FERROUS SULFATE, DRIED

New specifications prepared at the 53rd JECFA (1999) and published in FNP 52 Add 7 (1999). PTMDI 0.8 mg/kg bw for iron, established at the 27th JECFA in 1983.

DEFINITION Ferrous sulfate, dried consists primarily of the monohydrate with smaller amounts of the tetrahydrate

Chemical formula $FeSO_4 \cdot H_2O$
$FeSO_4 \cdot 4H_2O$

Formula weight Monohydrate: 169.91
Tetrahydrate: 223.91

Assay Not less than 86% and not more than 89% of $FeSO_4$

DESCRIPTION A greyish-white to buff-coloured powder

FUNCTIONAL USES Nutrient supplement

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol.4) Dissolves slowly in water; insoluble in ethanol

Test for iron (Vol.4) Passes test

Test for sulfate (Vol. 4) Passes test

PURITY

Acid-insoluble matter Not more than 0.05%
Dissolve 2 g in 20 ml of freshly boiled dilute sulfuric acid (1 in 100), heat to boiling, and then digest in a covered beaker on a steam bath for 1 h. Filter through a tared filtering crucible, wash thoroughly, and dry at 105°. The weight of the insoluble residue does not exceed 1mg

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

Mercury Dithizone extraction solution

Dissolve 30 mg of dithizone in 1000 ml of chloroform, add 5 ml of alcohol, and mix. Store in a refrigerator. Shake a volume, which can be kept and used within a period of one month, with about half its volume of dilute nitric acid (1 in 100) and discard the nitric acid.

Dilute dithizone solution
Just prior to use, dilute 5 ml of Dithizone extraction solution with 25 ml of chloroform.

Hydroxylamine hydrochloride solution
Dissolve 20 g of hydroxylamine hydrochloride in water to make about 65 ml. Transfer the solution into a separator, add a few drops of thymol blue TS, and then add ammonium hydroxide until the solution assumes a yellow colour. Add 10 ml of sodium diethyldithiocarbamate solution (1 in 25), mix, and allow to stand for 5 min. Extract the solution with successive 10- to 15-ml portions of chloroform until a 5-ml test portion of the chloroform extract does not assume a yellow colour when shaken with a dilute cupric sulfate solution. Add 2.7 N hydrochloric acid until the extracted solution is pink, adding 1 or 2 drops more of thymol blue TS, if necessary, then dilute to 100 ml with water, and mix.

Mercury stock solution
Transfer 135.4 mg accurately weighed, of mercuric chloride into a 100-ml volumetric flask, dissolve in 1 N sulfuric acid, dilute to volume with the acid and mix. Dilute 5 ml of this solution to 500 ml with 1 N sulfuric acid. Each ml contains the equivalent of 10 µg of Hg.

Diluted standard mercury solution
On the day of use, transfer 100 ml of Mercury stock solution into a 100-ml volumetric flask, dilute to volume with 1 N sulfuric acid, and mix. Each ml contains the equivalent of 1 µg of Hg.

Sodium citrate solution
Dissolve 250 g of sodium citrate dihydrate in 1000 ml of water.

Sample solution
Dissolve 3 g of the sample in 30 ml of 1.7 N nitric acid by heating on a steam bath. Cool to room temperature in an ice bath, stir, and filter through S and S No. 589, or equivalent, filter paper that has been previously washed with 1.7 N nitric acid, followed by water. To the filtrate add 20 ml of Sodium citrate solution and 1 ml of Hydroxylamine hydrochloride solution.

Procedure
Work in subdued light. Prepare a control containing 3 ml of Diluted standard mercury solution, 30 ml of 1.7 N nitric acid, 5 ml of Sodium citrate solution and 1 ml of Hydroxylamine hydrochloride solution. Using a pH meter, adjust the pH of the control and the sample solution to 1.8 with ammonium hydroxide and transfer them to separators. Extract with two 5-ml portions of Dithizone extraction solution followed by 5 ml of chloroform. Discard the aqueous solutions. Transfer into clean separators the combined extracts of the two solutions, add 10 ml of dilute hydrochloric acid (1 in 2), shake well and discard the chloroform layers. Wash the acid solutions with 3 ml of chloroform and discard the chloroform layers. Add to each separator 0.1 ml

of 0.05 M disodium EDTA and 2 ml of 6 N acetic acid, mix, and then slowly add 5 ml of ammonium hydroxide. Stopper the separators, and cool under a stream of cold water. Dry the outside of the separators. Through the tops of the separators, pour the solutions carefully to avoid losses, into separate beakers, and using pH meters, adjust their pH to 1.8 with 6 N ammonium hydroxide. Return the solutions to their original separators, add 5 ml of Diluted dithizone extraction solution and shake vigorously. Any colour developed in the Sample solution does not exceed that in the control.

METHOD OF ASSAY

Dissolve about 1 g of the sample, accurately weighed, in a mixture of 25 ml of 2 N sulfuric acid and 25 ml of recently boiled and cooled water, add orthophenanthroline TS, and immediately titrate with 0.1 N ceric sulfate. Perform a blank determination, and make any necessary correction. Each ml of 0.1 N ceric sulfate is equivalent to 27.80 mg of $FeSO_4 \cdot 7H_2O$.