

Arsenic, Method I (211): 2 µg per g.

Heavy metals, Method I (231): 20 µg per g.

Limit of iron—Using Aluminum Sesquichlorohydrate Propylene Glycol instead of Aluminum Chlorohydrate, proceed as directed in the test for *Limit of iron* under *Aluminum Chlorohydrate*. The limit is 150 µg per g.

Content of aluminum—Using Aluminum Sesquichlorohydrate Propylene Glycol instead of Aluminum Chlorohydrate, proceed as directed in the test for *Content of aluminum* under *Aluminum Chlorohydrate*. Use the result obtained to calculate the *Aluminum/chloride atomic ratio*.

Content of chloride—Using Aluminum Sesquichlorohydrate Propylene Glycol instead of Aluminum Chlorohydrate, proceed as directed in the test for *Content of chloride* under *Aluminum Chlorohydrate*. Use the result 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 1.26:1 and 1.90:1.

Assay—Calculate the percentage of anhydrous aluminum sesquichlorohydrate in the Aluminum Sesquichlorohydrate Propylene Glycol by the formula:

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

in which *Al* is the percentage of aluminum found in the test for *Content of aluminum*, *x* is the aluminum/chloride atomic ratio found in the test for *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 Subacetate Topical Solution

» Aluminum Subacetate Topical Solution yields, from each 100 mL, not less than 2.30 g and not more than 2.60 g of aluminum oxide (Al₂O₃), and not less than 5.43 g and not more than 6.13 g of acetic acid (C₂H₄O₂). It may be stabilized by the addition of not more than 0.9 per cent of boric acid.

Aluminum Subacetate Topical Solution may be prepared as follows.

Aluminum Sulfate	145 g
Acetic Acid	160 mL
Calcium Carbonate	70 g
Purified Water, a sufficient quantity, to make	1000 mL

Dissolve the Aluminum Sulfate in 600 mL of cold water, filter the solution, and add the Calcium Carbonate gradually, in several portions, with constant stirring. Then slowly add the Acetic Acid, mix, and set the mixture aside for 24 hours. Filter the product with the aid of vacuum if necessary, returning the first portion of the filtrate to the funnel. Wash the magma on the filter with small portions of cold water, until the total filtrate measures 1000 mL.

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 3.8 and 4.6.

Limit of boric acid—Proceed as directed in the test for *Limit of boric acid* under *Aluminum Acetate Topical Solution*.

Assay for aluminum oxide—

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

Procedure—Pipet 20 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 dilution into a 250-mL beaker, and proceed as directed for *Procedure* in the *Assay for aluminum oxide* under *Aluminum Acetate Topical Solution*, beginning with “add, in the order named.” Each mL of 0.05 M *Edetate disodium titrant* is equivalent to 2.549 mg of Al₂O₃.

Assay for acetic acid—Proceed as directed in the *Assay for acetic acid* under *Aluminum Acetate Topical Solution*.

Aluminum Sulfate

Al₂(SO₄)₃ · xH₂O (anhydrous) 342.15
Sulfuric acid, aluminum salt (3:2), hydrate.
Aluminum sulfate (2:3) hydrate [17927-65-0].
Anhydrous 342.16 [10043-01-3].

» Aluminum Sulfate contains not less than 54.0 percent and not more than 59.0 per cent of Al₂(SO₄)₃. It contains a varying amount of water of crystallization.

Packaging and storage—Preserve in well-closed containers.

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

pH (791): not less than 2.9, in a solution (1 in 20).

Water, Method I (921): not less than 41.0% and not more than 46.0%.

Heavy metals (231)—Dissolve 1.0 g in 2 mL of 1 N acetic acid, and dilute with water to 25 mL. The limit is 20 µg per g.

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 and then add 6 N ammonium hydroxide just until the color of the solution 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: not more than 2 mg of residue remains (0.4%).

Limit of ammonium salts—Heat 1 g with 10 mL of 1 N sodium hydroxide on a steam bath for 1 minute: the odor of ammonia is not perceptible.

