorbances of the *Aluminum standard solutions* and the *Sample solution*. From a linear regression equation calculated from the absorbances and concentrations of the *Aluminum standard solutions*, determine the aluminum content in the magnesium aluminum silicate.

Magnesium content

Lanthanum solution: Stir 88.30 g of lanthanum chloride ($LaCl_3$) with 500 mL of 6 N hydrochloric acid to dissolve, transfer with the aid of water to a 1000-mL volumetric flask, dilute with water to volume, and mix.

Magnesium standard stock solution: Place 1.000 g of magnesium in a 250-mL beaker containing 20 mL of water, and carefully add 20 mL of hydrochloric acid, warming, if necessary, to complete the reaction. Transfer the solution to a 1000-mL volumetric flask, dilute with water to volume, and mix. This solution contains the equivalent of 1 mg/mL of magnesium. Transfer 10.0 mL of this solution to a 1000-mL volumetric flask, dilute with water to volume, and mix.

Magnesium standard solutions: Transfer 5-, 10-, 15-, and 20-mL aliquots of the *Magnesium standard stock solution* to separate 100-mL volumetric flasks. To each flask add 20.0 mL of *Lanthanum solution*, dilute with water to volume, and mix.

Sample stock solution: Use the *Sample stock solution* prepared as directed for *Aluminum content*.

Sample solution: Transfer 25 mL of the *Sample stock solution* to a 50-mL volumetric flask, dilute with water to volume, and mix. Transfer 5.0 mL of this solution to a 100-mL volumetric flask, add 20.0 mL of *Lanthanum solution*, dilute with water to volume, and mix.

Instrumental conditions

(See *Atomic Absorption Spectroscopy* ⟨852⟩.)

Mode: Atomic absorption

Analytical wavelength: 285 nm

Lamp: Magnesium hollow-cathode

Flame: Reducing flame of acetylene–air

Analysis

Samples: *Magnesium standard solutions* and *Sample solution*

Determine the absorbances of the *Sample solution* and the *Magnesium standard solutions*. From a linear regression equation calculated from the absorbances and concentrations of the *Magnesium standard solutions*, determine the magnesium content in the magnesium aluminum silicate.

Ratio of aluminum content to magnesium content

Analysis: Using the results from the *Aluminum content* and the *Magnesium content*, determine the ratio of aluminum content to magnesium content.

Acceptance criteria

Type IA: 0.5–1.2

Type IB: 0.5–1.2

Type IC: 0.5–1.2

Type IIA: 1.4–2.8

IMPURITIES

Change to read:

- ▲**Arsenic** ⟨211⟩, *Procedures, Procedure 1*▲ (CN 1-Jun-2023)

Standard preparation: Transfer 5.0 mL (5 µg of arsenic) of the *Standard Arsenic Solution* to a 25-mL volumetric flask, and add dilute hydrochloric acid (1:25) to volume.

Test preparation: Transfer 13.3 g of Magnesium Aluminum Silicate to a 250-mL beaker containing 100 mL of dilute hydrochloric acid (1:25), mix, cover with a watch glass, and boil gently with occasional stirring for 15 min without allowing excessive foaming. Allow the insoluble material to settle, and decant the hot supernatant through a rapid-flow filter paper into a 200-mL volumetric flask, retaining as much sediment as possible in the beaker. Add 25 mL of hot dilute hydrochloric acid (1:25) to the residue in the beaker, stir, and heat to boiling. Allow the insoluble material to settle, and decant the supernatant through the filter into the 200-mL volumetric flask. Repeat the extraction with four additional 25-mL portions of hot dilute hydrochloric acid (1:25), decanting each hot supernatant through the filter into the volumetric flask. At the last extraction, transfer as much of the insoluble material as possible onto the filter. Cool the combined filtrates to room temperature, add dilute hydrochloric acid (1:25) to volume, and mix. Use 25 mL for the test.

Acceptance criteria: NMT 3 µg/g; the absorbance due to any red color from the *Test preparation* does not exceed that produced by the *Standard preparation*.

• **Lead**

Standard preparation: On the day of use, dilute 3.0 mL of lead nitrate stock solution TS with water to 100 mL. Each mL contains the equivalent of 3 µg of lead.

