

substituting 10 mL of water for the *Assay preparation*, and make any necessary correction. Each mL of 0.05 M *Eдетate disodium titrant* consumed is equivalent to 3.900 mg of Al(OH)<sub>3</sub>.

#### Assay for magnesium trisilicate—

*Potassium chloride solution*—Prepare a solution in water containing 5 g of potassium chloride per 100 mL.

*Magnesium standard solution*—Transfer 1.000 g of magnesium metal to a 1000-mL volumetric flask containing 50 mL of water, and slowly add 10 mL of hydrochloric acid. Dilute with water to volume, and mix. Transfer 5.0 mL of this solution to a 500-mL volumetric flask, dilute with water to volume, and mix.

*Standard preparations*—Transfer 16.0 mL, 18.0 mL, and 20.0 mL of *Magnesium standard solution* to separate 100-mL volumetric flasks, add 2.0 mL of *Potassium chloride solution* to each flask, dilute with water to volume, and mix. These *Standard preparations* contain 1.6, 1.8, and 2.0 μg of magnesium per mL, respectively. [NOTE—Prepare these solutions on the day of use.]

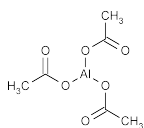
*Assay preparation*—Weigh and finely powder not fewer than 20 Tablets. Transfer an accurately weighed portion of the powder, equivalent to about 5 mg of magnesium trisilicate, to a 100-mL volumetric flask, and add 10 mL of 18 N sulfuric acid. Heat on a steam bath for 30 minutes with occasional swirling. Allow to cool, dilute with water to volume, and mix. Filter this solution, discarding the first 20 mL of the filtrate. Transfer 20.0 mL of the filtrate to a second 100-mL volumetric flask, add 2.0 mL of *Potassium chloride solution*, dilute with water to volume, and mix.

*Procedure*—Concomitantly determine the absorbance of the *Standard preparations* and the *Assay preparation* at the magnesium emission line at 285.2 nm, with an atomic absorption spectrophotometer (see *Spectrophotometry and Light-Scattering* (851)), equipped with a magnesium hollow-cathode lamp and a nitrous oxide-acetylene flame, using water as the blank. Plot the absorbances of the *Standard preparations*, in μg per mL, of magnesium, and draw the line best fitting the three plotted points. From the graph so obtained determine the concentration, C, in μg per mL, of magnesium in the *Assay preparation*. Calculate the quantity, in mg, of magnesium trisilicate (Mg<sub>2</sub>Si<sub>2</sub>O<sub>8</sub>) in the portion of Tablets taken by the formula:

$$0.5C(260.86 / 48.62)$$

in which 260.86 is the molecular weight of anhydrous magnesium trisilicate and 48.62 is twice the atomic weight of magnesium.

## Aluminum Acetate Topical Solution



C<sub>6</sub>H<sub>9</sub>AlO<sub>6</sub> 204.11  
Acetic acid, aluminum salt.  
Aluminum acetate [139-12-8].

» Aluminum Acetate Topical Solution yields, from each 100 mL, not less than 1.20 g and not more than 1.45 g of aluminum oxide (Al<sub>2</sub>O<sub>3</sub>), and not less than 4.24 g and not more than 5.12 g of acetic acid (C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>), corresponding to not less than 4.8 g and not more than 5.8 g of aluminum acetate (C<sub>6</sub>H<sub>9</sub>AlO<sub>6</sub>). Aluminum Acetate Topical Solution may be stabilized by the addition of not more than 0.6 per cent of Boric Acid.

Aluminum Subacetate Topical Solution . . . . .	545 mL
Glacial Acetic Acid . . . . .	15 mL
Purified Water, a sufficient quantity, to make . . . . .	1000 mL

Add the Glacial Acetic Acid to the Aluminum Subacetate Topical Solution and sufficient water to make 1000 mL. Mix, and filter, if necessary.

NOTE—Dispense only clear Aluminum Acetate Topical Solution.

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

**Identification**—It responds to the tests for *Aluminum* (191) and for the ferric chloride test for *Acetate* (191) with a deep red color upon the addition of ferric chloride TS. This color is destroyed by the addition of a mineral acid.

**pH** (791): between 3.6 and 4.4.

**Limit of boric acid**—Pipet 25 mL into 75 mL of water in a conical flask. Add 3 mL of phenolphthalein TS, then add 0.5 N sodium hydroxide VS from a buret until a faint pink color is obtained. Heat to boiling, and again neutralize. Add 150 mL of glycerin to the neutralized solution, and titrate with 0.5 N sodium hydroxide VS. Perform a blank determination in a similar manner. From the volume of 0.5 N sodium hydroxide VS used after the addition of the glycerin, subtract the volume used in the blank. Each mL of 0.5 N sodium hydroxide is equivalent to 30.92 mg of H<sub>3</sub>BO<sub>3</sub>.

