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Ammonium Sulfate

$(NH_4)_2SO_4$ 132.14

Ammonium sulfate CAS RN®: 7783-20-2.

DEFINITION

Ammonium Sulfate contains NLT 99.0% and NMT 100.5% of ammonium sulfate $[(NH_4)_2SO_4]$.

IDENTIFICATION

- **A. Identification Tests—General ⟨191⟩, *Chemical Identification Tests, Ammonium*:** A solution (1 in 20) meets the requirements.
- **B. Identification Tests—General ⟨191⟩, *Chemical Identification Tests, Sulfate*:** A solution (1 in 20) meets the requirements.

ASSAY

- **Procedure**

Sample: 2.5 g of Ammonium Sulfate

Titrimetric system

(See *Titrimetry* ⟨541⟩.)

Mode: Residual titration

Titrant: 1 N sodium hydroxide VS

Back titrant: 1 N sulfuric acid VS

Endpoint detection: Colorimetric

Blank: 50.0 mL of 1 N sodium hydroxide VS, accurately measured

Analysis: Add the *Sample* to a 500-mL conical flask and dissolve in 50 mL of water. Add 50.0 mL of *Titrant*, place a filter funnel loosely in the neck of the flask, and boil until ammonia is expelled (about 10–15 min), as determined with litmus paper. Cool, add 0.15 mL of thymol blue TS, and titrate the excess sodium hydroxide with the *Back titrant*. Perform a blank determination.

Calculate the percentage of ammonium sulfate $[(NH_4)_2SO_4]$ in the portion of the *Sample* taken:

$$\text{Result} = [(V_B - V_S) \times N_A \times F \times 100]/W$$

V_B = volume of the *Back titrant* consumed by the *Blank* (mL)

V_S = volume of the *Back titrant* consumed by the *Sample* (mL)

N_A = actual normality of the *Back titrant* (mEq/mL)

F = equivalency factor, 66.07 mg/mEq

W = weight of the *Sample* (mg)

Acceptance criteria: 99.0%–100.5%

IMPURITIES

- **Residue on Ignition ⟨281⟩**

Sample: 20 g

Analysis: Volatilize the *Sample* completely by slowly applying gentle heat. Cool the crucible, and moisten the residue with 0.5 mL of sulfuric acid. Ignite the crucible until white fumes of sulfur trioxide cease to evolve. Then, ignite at 800 ± 25° for 15 min.

Acceptance criteria: NMT 0.005%

- **Limit of Insoluble Matter**

Sample: 20 g

Analysis: Transfer the *Sample* to a covered beaker, and dissolve in 200 mL of water. Heat to boiling, and warm on a steam bath for 1 h. Filter the hot solution through a tared sintered-glass crucible of medium pore size (10–15 µm). Wash the beaker and the filter with hot water, dry the crucible at 105°, cool in a desiccator, and weigh.

Acceptance criteria: NMT 1 mg of insoluble matter is found (0.005%).

- **Limit of Phosphate**

Standard phosphate solution, Phosphate reagent A, and **Phosphate reagent B:** Prepare as directed for *Reagents, 6. General Tests for Reagents, 6.11 Phosphate in Reagents*.

Sample: 4.0 g

Control: 0.2 mL of *Standard phosphate solution*

Analysis

[NOTE—The tests for the *Sample* and the *Control* are made preferably in matched color-comparison tubes.]

Dissolve the *Sample* in 25 mL of 0.5 N sulfuric acid, add 1 mL each of *Phosphate reagent A* and *Phosphate reagent B*, and allow to stand at room temperature for 2 h. Proceed with the *Control* using the same quantities of the same reagents as in the test for the *Sample*.

Acceptance criteria: Any blue color obtained from the *Sample* should not exceed that produced from the *Control* (NMT 5 ppm).

- **CHLORIDE AND SULFATE ⟨221⟩, *Chloride***

Standard chloride solution: Transfer 165 mg of sodium chloride to a 100-mL volumetric flask. Dissolve in and dilute with water to volume. Transfer 10.0 mL to a 1000-mL volumetric flask, and dilute with water to volume to obtain a solution having a concentration of 10 µg/mL of chloride.

Acceptance criteria: A 2-g portion shows no more chloride than corresponds to 1.0 mL of the *Standard chloride solution* (NMT 5 ppm).

- **LIMIT OF NITRATE**

Standard nitrate solution and **Brucine sulfate solution:** Prepare as directed for *Reagents, 6. General Tests for Reagents, 6.9 Nitrate in Reagents*.

Sample solution: Dissolve 1.0 g in 3 mL of water by heating in a boiling water bath, and add *Brucine sulfate solution* to make 50 mL.

Control solution: To 1.0 mL of the *Standard nitrate solution* add 1.0 g of Ammonium Sulfate, then add *Brucine sulfate solution* to make 50 mL.

Blank: 50 mL of *Brucine sulfate solution*

Analysis: Heat the *Sample solution*, *Control solution*, and *Blank* in a boiling water bath for 15 min with periodic gentle swirling, then cool rapidly in an ice bath to room temperature. Adjust a suitable spectrophotometer with the *Blank* to zero absorbance at 410 nm. Determine the absorbance of the *Sample solution*, note the result, and adjust the instrument with the *Sample solution* to zero absorbance. Determine the absorbance of the *Control solution*.

Acceptance criteria: The absorbance reading for the *Sample solution* does not exceed that for the *Control solution* (NMT 10 ppm).

Change to read:

- **▲IRON ⟨241⟩, *Procedures, Procedure 1*▲** (CN 1-JUN-2023)

Sample solution: Dissolve 2.0 g in 40 mL of water, and add 2 mL of hydrochloric acid.

Acceptance criteria: NMT 5 ppm

SPECIFIC TESTS

- **MICROBIAL ENUMERATION TESTS ⟨61⟩** and **TESTS FOR SPECIFIED MICROORGANISMS ⟨62⟩:** The total aerobic microbial count does not exceed 1000 cfu/g, and the total combined molds and yeasts count does not exceed 10 cfu/g.

- **pH ⟨791⟩:** 5.0–6.0 in a solution (1 in 20)

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in well-closed containers. No storage requirements are specified.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
AMMONIUM SULFATE	Documentary Standards Support	SE2020 Simple Excipients

Chromatographic Database Information: Chromatographic Database

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