

monium chloride buffer TS and 0.04 g of eriochrome black TS trituration as the indicator. Titrate with 0.05 M edetate disodium VS until the red-purple color changes to blue and persists for 30 seconds. Each mL of 0.05 M edetate disodium VS is equivalent to 2.0152 mg of MgO.

**Assay for silicon dioxide**—Transfer about 1 g of Magnesium Aluminometasilicate, accurately weighed, to a suitable container, 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, 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 minutes. 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  $800 \pm 25^\circ$  for 1 hour. Cool, and weigh. Moisten the residue with 6 mL of hydrofluoric acid, evaporate to dryness, and ignite for 5 minutes. Cool, and weigh. The loss in weight represents the weight of silicon dioxide ( $\text{SiO}_2$ ).

### Magnesium Aluminosilicate

» Magnesium Aluminosilicate is a synthesized material that contains not less than 20.5 percent and not more than 27.7 percent of magnesium oxide ( $\text{MgO}$ ), not less than 27.0 percent and not more than 34.3 percent of aluminum oxide ( $\text{Al}_2\text{O}_3$ ), and not less than 14.4 percent and not more than 21.7 percent of silicon dioxide ( $\text{SiO}_2$ ), calculated on the dried basis.

**Packaging and storage**—Preserve in tight containers, and prevent exposure to excessive heat.

**Acid-consuming capacity**—Transfer about 0.2 g of Magnesium Aluminosilicate, accurately weighed, to a glass-stoppered flask, and add 100.0 mL of 0.1 N hydrochloric acid VS. Stopper the flask tightly, shake at  $37 \pm 2^\circ$  for 1 hour, and filter. Transfer 50.0 mL of the filtrate 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, and make any necessary correction: not less than 250 mL of 0.1 N hydrochloric acid is consumed per g of Magnesium Aluminosilicate, calculated on the dried basis.

**pH** (791)—Transfer 2 g of Magnesium Aluminosilicate to a suitable container, and add 50 mL of water. While stirring, immerse the pH electrodes in the suspension, and after 2 minutes record the pH: between 8.5 and 10.5.

**Other requirements**—It meets the requirements for *Identification*, *Loss on drying*, *Soluble salts*, *Alkalinity*, *Chloride*, *Sulfate*, *Arsenic*, *Iron*, and *Heavy metals* under *Magnesium Aluminometasilicate*.

**Assay for aluminum oxide, magnesium oxide, and silicon dioxide**—Proceed as directed in the *Assay for aluminum oxide*, the *Assay for magnesium oxide*, and the *Assay for silicon dioxide* under *Magnesium Aluminometasilicate*.

### Magnesium Aluminum Silicate

» 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 accompanying table.

Type	Viscosity (cps)		Al content/ Mg content	
	Min.	Max.	Min.	Max.
IA	225	600	0.5	1.2
IB	150	450	0.5	1.2
IC	800	2200	0.5	1.2
IIA	100	300	1.4	2.8

**Packaging and storage**—Preserve in tight containers.

**Labeling**—Label it to indicate its type.

**Identification**—Add 2 g in small portions to 100 mL of water, with intense agitation. Allow to stand for 12 hours 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 hours. Record the X-ray diffraction pattern (see *X-ray Diffraction* (941)), and calculate the  $d$  values: the largest peak corresponds to a  $d$  value between 15.0 and 17.2 angstrom units. 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 angstrom units: peaks are found between 1.492 and 1.504 angstrom units and between 1.510 and 1.540 angstrom units.

**Viscosity**—After determining the *Loss on drying*, weigh a quantity of Magnesium Aluminum Silicate test specimen 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  $25 \pm 2^\circ$ , that is sufficient to produce a mixture weighing 500 g. Blend for 3 minutes, accurately timed, at 14,000 to 15,000 rpm (high speed). [NOTE—Heat generated during blending causes a temperature rise to above  $30^\circ$ .] Transfer the contents of the blender to a 600-mL beaker, allow to stand for 5 minutes, and adjust, if necessary, to a temperature of  $33 \pm 3^\circ$ . Using a suitable rotational viscosimeter equipped with a spindle as specified below, operate the viscosimeter at 60 rpm for 6 minutes, accurately timed, and record the scale reading. For Type IA, use a spindle having 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 having 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 having an immersion depth of 4.05 cm (No. 4 spindle). For Types IB and IIA, use a No. 2 spindle.

