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Ferric Ammonium Citrate

» Ferric Ammonium Citrate contains not less than 16.5 percent and not more than 18.5 percent 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.

Change to read:

Mercury—

Mercury Stock Solution and *Standard Mercury Solution*—▲Proceed as directed in *Mercury* ⟨261⟩*, Procedures, Procedure 1*.▲ (CN 1-Jun-2023)

Mercury Detection Instrument, Aeration Apparatus, and *Stannous Chloride Solution*—▲Proceed as directed in *Mercury* ⟨261⟩*, Procedures, Procedure 2 and Procedure 3*.▲ (CN 1-Jun-2023)

Standard solutions—Transfer 0.25, 0.50, 1.0, and 3.5 mL of *Standard Mercury 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 ng 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* 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 *Atomic Absorption Spectroscopy* ⟨852⟩) 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).

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

Topic/Question	Contact	Expert Committee
FERRIC AMMONIUM CITRATE	Documentary Standards Support	SM52020 Small Molecules 5
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM52020 Small Molecules 5

Chromatographic Database Information: Chromatographic Database

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