Labeling—The label states the content of ammonium chloride in terms of weight and of milliequivalents in a given volume. The label states also the total osmolar concentration in mOsmol per L or per mL. The label states that the Injection is not for direct injection but is to be diluted with Sodium Chloride Injection to the appropriate strength before use.

USP Reference standards 〈11〉—
USP Endotoxin RS

Identification—It responds to the tests for *Ammonium* 〈191〉 and for *Chloride* 〈191〉.

Bacterial endotoxins 〈85〉—It contains not more than 1.72 USP Endotoxin Units per mEq of chloride.

pH 〈791〉: between 4.0 and 6.0, in a concentration of not more than 100 mg of ammonium chloride per mL.

Particulate matter 〈788〉: meets the requirements for small-volume injections.

Chloride content—Transfer an accurately measured volume of Injection, evaporated, if necessary, equivalent to about 2 g of ammonium chloride, to a 200-mL volumetric flask, dilute with water to volume, and mix. Transfer 10.0 mL of this solution to a conical flask, add 10 mL of glacial acetic acid, 75 mL of methanol, and 0.5 mL of eosin Y TS. Titrate, with shaking, with 0.1 N silver nitrate VS to a pink endpoint. Each mL of 0.1 N silver nitrate is equivalent to 3.545 mg of Cl. The content of Cl is between 63.0% and 70.3% of the labeled amount of ammonium chloride.

Other requirements—It meets the requirements under *Injections* 〈1〉.

Assay—Transfer an accurately measured volume of Injection, equivalent to about 200 mg of ammonium chloride, to a 500-mL Kjeldahl flask, dilute with water to 200 mL, mix, and add 50 mL of sodium hydroxide solution (2 in 5). Immediately connect the flask by means of a distillation trap to a well-cooled condenser, the delivery tube of which dips into 40 mL of boric acid solution (1 in 25) contained in a suitable receiver. Heat to boiling, and distill about 200 mL. Cool the liquid in the receiver, if necessary, then add methyl red TS, and titrate with 0.1 N sulfuric acid VS. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N sulfuric acid is equivalent to 5.349 mg of NH_4Cl.

Ammonium Chloride Delayed-Release Tablets

» Ammonium Chloride Delayed-Release Tablets contain not less than 94.0 per cent and not more than 106.0 percent of the labeled amount of NH_4Cl. Ammonium Chloride Delayed-Release Tablets are enteric-coated.

Packaging and storage—Preserve in tight containers.

Identification—A filtered solution of finely powdered Tablets, equivalent to ammonium chloride solution (1 in 10), responds to the tests for *Ammonium* 〈191〉 and for *Chloride* 〈191〉.

Disintegration 〈701〉: 2 hours, determined as directed for *Enteric-Coated Tablets*.

Limit of thiocyanate—Powder and dissolve in water a sufficient number of Tablets to make about 25 mL of ammonium chloride solution (1 in 10), and filter. Acidify 10 mL of the solution with hydrochloric acid, and add a few drops of ferric chloride TS: no reddish orange color is produced.

Assay—Weigh and finely powder not fewer than 20 Tablets. Transfer an accurately weighed portion of the powder, equivalent to about 200 mg of ammonium chloride, to a 500-mL Kjeldahl flask, and add 200 mL of water and 50 mL of sodium hydroxide solution (2 in 5). Immediately connect the flask by means of a distillation trap to a well-cooled condenser, the delivery tube of which dips into 40 mL of boric acid solution (1 in 25) contained in a suitable receiver. Heat to boiling, and distill about 200 mL. Cool the liquid in the receiver, if necessary, then add methyl red TS, and titrate with 0.1 N sulfuric acid VS. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N sulfuric acid is equivalent to 5.349 mg of NH_4Cl.

Ferric Ammonium Citrate

» Ferric Ammonium Citrate contains not less than 16.5 percent and not more than 18.5 per cent of iron (Fe).

Packaging and storage—Preserve in tight, light-resistant containers, in a cool place.

Identification—

A: Ignite about 0.5 g: it chars, and leaves a residue of iron oxide.

B: To 10 mL of a solution of Ferric Ammonium Citrate (1 in 100) add 6 N ammonium hydroxide dropwise: the solution darkens, but no precipitate forms.

C: To 5 mL of a solution of Ferric Ammonium Citrate (1 in 100) add 0.3 mL of potassium permanganate TS and 4 mL of mercuric sulfate TS, and heat the mixture to boiling: a white precipitate forms.

Ferric citrate—To a solution of Ferric Ammonium Citrate (1 in 100) add potassium ferrocyanide TS: no blue precipitate is formed.

Sulfate 〈221〉—Dissolve 100 mg in 1 mL of 2.7 N hydrochloric acid, and dilute with water to 30 mL. Add 3 mL of barium chloride TS, dilute with water to 50 mL, and mix: any turbidity formed after 10 minutes is not greater than that produced in a similarly treated control solution containing 0.31 mL of 0.020 N sulfuric acid (0.3%).

Oxalate—Transfer 1 g to a 125-mL separator, dissolve in 10 mL of water, add 2 mL of hydrochloric acid, and extract successively with one 50-mL portion and one 20-mL portion of ether. Transfer the combined ether extracts to a 150-mL beaker, add 10 mL of water, and remove the ether by evaporation on a steam bath. Add 1 drop of glacial acetic acid and 1 mL of calcium acetate solution (1 in 20): no turbidity is produced within 5 minutes.

Mercury—

Mercury Stock Solution and *Standard Mercury Solution*—Proceed as directed for *Method I* under *Mercury* 〈261〉.

