

- **USP REFERENCE STANDARDS** (11)  
USP Altretamine RS

## Ammonium Alum

$\text{AlNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$	453.33
$\text{AlNH}_4(\text{SO}_4)_2$	237.15
Sulfuric acid, aluminum ammonium salt (2:1:1), dodecahydrate;	
Aluminum ammonium sulfate (1:1:2), dodecahydrate [7784-26-1].	
Anhydrous [7784-25-0].	

### DEFINITION

Ammonium Alum contains NLT 99.0% and NMT 100.5% of ammonium alum [ $\text{AlNH}_4(\text{SO}_4)_2$ ], calculated on the dried basis.

### IDENTIFICATION

- **A.**  
Sample solution: 50 mg/mL  
Analysis: Add 1 N sodium hydroxide dropwise to the Sample solution.  
Acceptance criteria: A precipitate is formed, and it dissolves in an excess of the reagent with the evolution of ammonia, recognizable by its alkaline effect upon moistened red litmus paper exposed to the vapor.
- **B. IDENTIFICATION TESTS—GENERAL, Aluminum (191)**  
Sample solution: 50 mg/mL  
Acceptance criteria: Meets the requirements

### Change to read:

- **C. IDENTIFICATION TESTS—GENERAL (191), Sulfate**  
Sample solution: 50 mg/mL  
Analysis: Proceed as directed in *Identification Tests—General, Sulfate (191)*, except centrifuge the neutral solutions of sulfates and use the supernatants for test B.  
• (CN 1-May-2018)  
Acceptance criteria: Meets the requirements

### ASSAY

- **PROCEDURE**  
Edetate disodium titrant: Prepare and standardize as directed in *Reagents, Volumetric Solutions, Edetate Disodium, Twentieth-Molar (0.05 M)*.  
Sample: 800 mg of Ammonium Alum  
Analysis: Transfer the Sample to a 400-mL beaker, moisten with 1 mL of glacial acetic acid, and add 50 mL of water, 50.0 mL of Edetate disodium titrant and 20 mL of acetic acid–ammonium acetate buffer TS. Warm on a steam bath until the solution is complete, and boil gently for 5 min. Cool, add 50 mL of alcohol and 2 mL of dithizone TS, and titrate the excess edetate disodium with 0.05 M zinc sulfate VS to a bright rose-pink color. Perform a blank determination, and make any necessary correction. Each mL of 0.05 M Edetate disodium titrant is equivalent to 11.86 mg of  $\text{AlNH}_4(\text{SO}_4)_2$ .  
Acceptance criteria: 99.0%–100.5% on the dried basis

### IMPURITIES

#### Delete the following:

- **HEAVY METALS, Method I (231)**  
Test preparation: Dissolve 1 g in 20 mL of water, and add 5 mL of 0.1 N hydrochloric acid. Evaporate the solution in a porcelain evaporating dish to dryness. Treat the residue with 20 mL of water, and add 50 mg of hydroxylamine hydrochloride. Heat the solution on a

steam bath for 10 min, cool, and dilute with water to 25 mL.

Analysis: Proceed as directed in the chapter, except add 50 mg of hydroxylamine hydrochloride to the Standard Preparation.

Acceptance criteria: 20 ppm • (Official 1-Jan-2018)

### IRON

Sample solution: 6.7 mg/mL

Analysis: Add 5 drops of potassium ferrocyanide TS to 20 mL of the Sample solution.

Acceptance criteria: No blue color is produced immediately.

### SPECIFIC TESTS

#### LOSS ON DRYING (731)

Sample: 2.0 g

Analysis: Transfer the Sample, in a tared porcelain crucible, to a muffle furnace at 200°. Increase the temperature to 300°, and dry at 300° to a constant weight. Cool in a desiccator, and weigh.

Acceptance criteria: 45.0%–48.0%

#### LIMIT OF ALKALIES AND ALKALINE EARTHS

Sample: 1 g

Analysis: Completely precipitate the aluminum from a boiling solution of the Sample in 100 mL of water by the addition of sufficient 6 N ammonium hydroxide to render the solution distinctly alkaline to methyl red TS, and filter. Evaporate the filtrate to dryness, and ignite.

Acceptance criteria: The weight of the residue is NMT 5 mg (0.5%).

## Potassium Alum

$\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$	474.39
$\text{AlK}(\text{SO}_4)_2$	258.21
Sulfuric acid, aluminum potassium salt (2:1:1), dodecahydrate;	
Aluminum potassium sulfate (1:1:2) dodecahydrate [7784-24-9].	
Anhydrous [10043-67-1].	

### DEFINITION

Potassium Alum contains NLT 99.0% and NMT 100.5% of potassium alum [ $\text{AlK}(\text{SO}_4)_2$ ], calculated on the dried basis.

### IDENTIFICATION

- **A.**  
Sample solution: 50 mg/mL in water  
Analysis: Add 1 N sodium hydroxide dropwise to the Sample solution.  
Acceptance criteria: A precipitate is formed that dissolves in an excess of the reagent. Ammonia is not evolved (distinction from ammonium alum).
- **B.**  
Analysis: Hold it in a nonluminous flame.  
Acceptance criteria: A violet color is imparted to the flame.
- **C.**  
Sample solution: Saturated solution in water  
Analysis: Add 10 mL of sodium bitartrate TS to 5 mL of the Sample solution.  
Acceptance criteria: A white, crystalline precipitate is formed within 30 min.
- **D. IDENTIFICATION TESTS—GENERAL, Aluminum (191) AND Sulfate (191)**  
Sample solution: 50 mg/mL in water  
Acceptance criteria: Meets the requirements

### ASSAY

#### PROCEDURE

Edetate disodium titrant: Prepare and standardize as directed in *Reagents, Indicators, and Solutions—*