**Microbial enumeration tests** (61) and **Tests for specified microorganisms** (62)—Its total aerobic microbial count does not exceed 1000 cfu per g, and it meets the requirements of the test for absence of *Escherichia coli*.

**pH** (791): between 9.0 and 10.0, in a suspension (5 in 100) in water.

**Loss on drying** (731)—Dry it at 110° to constant weight: it loses not more than 8.0% of its weight.

**Acid demand**—After determining the *Loss on drying*, weigh a quantity of Magnesium Aluminum Silicate equivalent to 5.00 g, and disperse in 500 mL of water with the aid of a suitable blender fitted with a 1-liter 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 seconds, and add a 1.0-mL portion at 785 seconds. Determine the pH potentiometrically at 840 seconds: the pH is not more than 4.0.

**Arsenic, Method I** (211)—Prepare the *Test Preparation* as follows. Transfer 13.3 g to a 250-mL beaker containing 100 mL of dilute hydrochloric acid (1 in 25), mix, cover with a watch glass, and boil gently, with occasional stirring, for 15 minutes 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 in 25) to the residue in the beaker, stir, 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 in 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 in 25) to volume, and mix.

*Procedure*—Use a 25-mL aliquot of *Test Preparation* for the *Procedure*. The absorbance due to any red color from the *Test Preparation* does not exceed that produced by 5.0 mL of *Standard Preparation* (5 µg of As) when treated with the same quantities of the same reagents and in the same manner. The limit is 3 ppm.

**Lead**—[NOTE—The *Standard preparation* and *Test preparation* may be modified if necessary, to obtain solutions, of suitable concentrations, adaptable to the linear or working range of the instrument.]

*Standard preparation*—On the day of use, dilute 3.0 mL of *Lead Nitrate Stock Solution* (see *Heavy Metals* (231)) with water to 100 mL. Each mL of the *Standard preparation* contains the equivalent of 3 µg of lead.

*Test preparation*—Transfer 10.0 g of Magnesium Aluminum Silicate to a 250-mL beaker containing 100 mL of dilute hydrochloric acid (1 in 25), stir, cover with a watch glass, and boil for 15 minutes. 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, 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 to 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, and mix.

*Procedure*—Determine the absorbances of the *Test preparation* and the *Standard preparation* at 284 nm in a suitable atomic absorption spectrophotometer equipped with a lead hollow-cathode lamp, deuterium arc background correction, and a single-slot burner, using an oxidizing flame of air and acetylene. The absorbance of the *Test preparation* is not greater than that of the *Standard preparation* (0.0015%).

**Assay for aluminum and magnesium content**—[NOTE—The *Standard preparations* and *Assay preparations* may be diluted quantitatively with water, if necessary, to obtain solutions, of suitable concentrations, adaptable to the linear or working range of the instrument.]

*Lanthanum solution*—Stir 88.30 g of lanthanum chloride (LaCl<sub>3</sub>) with 500 mL of 6 N hydrochloric acid until solution is complete, transfer with the aid of water to a 1000-mL volumetric flask, and dilute with water to volume.

*Specimen preparation*—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° to 1200° for 15 minutes. Cool, place the crucible in a 100-mL beaker containing 25 mL of dilute nitric acid (1 in 20), 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 until solution is complete. Pour the contents into a 250-mL beaker, and remove the crucible. Warm the solution, and 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.

*Aluminum standard preparations*—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 of aluminum per mL. Transfer 2-, 5-, and 10-mL aliquots to separate 100-mL volumetric flasks containing 200 mg of sodium chloride, dilute each with water to volume, and mix.

*Aluminum assay preparation*—Pipet 20 mL of *Specimen preparation* into a 100-mL volumetric flask. Add 20 mL of a solution of sodium chloride (1 in 100), dilute with water to volume, and mix.

*Procedure for aluminum*—In a suitable atomic absorption spectrophotometer equipped with an aluminum hollow-cathode lamp and a single-slot burner, using an oxidizing acetylene-air-nitrous oxide flame, determine the absorbances of the *Aluminum assay preparation* and each of the *Aluminum standard preparations* at 309 nm. From a linear regression equation calculated from the absorbances and concentrations of the *Aluminum standard preparations*, determine the aluminum content of the Magnesium Aluminum Silicate.

