

sorbances 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

$(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ 1235.86
Molybdate ($\text{Mo}_7\text{O}_{24}^{6-}$), hexaammonium, tetrahydrate;
Hexaammonium molybdate tetrahydrate [12054-85-2].

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