Iron—To 20 mL of a solution (1 in 150) add 0.3 mL of potassium ferrocyanide TS: no blue color is produced immediately.

Assay—

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

Procedure—Transfer about 7.5 g of Aluminum Sulfate, accurately weighed, to a 250-mL volumetric flask, and dissolve in water. Dilute with water to volume, mix, and pipet 10 mL of the solution into a 250-mL beaker. Proceed as directed in the *Assay for aluminum oxide* under *Aluminum Acetate Topical Solution*, beginning with “add, in the order named.” Each mL of 0.05 M *Edetate disodium titrant* is equivalent to 8.554 mg of Al₂(SO₄)₃.

Aluminum Sulfate and Calcium Acetate for Topical Solution

» Aluminum Sulfate and Calcium Acetate for Topical Solution contains not less than 90.0 per cent and not more than 110.0 per cent of the labeled amounts of aluminum sulfate tetradecahydrate $[\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}]$ and calcium acetate monohydrate $(\text{C}_4\text{H}_6\text{CaO}_4 \cdot \text{H}_2\text{O})$.

Packaging and storage—Preserve in single-unit containers, and protect from excessive heat.

Identification—

A: Place approximately 0.25 g of Aluminum Sulfate and Calcium Acetate for Topical Solution in a test tube. Add 10 mL of water and 0.25 g of calcium carbonate. Heat on a steam bath for 10 minutes, and filter. Add 3 to 4 drops of ferric chloride TS to the filtrate. A reddish-brown color or precipitate indicates acetate. [NOTE—After the addition of the ferric chloride TS, the solution may be heated for 1 minute to speed the reaction.]

B: Suspend 2 g of sample in 50 mL of water, and filter. The filtrate meets the requirements of the tests for *Sulfate* (191) and for *Calcium* (191).

pH (791): between 4.0 and 4.8 in a solution (1 in 200).

Assay for aluminum sulfate—

Assay preparation—Transfer 10 g of Aluminum Sulfate and Calcium Acetate for Topical Solution, accurately weighed, to a 1000-mL volumetric flask. Add 100 mL of 1.2 M hydrochloric acid and approximately 250 mL of water. Heat on a steam bath or hot plate until dissolved. Cool, dilute with water to volume, and mix. [NOTE—Retain a portion of this *Assay preparation* for the *Assay for calcium acetate*.]

Procedure—Transfer a 5.0-mL aliquot of the *Assay preparation* to a 250-mL conical flask. Add 40.0 mL of 0.01 M edetate disodium VS and 20 mL of acetic acid–ammonium acetate buffer TS, and mix well. Add 50 mL of alcohol and 2 mL of dithizone TS. [NOTE—Follow the given order of addition.] Titrate with 0.02 M zinc sulfate VS until the color changes from green-violet to clear rose-pink. Perform a blank titration, substituting 5.0 mL of water for the *Assay preparation*. Each mL of 0.01 M edetate disodium is equivalent to 2.972 mg of aluminum sulfate tetradecahydrate $[\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}]$. Calculate the percentage of aluminum sulfate tetradecahydrate $[\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}]$ by the formula:

$$[(1000)(100)C_F M(V_B - V_U)] / 5.0 M_T W$$

in which 1000/5.0 is the dilution factor; 100 is the conversion factor to percentage; C_F is the conversion factor (2.972 mg of sample per mL of 0.01 M edetate disodium); M is the actual molarity of the titrant; V_B is the blank titration volume, in mL; V_U is the sample titration volume, in mL; M_T is the theoretical molarity of the titrant (0.02); and W is the weight of the sample, in mg.

