Ammonium Phosphate

$(NH_4)_2HPO_4$ 132.06
Phosphoric acid, diammonium salt;
Diammonium phosphate [7783-28-0].

DEFINITION
Ammonium Phosphate contains NLT 96.0% and NMT 102.0% of $(NH_4)_2HPO_4$.

IDENTIFICATION
- **A. IDENTIFICATION TESTS—GENERAL,** *Ammonium* ⟨**191**⟩**:** A solution (1 in 20) meets the requirements.
- **B. IDENTIFICATION TESTS—GENERAL,** *Phosphate* ⟨**191**⟩**:** A solution (1 in 20) meets the requirements.

ASSAY
- **PROCEDURE**
  Sample: 600 mg of Ammonium Phosphate
  Titrimetric system
  (See *Titrimetry* ⟨541⟩.)
  Mode: Direct titration
  Titrant: 0.1 N sulfuric acid VS
  Endpoint detection: potentiometric
  Analysis: Dissolve the *Sample* in 40 mL of water, and titrate with 0.1 N sulfuric acid VS to a pH of 4.6. Each mL of 0.1 N sulfuric acid is equivalent to 13.21 mg of $(NH_4)_2HPO_4$.
  Acceptance criteria: 96.0%–102.0%

IMPURITIES
- **CHLORIDE AND SULFATE,** *Chloride* ⟨**221**⟩**:** A 1.0-g portion shows no more chloride than corresponds to 0.40 mL of 0.020 N hydrochloric acid (0.03%).
- **CHLORIDE AND SULFATE,** *Sulfate* ⟨**221**⟩**:** A 0.20-g portion shows no more sulfate than corresponds to 0.30 mL of 0.020 N sulfuric acid (0.15%).
- **ARSENIC,** *Method I* ⟨**211**⟩**:** NMT 3 ppm
- **HEAVY METALS** ⟨**231**⟩
  Test preparation: Dissolve 2.0 g in 25 mL of water.
  Acceptance criteria: NMT 10 ppm

SPECIFIC TESTS
- **pH** ⟨**791**⟩**:** 7.6–8.2, in a solution (1 in 100)

ADDITIONAL REQUIREMENTS
- **PACKAGING AND STORAGE:** Preserve in tight containers.

Ammonium Sulfate

$(NH_4)_2SO_4$ 132.14
Ammonium sulfate [7783-20-2].

DEFINITION
Ammonium Sulfate contains NLT 99.0% and NMT 100.5% of $(NH_4)_2SO_4$.

IDENTIFICATION
- **A. IDENTIFICATION TESTS—GENERAL,** *Ammonium* ⟨**191**⟩**:** A solution (1 in 20) meets the requirements.
- **B. IDENTIFICATION TESTS—GENERAL,** *Sulfate* ⟨**191**⟩**:** 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 1 N sodium hydroxide VS, 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 1 N sulfuric acid VS. Perform a blank determination. Calculate the percentage of ammonium sulfate $[(NH_4)_2SO_4]$ in the *Sample* taken:

$$\text{Result} = [(B - V) \times N \times F \times 100]/W$$

B = 1 N sulfuric acid VS consumed by the *Blank* (mL)
V = 1 N sulfuric acid VS consumed by the *Sample* (mL)
N = 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
  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 porosity (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 *Phosphate in Reagents* under *Reagents, Indicators, and Solutions—General Tests for 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,** *Chloride* ⟨**221**⟩
  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 *Standard chloride solution* (NMT 5 ppm).
- **LIMIT OF NITRATE**
  Standard nitrate solution and **Brucine sulfate solution:** Prepare as directed for *Nitrate in Reagents* under *Reagents, Indicators, and Solutions—General Tests for 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.