**Heavy metals** (231)—Dilute 2 mL of it with water to 25 mL: the limit is 0.001%.

#### Assay for aluminum oxide—

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

*Procedure*—Pipet 25 mL of Topical Solution into a 250-mL volumetric flask, add 5 mL of hydrochloric acid, dilute with water to volume, and mix. Pipet 25 mL of this solution into a 250-mL beaker, and add, in the order named and with continuous stirring, 25.0 mL of *Eдетate disodium titrant* and 20 mL of acetic acid-ammonium acetate buffer TS, then heat the solution near the boiling point for 5 minutes. Cool, and add 50 mL of alcohol and 2 mL of dithizone TS. Titrate the solution with 0.05 M zinc sulfate VS to a bright rose-pink color. Perform a blank determination, substituting water for the sample, and make any necessary correction. Each mL of 0.05 M *Eдетate disodium titrant* is equivalent to 2.549 mg of Al<sub>2</sub>O<sub>3</sub>.

**Assay for acetic acid**—Pipet 20 mL of Topical Solution into a Kjeldahl flask containing a mixture of 20 mL of phosphoric acid and 150 mL of water. Connect the flask to a condenser, the delivery tube from which dips beneath the surface of 50.0 mL of 0.5 N sodium hydroxide VS contained in a receiving flask. Distill about 160 mL, then remove the delivery tube from below the surface of the liquid, allow the distilling flask to cool, add 50 mL of water, and distill an additional 40 to 45 mL into the receiving flask. Add phenolphthalein TS to the distillate, and titrate the excess 0.5 N sodium hydroxide VS with 0.5 N sulfuric acid VS. Each mL of 0.5 N sodium hydroxide is equivalent to 30.03 mg of C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>.

## Aluminum Chloride

AlCl<sub>3</sub> · 6H<sub>2</sub>O 241.43  
Aluminum chloride, hexahydrate.  
Aluminum chloride hexahydrate [7784-13-6].  
Anhydrous 133.34 [7446-70-0].

» Aluminum Chloride contains not less than 95.0 percent and not more than 102.0 percent of AlCl<sub>3</sub>, calculated on the anhydrous basis.

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

**Identification**—A solution (1 in 10) responds to the tests for *Aluminum* (191) and for *Chloride* (191).

**Water**, *Method I* (921): between 42.0% and 48.0%.

**Sulfate**—The addition of 0.2 mL of barium chloride TS to 10 mL of a solution (1 in 100) produces no turbidity within 1 minute.

**Limit of alkalis and alkaline earths**—To a boiling solution of 1.0 g in 150 mL of water add a few drops of methyl red TS, then add 6 N ammonium hydroxide until the color of the solution just changes to a distinct yellow. Add hot water to restore the volume to 150 mL, and filter while hot. Evaporate 75 mL of the filtrate to dryness, and ignite to constant weight: the weight of the residue does not exceed 2.5 mg (0.5%).

**Heavy metals**, *Method I* (231)—Dissolve 1 g in 1 mL of 1 N acetic acid and sufficient water to make 25 mL: the limit is 0.002%.

**Iron** (241)—Dissolve 1.0 g in 45 mL of water, and add 2 mL of hydrochloric acid: the limit is 0.001%.

**Assay**—

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

*Procedure*—Transfer to a 250-mL volumetric flask about 5 g of Aluminum Chloride, accurately weighed, dissolve in and dilute with water to volume, and mix. Transfer 10.0 mL of the solution to 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 minutes. 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 assay preparation, and make any necessary correction. Each mL of 0.05 M *Edetate disodium titrant* is equivalent to 6.667 mg of  $\text{AlCl}_3$ .

## Aluminum Chlorohydrate

$\text{Al}_x(\text{OH})_{3y-z}\text{Cl}_z \cdot \text{H}_2\text{O}$

Aluminum chlorohydroxide.

Aluminum hydroxychloride.

Dihydrate [12042-91-0].

Anhydrous [1327-41-9].

Aluminum chlorohydroxide, dihydrate.

Aluminum hydroxychloride, dihydrate.

Dihydrate 210.48 [12042-91-0].

Anhydrous 174.45 [1327-41-9].

