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Ammonium Molybdate

$(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ 1236.00

Molybdate ($\text{Mo}_7\text{O}_{24}^{6-}$), hexaammonium, tetrahydrate;

Hexaammonium molybdate tetrahydrate CAS RN®: 12054-85-2; UNII: OGI154X474.

DEFINITION

Ammonium Molybdate contains NLT 99.3% and NMT 101.8% of $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$.

IDENTIFICATION

• PROCEDURE

Sample: 0.6 g

Analysis: Dissolve the *Sample* in 1.4 mL of water and 1.45 mL of ammonium hydroxide. Cool this mixture, and add slowly, with mixing, 7.2 mL of a well-cooled mixture of 3.2 mL of nitric acid and 4 mL of water. Allow to stand for 24–48 h, and pass through a sintered-glass filter. To 5 mL of the filtrate add 2 mL of dibasic sodium phosphate TS.

Acceptance criteria: A yellow precipitate is formed, and it is soluble in an excess of 6 N ammonium hydroxide.

ASSAY

• PROCEDURE

Sample solution: Dissolve 0.7 g of Ammonium Molybdate in 100 mL water. Adjust with dilute nitric acid to a pH of 4.0. Add saturated hexamethylenetetramine solution to achieve a pH of 5–6.

Analysis: Heat the *Sample solution* to 60°, and add 0.2 mL of 0.1% 4-[2-pyridylazo]resorcinol solution in alcohol. Titrate with 0.1 M lead nitrate VS from the yellow color to the first permanent pink endpoint. Carry out a blank titration. Each mL of 0.1 M lead nitrate is equivalent to 17.66 mg of $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$.

Acceptance criteria: 99.3%–101.8%

IMPURITIES

INORGANIC IMPURITIES

• ARSENATE, PHOSPHATE, AND SILICATE

Sample solution: Dissolve 2.5 g of the analyte in 70 mL of water in a container other than glass.

Control solution: Dissolve 0.5 g of the analyte in 70 mL of water in a container other than glass, and add an amount of sodium silicate solution equivalent to 0.02 mg of silica (SiO_2).

Analysis

Samples: *Sample solution* and *Control solution*

Adjust with 1.2 N hydrochloric acid to a pH of between 3 and 4, transfer to a glass container, add 2 mL of bromine TS, and adjust with 1.2 N hydrochloric acid to a pH of 1.8 ± 0.1 . Heat almost to boiling, and cool to room temperature. Dilute with water to 90 mL, add 10 mL of hydrochloric acid, and transfer to a separator. Add 1 mL of butyl alcohol and 30 mL of 4-methyl-2-pentanone, shake vigorously, and allow the phases to separate. Discard the aqueous phase, and wash the ketone phase with three successive 10-mL portions of 1.2 N hydrochloric acid, discarding the washings. To the washed ketone phase add 10 mL of 1.2 N hydrochloric acid to which has just been added 0.2 mL of a freshly prepared solution (1 in 50) of stannous chloride in hydrochloric acid.

Acceptance criteria: Any blue color in the *Sample solution* does not exceed that in the *Control solution* (NMT 10 ppm).

• [CHLORIDE AND SULFATE, Chloride\(221\)](#): A 0.5-g portion shows no more chloride than 0.30 mL of 0.001 N hydrochloric acid (NMT 20 ppm).

• [CHLORIDE AND SULFATE, Sulfate\(221\)](#): A 0.25-g portion shows no more sulfate than corresponds to 1.0 mL of 0.001 N sulfuric acid (NMT 200 ppm).

• INSOLUBLE SUBSTANCES

Sample: 20 g

Analysis: Dissolve the *Sample* in 200 mL of water in a beaker, heat to boiling, cover, and heat on a steam bath for 1 h. Pass the hot solution through a tared filtering crucible, wash the insoluble residue with hot water, and dry at 105° for 2 h.

Acceptance criteria: The weight of the residue is NMT 1 mg (0.005%).

• NITRATE

Sample: 1 g

Analysis: Dissolve the *Sample* in 10 mL of water containing 5 mg of sodium chloride, and add 0.10 mL of a solution (1 in 1000) of indigo carmine in 3.6 N sulfuric acid.

Acceptance criteria: The blue color is not completely discharged in 5 min.

• **MAGNESIUM AND ALKALI SALTS**

Sample: 5.0 g

Analysis: Dissolve the *Sample* in 50 mL of water, and filter. To the filtrate add 0.5 g of sodium carbonate and 25 mL of 2.5 N sodium hydroxide. Boil the solution gently for 5 min, cool, and pass through an ignited and tared filter. Wash the filter with 1 N ammonium hydroxide. Ignite the filter at $800 \pm 25^\circ$ for 30 min.

Acceptance criteria: The weight of the residue does not exceed 1 mg (NMT 0.02%).

• **PHOSPHATE**

Standard solution: Dissolve 143.3 mg of dried monobasic potassium phosphate in water to make 1000 mL, and then dilute 1.0 mL of this solution with 3 N ammonium hydroxide to 100 mL.

Sample solution: Dissolve 20 g of the analyte in 100 mL of 3 N ammonium hydroxide.

Analysis

Samples: *Standard solution* and *Sample solution*

Add 3.5 mL of ferric nitrate solution (1 in 10), and allow to stand for 15 min. Warm gently to coagulate the precipitate, and filter. Wash the filter several times with 1.5 N ammonium hydroxide, then wash the filter with 60 mL of warm 4 N nitric acid to dissolve the residue on the filter, collecting the filtrate in a glass-stoppered, 250-mL conical flask. Add 13 mL of ammonium hydroxide, warm to 40° , add 50 mL of ammonium molybdate TS, shake for 5 min, and allow to stand at 40° for 2 h.

Acceptance criteria: Any yellow precipitate formed from the *Sample solution* does not exceed that from the *Standard solution* (5 ppm).

ADDITIONAL REQUIREMENTS

• **PACKAGING AND STORAGE:** Preserve in tight containers.

Auxiliary Information - Please [check for your question in the FAQs](#) before contacting USP.

Topic/Question	Contact	Expert Committee
AMMONIUM MOLYBDATE	Documentary Standards Support	SM32020 Small Molecules 3

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