FERRIC AMMONIUM CITRATE

Prepared at the 28th JECFA (1984), published in FNP 31/2 (1984) and in FNP 52 (1992). Metals and arsenic specifications revised at the 63rd JECFA (2004). A PMTDI of 0.8 mg/kg bw for iron was established at the 29th JECFA (1985)

SYNONYMS Iron ammonium citrate, ammonium ferric citrate, ammonium iron citrate, ammonium iron (III) citrate, INS No. 381

DEFINITION A complex salt of undetermined structure, composed of iron, ammonia and citric acid; there are two types of salts - brown and green - containing different amounts of iron

Chemical names Ferric ammonium citrate, ammonium iron (III) citrate

Assay Not less than 16.5% and not more than 22.5% of iron (Fe) for the brown salt, and not less than 14.5% and not more than 16.0% of iron (Fe) for the green salt.

DESCRIPTION Brown salt: thin, transparent brown, reddish brown, or garnet red scales or granules, or a brownish yellow powder; odourless or has a slight ammoniacal odour

Green salt: thin, transparent green scales, granules, powder, or transparent green crystals; odourless

FUNCTIONAL USES Nutrient, dietary supplement (brown salt)
Nutrient, dietary supplement, anticaking agent for sodium chloride(green salt)

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Very soluble in water; insoluble in ethanol

Test for iron and ferric salts Ignite 0.5 g of the sample gently, and dissolve the residue in 5 ml of dilute hydrochloric acid TS. The solution gives positive tests for *iron* and for *ferric salts*.

Test for citrate To 5 ml of a 1-in-10 solution of the sample add 0.3 ml of potassium permanganate TS and 4 ml of mercuric sulfated TS and then heat the mixture to boiling. A white precipitated forms.

Test for ferric and ammonium salt Dissolve 0.5 g of the sample in 5 ml of water, and add 5 ml of sodium hydroxide TS. A reddish brown precipitate forms and ammonia is evolved when the mixture is heated.

PURITY

Ferric citrate Add potassium ferrocyanide TS to a 1 in 100 solution of the sample. No blue precipitation forms.

Oxalate Transfer 1 g of the sample into a 125-ml-separator, dissolve in 10 ml of water, add 2 ml of hydrochloric acid, and extract successively with 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) to the residual aqueous solution. No turbidity is produced within 5 min.

Sulfates — Not more than 0.3%
Dissolve a 100 mg sample in 1 ml of diluted hydrochloric acid TS, and dilute to 30 to 40 ml with water. Proceed as directed in the Limit Test for Sulfates, beginning with the addition of 3 ml of barium chloride TS. Any turbidity produced does not exceed that shown in a control containing 0.6 ml of 0.01 N sulfuric acid.

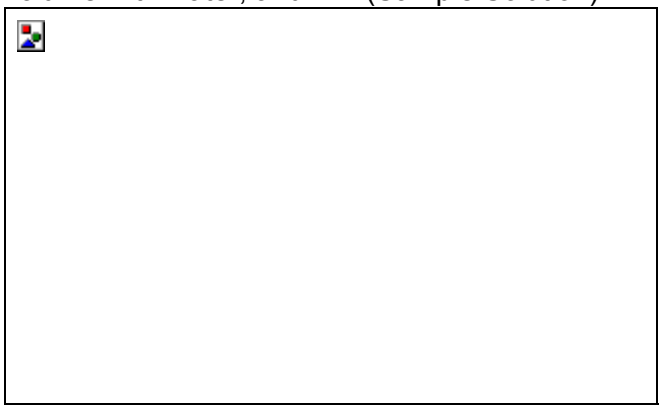
Lead (Vol. 4) — Not more than 2 mg/kg
Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4, “Instrumental Methods.”

Mercury — Not more than 1 mg/kg
See description under TESTS

TESTS

PURITY TESTS

Arsenic — Assemble the special distillation apparatus as shown in Fig. 1. Transfer 2 g of the sample, 50 ml of hydrochloric acid, and 5 g of cuprous chloride into the distilling flask (B). Reassemble the distillation apparatus and apply gentle suction to flask F to produce a continuous stream of bubbles. Heat the solution in flask B to boiling and distil until between 30 and 35 ml of distillate has been collected in flask D. Quantitatively transfer the distillate to a 100-ml volumetric flask with the aid of water, dilute to volume with water, and mix (Sample Solution).