*Magnesium standard preparations*—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 of magnesium per mL. Transfer 10.0 mL of this solution to a 1000-mL volumetric flask, dilute with water to volume, and mix. Transfer 5-, 10-, 15-, and 20-mL aliquots to separate 100-mL volumetric flasks. To each flask add 20.0 mL of *Lanthanum solution*, dilute with water to volume, and mix.

*Magnesium assay preparation*—Transfer a 25-mL aliquot of *Specimen preparation* to a 50-mL volumetric flask, dilute with water to volume, and mix. Transfer a 5.0-mL aliquot of this dilution to a 100-mL volumetric flask, add 20.0 mL of *Lanthanum solution*, dilute with water to volume, and mix.

*Procedure for magnesium*—In a suitable atomic absorption spectrophotometer equipped with a magnesium hollow-cathode lamp and a single-slot burner, using a reducing acetylene-air flame, determine the absorbances of the *Magnesium assay preparation* and each of the *Magnesium standard preparations* at 285 nm. From a linear regression equation calculated from the absorbances and concentrations of the *Magnesium standard preparations*, determine the magnesium content of the Magnesium Aluminum Silicate.

## Magnesium Silicate

» Magnesium Silicate is a compound of magnesium oxide and silicon dioxide. It contains not less than 15.0 percent of magnesium oxide (MgO) and not less than 67.0 percent of silicon dioxide (SiO<sub>2</sub>), calculated on the ignited basis.

**Packaging and storage**—Preserve in well-closed containers.

### Identification—

**A:** Mix about 500 mg with 10 mL of 3 N hydrochloric acid, filter, and neutralize the filtrate to litmus paper with 6 N ammonium hydroxide: the neutralized filtrate responds to the tests for *Magnesium* (191).

**B:** 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 Silicate, and again fuse: silica floats about in the bead, producing, upon cooling, an opaque bead with a web-like structure.

**pH** (791): between 7.0 and 10.8, determined in a well-mixed aqueous suspension (1 in 10).

**Loss on drying** (731)—Dry it at 105° for 2 hours: it loses not more than 15.0% of its weight. (Retain the dried specimen for the test for *Loss on ignition*.)

**Loss on ignition** (733)—Ignite the specimen retained from the test for *Loss on drying* at 900° to 1000° for 20 minutes: the previously dried specimen loses not more than 15% of its weight.

**Soluble salts**—Boil 10.0 g with 150 mL of water for 15 minutes. Cool to room temperature, allow the mixture to stand for 15 minutes, filter with the aid of suction, transfer the filtrate to a 200-mL volumetric flask, dilute with water to volume, and mix. Evaporate 50.0 mL of this solution, representing 2.5 g of the Silicate, in a tared platinum dish to dryness, and ignite gently to constant weight: the weight of the residue does not exceed 75.0 mg (3.0%).

### Fluoride—

**Indicator solution**—Prepare a solution containing 100 mg of lanthanum alizarin complexan mixture per mL in 60% isopropyl alcohol. Filter the solution if it is not clear.

**Test preparation**—Prepare a slurry consisting of 5.0 g of Magnesium Silicate and 45 mL of 0.1 N hydrochloric acid, stir at room temperature for 15 minutes, and pass through a 0.45- $\mu$ m filter into a 50-mL volumetric flask. Wash the filter with five 1-mL portions of 0.1 N hydrochloric acid, collecting the washings in the flask, dilute with 0.1 N hydrochloric acid to volume, and mix.

**Procedure**—Transfer 5.0 mL of the *Test preparation* to a 25-mL volumetric flask, add 5.0 mL of *Indicator solution*, dilute with water to volume, mix, and allow to stand for 1 hour in diffuse light at ambient temperature. Determine the absorbance of this solution in a 1-cm cell with a suitable spectrophotometer, at the wavelength of maximum absorbance at about 620 nm, against a blank consisting of 5.0 mL of 0.1 N hydrochloric acid, 5.0 mL of *Indicator solution*, and 15.0 mL of water. The absorbance is not greater than that produced by 5.0 mL of a solution containing 2.21  $\mu$ g of sodium fluoride per mL of 0.1 N hydrochloric acid, when treated in the same manner as the *Test preparation* (10 ppm).

