

Total ash <561>: not more than 0.1%.

Heavy metals, Method II <231>: not more than 0.001%.

Alkaline impurities—Weigh 5.0 g of Linoleoyl Polyoxylglycerides, add 10 mL of alcohol and 0.05 mL of bromophenol blue TS, and mix well. Titrate with 0.01 N hydrochloric acid VS to change the color to yellow: not more than 1.0 mL of 0.01 N hydrochloric acid is required.

Limit of free ethylene oxide and dioxane—Proceed as directed in the test for *Limit of free ethylene oxide and dioxane* under *Caprylocaproyl Polyoxylglycerides*: not more than 1 µg of ethylene oxide per g is found; and not more than 10 µg of dioxane per g is found.

Limit of free glycerol—Proceed as directed in the test for *Limit of free glycerol* under *Caprylocaproyl Polyoxylglycerides*: not more than 5.0% is found.

Magnesium Aluminometasilicate

» 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: not less than 29.1 percent and not more than 35.5 percent of aluminum oxide (Al_2O_3), not less than 11.4 percent and not more than 14.0 percent of magnesium oxide (MgO), and not less than 29.2 percent and not more than 35.6 percent of silicon dioxide (SiO_2), calculated on the dried basis.

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.

Identification—

A: 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: the solution meets the requirements of the tests for *Aluminum* <191>.

B: The filtrate retained from *Identification* test A meets the requirements of the tests for *Magnesium* <191>.

C: 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: silica floats about in the bead producing, upon cooling, an opaque bead with a web-like structure.

Acid-consuming capacity—Transfer about 0.2 g of Magnesium Aluminometasilicate, 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 210 mL of 0.1 N hydrochloric acid is consumed per g of Magnesium Aluminometasilicate, calculated on the dried basis.

pH <791>—Transfer 2 g of Magnesium Aluminometasilicate 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 6.5 and 8.5 for Type I-A, and between 8.5 and 10.5 for Type I-B.

Loss on drying <731>—Dry it at 110° for 7 hours: it loses not more than 20.0% of its weight.

Soluble salts—Transfer 10.0 g of Magnesium Aluminometasilicate to a suitable container, add 150 mL of water, and boil gently for 15 minutes, 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 hours. The residue weighs not more than 0.020 g: not more than 1.6% is found.

Alkalinity—Add 2 drops of phenolphthalein TS to 20 mL of the diluted filtrate retained from the test for *Soluble salts*, containing 1 g of Magnesium Aluminometasilicate: if a pink color is produced, not more than 0.50 mL of 0.1 N hydrochloric acid is required to discharge it.

Chloride <221>—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: not more than 0.053% is found.

Sulfate <221>—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: not more than 0.480% is found.

Arsenic, Method I <211>: 3 µg per g.

Iron <241>—

Test Preparation—To 0.11 g of Magnesium Aluminometasilicate add 8 mL of 2 N nitric acid, boil for 1 minute, and cool. Dilute with water to 100 mL, and centrifuge. Dilute 30 mL of the supernatant with water to 45 mL: the limit is 0.03%.

Heavy metals, Method I <231>—

Test Preparation—Transfer 2.67 g of Magnesium Aluminometasilicate to a suitable container, add 20 mL of water and 8 mL of hydrochloric acid, and evaporate to dryness on a water bath. To the residue add 5 mL of 1 N acetic acid and 20 mL of water, boil for 2 minutes, add 0.4 g of hydroxylamine hydrochloride, and heat to boiling. Cool, dilute with water to 100 mL, and filter. Use 25 mL of the filtrate as the *Test Preparation*.

Monitor Preparation—Transfer another 25 mL of the diluted filtrate to a suitable container, and add 2.0 mL of *Standard Lead Solution*.

Standard Preparation—Transfer 2 mL of hydrochloric acid to a suitable container, and evaporate to dryness on a water bath. To the residue add 2.0 mL of *Standard Lead Solution* and 0.1 g of hydroxylamine hydrochloride. Dilute with water to 25 mL: the limit is 30 µg per g.

Assay for aluminum oxide—

Edetate disodium titrant—Prepare and standardize as directed in the Assay under *Ammonium Alum*.

Assay preparation—Transfer about 1.25 g of Magnesium Aluminometasilicate, accurately weighed, 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 minutes. To this solution add 8 mL of hydrochloric acid, and heat on a water bath for 10 minutes. 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, and mix. Centrifuge, and use the supernatant as the *Assay preparation*. Retain a portion of the *Assay preparation* for use in the *Assay for magnesium oxide*.

Procedure—Transfer 20.0 mL of the *Assay preparation* 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 minutes. 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, and make any necessary correction. Each mL of 0.05 M *Edetate disodium titrant* is equivalent to 2.5490 mg of Al_2O_3 .

Assay for magnesium oxide—Transfer 50.0 mL of the *Assay preparation* retained from the *Assay for aluminum oxide* to a suitable container, add 50 mL of water and 25 mL of a triamine solution (1 in 2), and shake well. Add 25 mL of ammonia–am-

monium chloride buffer TS and 0.04 g of eriochrome black TS titration 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 (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 (MgO), not less than 27.0 percent and not more than 34.3 percent of aluminum oxide (Al_2O_3), and not less than 14.4 percent and not more than 21.7 percent of silicon dioxide (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° .] 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*.