Prepare Standard and Blank Solution in the same manner, using 6.0 ml of Standard Arsenic Solution (1 Tg/ml, see *Limit test*, Method II in place of the sample in the Standard Solution, and 6.0 ml of water in the Blank Solution. Transfer 50 ml of the Sample Solution into the generator flask (Apparatus in Method II in *Limit test*), and continue as directed in the Procedure under Arsenic Test, beginning "0.5 ml of Stannous Chloride Solution, and mix..." Modify the Procedure by using 5.0 g of Devarda's metal in place of the 3.0 g of 20-mesh granular zinc, and maintain the temperature of the reaction mixture in the generator flask between 25^{o} and 27^{o}. Treat 50.0 ml each of the Standard Solution and of the Blank Solution in the same manner and under the same condition. Determine the absorbance at 525 nm produced by each solution as directed under Procedure. Calculate the arsenic content (in mg/kg) of the sample by the

formula

where A_U is the absorbance produced by the Sample Solution, A_S is the absorbance produced by the Standard Solution, and A_B is the absorbance produced by the Blank Solution. (Note: If A_B exceeds 0.300, different samples of reagent-grade cuprous chloride and Devarda's Method should be tested for arsenic content by the procedure described herein, and lots of these reagents should be selected that will give blank readings that do not exceed 0.300.)

Lead (Vol. 4)

Note: The following method has been found to be satisfactory when the particular atomic absorption spectrophotometer specified is used. The method may be modified as necessary for use with other suitable atomic absorption spectrophotometers capable of determining lead in the sample at the limit specified.

Standard preparation

Transfer 10 ml of lead nitrate Stock Solution (see Limit Test for Heavy Metals) into a 500-ml volumetric flask, dilute to volume with water, and mix. This solution should be prepared on the day of use. Each ml contains the equivalent of 2 µg of lead ion (Pb).

Sample preparation

Transfer about 15 g of the sample, accurately weighed, into 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 to volume with water, and mix.

Procedure: Using Perkin-Elmer 403 atomic absorption spectrophotometer equipped with a deuterium arc background corrector, digital readout device, and a burner head capable of handling 15% solids content. Blank the instrument with water following the manufacturer's operating instructions. Aspirate a portion of the Standard Preparation, and record the absorbance as A_S: then aspirate a portion of the Sample Preparation, and record the absorbance as A_U. Calculate the lead content, in mg/kg, of the sample taken by the formula

where C is the concentration of Pb in the Standard Preparation, in µg per ml, and W is the weight of the sample taken, in g.

Mercury (Vol. 4)

Standard Preparation

Prepare a solution containing 1 µg of mercury (Hg) per ml as directed for Standard Preparation under Mercury Limit Test. Pipet 0.25, 0.50, 1.0 and 3.5 ml of this solution into each of four glass-stoppered bottles of about 300-ml capacity, such as BOD (Biological Oxygen Demand) bottles. Dilute the contents of each bottle to 100 ml with water, and mix. This solutions contains the equivalent of 0.25, 0.50, 1.0 and 3.5 ppm of Hg, respectively.

Sample Preparation

Transfer 1.000 g of the sample into a 200-ml screwcap centrifuge bottle, and add 5 ml of nitric acid and 5 ml of hydrochloric acid. Close the bottle tightly with a Teflon-lined screwcap, digest on a steam bath for 1 h, and cool. Quantitatively transfer into a suitable glass-stoppered bottle (see Standard Preparation), dilute to 100 ml with water, and bubble air through

the sample for 2 min. Prepare a reagent blank in the same manner. The apparatus and procedure to be used are described under the *Mercury Limit Test.*

Procedure

Add 5 ml of a 10% stannous chloride solution (prepared fresh each week by dissolving 20 g of $SnCl_2 \cdot 2H_2O$ in 40 ml of warm hydrochloric acid and diluting with 160 ml of water) to the solution to be tested, and immediately insert the bubbler of the mercury analysis apparatus. Obtain the absorbance reading by following the instrument manufacturer's operating instructions. Correct the sample readings for the reagent blank, and determine the mercury concentration of the Sample Preparation from standard curve prepared by plotting the readings obtained with the Standard Preparations against mercury concentration, in mg/kg.

METHOD OF ASSAY

Transfer about 1 g of the sample, accurately weighed, into a 250 ml glass-stoppered Erlenmeyer flask, and dissolve in 25 ml of water and 5 ml of hydrochloric acid. Add 4 g of potassium iodide, stopper, and allow to stand protected from light for 15 min. Add 100 ml of water, and titrate the liberated iodine with 0.1 N sodium thiosulfate, 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).