Assay for calcium acetate—

Procedure—Transfer a 5.0-mL aliquot of the *Assay preparation* retained from the *Assay for aluminum sulfate* to a 250-mL conical flask. Add 1 to 2 mL of 50% triethanolamine to mask the aluminum. Mix well. Add 100 mL of water, 15 mL of 1 N sodium hydroxide, and approximately 300 mg of hydroxy naphthol blue. [NOTE—Follow the given order of addition.] Titrate the solution with 0.01 M edetate disodium VS. The indicator will change from purple to a clear blue color at the endpoint. Each mL of 0.01 M edetate disodium is equivalent to 1.762 mg of calcium acetate monohydrate $(\text{C}_4\text{H}_6\text{CaO}_4 \cdot \text{H}_2\text{O})$. Calculate

the percentage of calcium acetate monohydrate $(\text{C}_4\text{H}_6\text{CaO}_4 \cdot \text{H}_2\text{O})$ by the formula:

$$[(1000)(100)V_U C_F M] / 5.0 M_T W$$

in which 1000/5.0 is the dilution factor; 100 is the conversion factor to percentage; V_U is the sample titration volume, in mL; C_F is the conversion factor (1.762 mg of sample per mL of 0.01 M edetate disodium); M is the actual molarity of the titrant; M_T is the theoretical molarity of the titrant (0.01); and W is the weight of the sample, in mg.

Aluminum Sulfate and Calcium Acetate Tablets for Topical Solution

» Aluminum Sulfate and Calcium Acetate Tablets for Topical Solution contain not less than 90.0 percent and not more than 110.0 per cent of the labeled amounts of aluminum sulfate tetradecahydrate $[\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}]$ and calcium acetate monohydrate $(\text{C}_4\text{H}_6\text{CaO}_4 \cdot \text{H}_2\text{O})$.

Packaging and storage—Preserve in tight containers, and avoid excessive heat.

Identification—

A: Suspend 2 g of ground Tablet powder in 50 mL of water, and filter. Mix 2 mL of the filtrate with 2 mL of water and 2 drops of 3 N hydrochloric acid: the solution responds to the ammonium hydroxide test for *Aluminum* (191). [NOTE—Retain the remaining filtrate for *Identification* test B.]

B: A portion of the filtrate retained from *Identification* test A responds to the tests for *Sulfate* (191) and for *Calcium* (191).

Disintegration (701): 10 minutes.

Uniformity of dosage units (905): meet the requirements for *Weight Variation*.

pH (791): between 4.0 and 4.8, in a solution (2 g of ground Tablet powder in 500 mL of water).

Loss on drying (731)—Dry ground Tablet powder at 150° for 15 minutes: it loses not more than 18% of its weight.

Assay for aluminum sulfate—

Assay preparation—Finely powder and mix not fewer than 20 Tablets. Weigh accurately a portion of the powder, equivalent to about 2.8 g of aluminum sulfate, and transfer to a 1000-mL volumetric flask. Add 100 mL of 1.2 N hydrochloric acid and 100 mL of water, and heat on a steam bath, with occasional swirling, to dissolve the powder. Allow the solution to cool, dilute with water to volume, and mix. [NOTE—Retain a portion of this *Assay preparation* for the *Assay for calcium acetate*.]

Procedure—Transfer 25.0 mL of the *Assay preparation* to a 250-mL conical flask. Add 40.0 mL of 0.01 M edetate disodium VS and 20 mL of acetic acid–ammonium acetate buffer TS, and mix by swirling. Add 50 mL of alcohol and 2 mL of dithizone TS, and titrate with 0.02 M zinc sulfate VS until the color changes from green-violet to a clear rose-pink. Perform a blank determination, substituting 25 mL of water for the *Assay preparation*, and make any necessary correction. Each mL of 0.01 M edetate disodium is equivalent to 2.972 mg of $\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}$.

Assay for calcium acetate—Transfer 20.0 mL of the *Assay preparation* retained from the *Assay for aluminum sulfate* to a 125-mL conical flask. With constant stirring, add in the order named, about 0.5 mL of triethanolamine, 10 mL of ammonia–ammonium chloride buffer TS, and 3 drops of a solution prepared by dissolving 500 mg of eriochrome black T in 10 mL of methanol, and titrate with 0.01 M edetate disodium VS to a violet endpoint. Each mL of 0.01 M edetate disodium is equivalent to 1.762 mg of $\text{C}_4\text{H}_6\text{CaO}_4 \cdot \text{H}_2\text{O}$.