Sample: 10.0 g

Test preparation: Transfer the *Sample* to a 250-mL beaker containing 100 mL of dilute hydrochloric acid (1:25), stir, cover with a watch glass, and boil for 15 min. Cool to room temperature, and allow the insoluble matter to settle. Decant the supernatant through a rapid-flow filter paper into a 400-mL beaker. Add 25 mL of hot water to the insoluble matter in the 250-mL beaker, and stir. Allow the insoluble matter to settle, and decant the supernatant through the filter into the 400-mL beaker. Repeat the extraction with two additional 25-mL portions of water, decanting each supernatant portion through the filter into the 400-mL beaker. Wash the filter with 25 mL of hot water, collecting this filtrate in the 400-mL beaker. Concentrate the combined extracts by gentle boiling to approximately 20 mL. If a precipitate appears, add 2–3 drops of nitric acid, heat to boiling, and cool to room temperature. Filter the concentrated extracts through a rapid-flow filter paper into a 50-mL volumetric flask. Transfer the remaining contents of the 400-mL beaker through the filter paper and into the flask with water. Dilute with water to volume.

Instrumental conditions

(See *Atomic Absorption Spectroscopy* ⟨852⟩.)

Mode: Atomic absorption spectrophotometer equipped with a deuterium arc background correction and a single-slot burner

Analytical wavelength: 284 nm

Lamp: Lead hollow-cathode

Flame: Oxidizing flame of air and acetylene

Acceptance criteria: The absorbance of the *Test preparation* is NMT that of the *Standard preparation* (15 µg/g).

SPECIFIC TESTS

• **Microbial Enumeration Tests** ⟨61⟩ and **Tests for Specified Microorganisms** ⟨62⟩**:** Its total aerobic microbial count does not exceed 10^3 cfu/g, and it meets the requirements of the test for absence of *Escherichia coli*.

• **pH** ⟨791⟩

Sample suspension: 50 mg/mL

Acceptance criteria: 9.0–10.0

• **Loss on Drying** ⟨731⟩

Analysis: Dry at 110° to constant weight.

Acceptance criteria: NMT 8.0%

• **Viscosity**

Sample: After determining the *Loss on Drying*, weigh a quantity of Magnesium Aluminum Silicate, equivalent to 25.0 g on the dried basis. Over a period of a few seconds, transfer the undried test specimen to a suitable 1-L blender jar containing an amount of water, maintained at a temperature of 25 ± 2°, that is sufficient to produce a mixture weighing 500 g. Blend for 3 min, accurately timed, at 14,000–15,000 rpm (high speed).[1]

[Note—Heat generated during blending causes a temperature rise to above 30°.]

Analysis: Transfer the contents of the blender to a 600-mL beaker, and allow to stand for 5 min. The sample temperature should be 33 ± 3°.

Using a suitable rotational viscometer[2] equipped with a spindle as specified below, operate the viscometer at 60 rpm for 6 min, accurately timed, and record the scale reading.

For Type IA, use a spindle with a cylinder 1.87 cm in diameter and 0.69 cm high attached to a shaft 0.32 cm in diameter, the distance from the top of the cylinder to the lower tip of the shaft being 2.54 cm, and the immersion depth being 5.00 cm (No. 2 spindle). If the scale reading is greater than 90% of full scale, repeat the measurement, using a spindle similar to the No. 2 spindle but with the cylinder 1.27 cm in diameter and 0.16 cm high instead (No. 3 spindle).

For Type IC, use a No. 3 spindle. If the scale reading is greater than 90% of full scale, repeat the measurement using a spindle consisting of a cylindrical shaft 0.32 cm in diameter and with an immersion depth of 4.05 cm (No. 4 spindle).

For Types IB and IIA, use a No. 2 spindle.

Acceptance criteria

Type IA: 225–600 mPa · s

Type IB: 150–450 mPa · s

Type IC: 800–2200 mPa · s

Type IIA: 100–300 mPa · s

• **Acid Demand**

Sample: After determining the *Loss on Drying*, weigh a quantity of Magnesium Aluminum Silicate equivalent to 5.00 g.

Analysis: Disperse the *Sample* in 500 mL of water with the aid of a suitable blender fitted with a 1-L jar. Using a stopwatch, designate zero time. With constant mixing, add 3.0-mL portions of 0.100 N hydrochloric acid at 5, 65, 125, 185, 245, 305, 365, 425, 485, 545, 605, 665, and 725 s, and add a 1.0-mL portion at 785 s. Determine the pH potentiometrically at 840 s.

Acceptance criteria: NMT 4.0

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers.

• **Labeling:** Label it to indicate its type.

[1] A suitable blender is available from Waring as Waring Commercial Blender Model 7009G or equivalent with 1-L glass jar and tachometer adapter, Model CAC24 or equivalent.

[2] A suitable viscometer is available from Brookfield as Viscometer Model LVF or LVT, or equivalent.

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

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

Chromatographic Database Information: Chromatographic Database

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