**Free alkali**—Add 2 drops of phenolphthalein TS to 20 mL of the diluted filtrate prepared in the test for *Soluble salts*, representing 1 g of Magnesium Silicate: if a pink color is produced, not more than 2.5 mL of 0.1 N hydrochloric acid is required to discharge it.

**Lead** (251)—Dissolve 1.0 g in 20 mL of 3 N hydrochloric acid, evaporate on a steam bath to about 10 mL, dilute with water to about 20 mL, and cool: the limit is 0.001%.

**Ratio of SiO<sub>2</sub> to MgO**—Divide the percentage of SiO<sub>2</sub> obtained in the *Assay for silicon dioxide* by the percentage of MgO obtained in the *Assay for magnesium oxide*: the quotient obtained is between 2.50 and 4.50.

**Heavy metals** (231)—Boil 4.0 g with a mixture of 50 mL of water and 10 mL of hydrochloric acid for 20 minutes, adding water to maintain the volume during the boiling. Add ammonium hydroxide until the mixture is only slightly acid to litmus paper. Filter with the aid of suction, and wash with 15 to 20 mL of water, combining the washings with the original filtrate. Add 2 drops of phenolphthalein TS, then add a slight excess of 6 N ammonium hydroxide. Discharge the pink color with dilute hydrochloric acid (1 in 100), then add 8 mL of dilute hydrochloric acid (1 in 100). Dilute with water to 100 mL, and use 25 mL of the solution for the test: the limit is 20  $\mu$ g per g.

**Assay for magnesium oxide**—Weigh accurately about 1.5 g, and transfer to a 250-mL conical flask. Add 50.0 mL of 1 N sulfuric acid VS, and digest on a steam bath for 1 hour. Cool to room temperature, add methyl orange TS, and titrate the excess acid with 1 N sodium hydroxide VS. Each mL of 1 N sulfuric acid is equivalent to 20.15 mg of MgO.

**Assay for silicon dioxide**—Transfer about 700 mg of Magnesium Silicate, accurately weighed, to a small platinum dish. Add 10 mL of 1 N sulfuric acid, and heat on a steam bath to dryness, leaving the dish uncovered. Treat the residue with 25 mL of water, and digest on a steam bath for 15 minutes. Decant the supernatant through an ashless filter paper, with the aid of suction, and wash the residue, by decantation, three times with hot water, passing the washings through the filter paper. Finally transfer the residue to the filter, and wash thoroughly with hot water. Transfer the filter paper and its contents to the platinum dish previously used. Heat to dryness, incinerate, ignite strongly for 30 minutes, cool, and weigh. Moisten the residue with water, and add 6 mL of hydrofluoric acid and 3 drops of sulfuric acid. Evaporate to dryness, ignite for 5 minutes, cool, and weigh: the loss in weight represents the weight of SiO<sub>2</sub>.

## Magnesium Stearate

Portions of the monograph text that are national *USP* text, and are not part of the harmonized text, are marked with symbols (\*,\*) to specify this fact.

Octadecanoic acid, magnesium salt;  
Magnesium stearate [557-04-0].

### DEFINITION

Magnesium Stearate is a compound of magnesium with a mixture of solid organic acids, and consists chiefly of variable proportions of magnesium stearate and magnesium palmitate. The fatty acids are derived from edible sources. It contains NLT 4.0% and NMT 5.0% of Mg, calculated on the dried basis.

### IDENTIFICATION

#### • A. IDENTIFICATION TESTS—GENERAL, *Magnesium* (191)

**Sample solution:** Mix 5.0 g with 50 mL of peroxide-free ether, 20 mL of diluted nitric acid, and 20 mL of water in a round-bottom flask. Connect the flask to a reflux condenser, and reflux until dissolution is complete. Allow to cool, and transfer the contents of the flask to a separator. Shake, allow the layers to separate, and transfer the aqueous layer to a flask. Extract the ether layer with two 4-mL portions of water, and add these aqueous extracts to the main aqueous extract. Wash the aqueous extract with 15 mL of peroxide-free ether, transfer the aqueous extract to a 50-mL volumetric flask, and dilute with water to volume. Retain the unused portion of this solution for the chloride and sulfate impurity tests.

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

#### • B. The retention times of the peaks corresponding to stearic acid and palmitic acid of the *Sample solution* correspond to