» Aluminum Chlorohydrate consists of complex basic aluminum chloride that is polymeric and loosely hydrated and encompasses a range of aluminum-to-chloride atomic ratios between 1.91:1 and 2.10:1. It contains the equivalent of not less than 90.0 per cent and not more than 110.0 percent of the labeled amount of anhydrous aluminum chlorohydrate.

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

**Labeling**—The label states the content of anhydrous aluminum chlorohydrate.

**Identification**—A solution (1 in 10) responds to the tests for *Aluminum* (191) and for *Chloride* (191).

**pH** (791): between 3.0 and 5.0, in a solution [15 in 100 (w/w)].

**Arsenic**, *Method I* (211): 2 µg per g.

**Heavy metals**, *Method I* (231): 0.002%.

**Limit of iron**—

*Standard preparation*—Transfer 2.0 mL of *Standard Iron Solution*, prepared as directed under *Iron* (241), to a 50-mL beaker.

*Test preparation*—Transfer 2.7 g of Aluminum Chlorohydrate to a 100-mL volumetric flask, dilute with water to volume, and mix. Transfer 5.0 mL of this solution to a 50-mL beaker.

*Procedure*—To each of the beakers containing the *Standard preparation* and the *Test preparation* add 5 mL of 6 N nitric acid, cover with a watch glass, and boil on a hot plate for 3 to 5 minutes. Allow to cool, add 5 mL of *Ammonium Thiocyanate Solution*, prepared as directed under *Iron* (241), transfer to separate 50-mL color comparison tubes, dilute with water to volume, and mix: the color of the solution from the *Test preparation* is not darker than that of the solution from the *Standard preparation* (150 µg per g).

**Content of aluminum**—

*Edetate disodium titrant*—Prepare and standardize as directed in the *Assay* under *Ammonium Alum*, except to use 37.2 g of edetate disodium instead of 18.6 g.

*Test solution*—Transfer about 200 mg of Aluminum Chlorohydrate, accurately weighed, to a 250-mL beaker, add 20 mL of water and 5 mL of hydrochloric acid, boil on a hot plate for not less than 5 minutes, and allow to cool.

*Procedure*—To the *Test solution* add 25.0 mL of *Edetate disodium titrant*, and adjust with 2.5 N ammonium hydroxide or 1 N acetic acid to a pH of  $4.7 \pm 0.1$ . Add 20 mL of acetic acid-ammonium acetate buffer TS, 50 mL of alcohol, and 5 mL of dithizone TS. The pH of this solution should be  $4.7 \pm 0.1$ . Titrate with 0.1 M zinc sulfate VS until the color changes from green-violet to rose-pink. Perform a blank titration, and make any necessary correction. Each mL of 0.1 M *Edetate disodium titrant* consumed is equivalent to 2.698 mg of aluminum (Al). Use the aluminum content thus obtained to calculate the *Aluminum/chloride atomic ratio*.

**Content of chloride**—Transfer about 700 mg of Aluminum Chlorohydrate, accurately weighed, to a 250-mL beaker, and add 100 mL of water and 10 mL of diluted nitric acid with stirring. Titrate with 0.1 N silver nitrate VS using a glass silver-silver chloride electrode and a silver billet electrode system, determining the endpoint potentiometrically. Each mL of 0.1 N silver nitrate is equivalent to 3.545 mg of chloride (Cl). Use the chloride content thus obtained to calculate the *Aluminum/chloride atomic ratio*.

**Aluminum/chloride atomic ratio**—Divide the percentage of aluminum found in the test for *Content of aluminum* by the percentage of chloride found in the test for *Content of chloride*, and multiply by 35.453/26.98, in which 35.453 and 26.98 are the atomic weights of chlorine and aluminum, respectively: the ratio is between 1.91:1 and 2.10:1.

**Assay**—Calculate the percentage of anhydrous aluminum chlorohydrate in the Aluminum Chlorohydrate by the formula:

$$\text{Al} \{ (26.98x + [17.01(3x - 1)] + 35.453) / 26.98x \}$$

in which *Al* is the percentage of aluminum as obtained in the test for *Content of aluminum*, *x* is the aluminum/chloride atomic ratio, 26.98 is the atomic weight of aluminum, 17.01 is the molecular weight of the hydroxide anion (OH), and 35.453 is the atomic weight of chlorine (Cl).

## Aluminum Chlorohydrate Solution

» Aluminum Chlorohydrate Solution consists of complex basic aluminum chloride that is polymeric and encompasses a range of aluminum-to-chloride ratios between 1.91:1 and 2.10:1. The following solvents may be used: water, propylene glycol, dipropylene glycol, or alcohol. It contains the equivalent of not less than 90.0 per cent and not more than 110.0 per cent of the labeled con-