Mercury Detection Instrument, Aeration Apparatus, and *Stannous Chloride Solution*—Proceed as directed for *Method IIa* and *Method IIb* under *Mercury* 〈261〉.

Standard solutions—Transfer 0.25, 0.50, 1.0, and 3.5 mL of *Mercury Stock Solution* to four separate glass-stoppered bottles, such as biological oxygen-demand bottles, of about 300-mL capacity. Dilute the contents of each bottle with water to 100 mL, and mix. These solutions contain the equivalent of 2.5, 5.0, 10.0, and 35.0 µg of mercury per mL, respectively.

Test solution—Transfer about 1.000 g of Ferric Ammonium Citrate, accurately weighed, to a 200-mL centrifuge bottle with a polytef-lined screw cap, and add 5 mL of nitric acid and 5 mL of hydrochloric acid. Close the bottle tightly, digest on a steam bath for 1 hour, and cool. Quantitatively transfer the solution to a suitable glass-stoppered bottle, dilute with water to 100 mL, and bubble air through the solution for 2 minutes. Prepare a reagent blank in the same manner.

Procedure—Add 5 mL of stannous chloride solution (1 in 10) to each solution, and immediately insert the bubbler of the *Aeration Apparatus*. Obtain the absorbances as directed by the instrument manufacturer's operating instructions. Perform a blank determination, and make any necessary correction. Plot the absorbances of the *Standard solutions* versus concentrations, in µg per mL, of mercury, and draw the straight line best fitting the plotted points. From the graph so obtained, determine the concentration, in µg per g, of mercury in the *Test solution:* not more than 10 µg per g is found.

Limit of lead—

Standard stock solution—Dissolve about 159.8 mg of lead nitrate, accurately weighed, in 100 mL of water containing 1 mL of nitric acid. Dilute with water to 1000.0 mL, and mix.

Standard solution—[NOTE—Prepare this solution on the day of use.] Transfer 10.0 mL of *Standard stock solution* to a 500-mL volumetric flask, dilute with water to volume, and mix. Each mL contains the equivalent of 2 µg of lead (Pb).

Test solution—Transfer about 15 g of Ferric Ammonium Citrate, accurately weighed, to a 100-mL volumetric flask (previously rinsed with nitric acid and water), dissolve in a mixture of 50 mL of water and 1 mL of nitric acid, dilute with water to volume, and mix.

Procedure—Using a suitable atomic absorption spectrophotometer (see *Spectrophotometry and Light-Scattering* ⟨851⟩) equipped with a deuterium arc background corrector, a digital readout device, and a burner head capable of handling 15% solids content, perform a blank determination with water, following the manufacturer's operating instructions. Separately aspirate portions of the *Standard solution* and the *Test solution*, and record the absorbances. Calculate the lead content, in µg per g, in the portion of Ferric Ammonium Citrate taken by the formula:

$$100(C / W)(A_U / A_S)$$

in which C is the concentration, in µg per mL, of lead in the *Standard solution;* W is the weight, in g, of Ferric Ammonium Citrate taken; and A_U and A_S are the absorbances of the *Test solution* and the *Standard solution*, respectively: not more than 10 µg per g is found.

Assay—Transfer about 1 g of Ferric Ammonium Citrate, accurately weighed, to a 250-mL conical flask, and dissolve in 25 mL of water and 5 mL of hydrochloric acid. Add 4 g of potassium iodide, insert the stopper, and allow to stand protected from light for 15 minutes. Add 100 mL of water, and titrate the liberated iodine with 0.1 N sodium thiosulfate VS, using starch TS as the indicator. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N sodium thiosulfate is equivalent to 5.585 mg of iron (Fe).

Ferric Ammonium Citrate for Oral Solution

» Ferric Ammonium Citrate for Oral Solution contains Ferric Ammonium Citrate and an effervescent mixture of a suitable organic acid and an alkali metal bicarbonate. It contains not less than 90.0 percent and not more than 110.0 percent of the labeled amount of Fe. It may contain one or more suitable flavors, colors, or stabilizing agents.

Packaging and storage—Preserve in tight, light-resistant containers, and store in a cool place.

Identification—A 6-g portion dissolves in 600 mL of water with effervescence. The collected gas meets the requirements of the test for *Bicarbonate* ⟨191⟩, and the resulting solution meets the requirements of the tests for *Iron* ⟨191⟩ and for *Citrate* ⟨191⟩.

Uniformity of dosage units ⟨905⟩**—**

FOR POWDER PACKAGED IN SINGLE-UNIT CONTAINERS: meets the requirements.

Deliverable volume ⟨698⟩**—**

FOR POWDER PACKAGED IN MULTIPLE-UNIT CONTAINERS: meets the requirements.

Assay—Transfer about 6 g of Ferric Ammonium Citrate for Oral Solution, accurately weighed, to a 250-mL conical flask, and dissolve in 100 mL of water. Allow the gas to escape, add 5 mL of hydrochloric acid and 4 g of potassium iodide, insert the stopper, and allow to stand protected from light for 15 minutes. Add 25 mL of water, and titrate the liberated iodine with 0.1 N sodium thiosulfate VS, using starch TS as the indicator. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N sodium thiosulfate is equivalent to 5.585 mg of Fe.

Ammonium Molybdate

$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ 1235.86

Molybdate ($Mo_7O_{24}^{6-}$), hexaammonium, tetrahydrate;

Hexaammonium molybdate tetrahydrate [12054-85-2].

DEFINITION

Ammonium Molybdate contains NLT 99.3% and NMT 101.8% of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$.

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 $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$.

  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