

FERROUS SULFATE

Prepared at the 53rd JECFA (1999) and published in FNP 52 Add 7 (1999), superseding specifications prepared at the 51st JECFA (1998), published in FNP 52 Add 6 (1998). PTMDI 0.8 mg/kg bw for iron, established at the 27th JECFA in 1983.

DEFINITION Ferrous sulfate occurs as the heptahydrate

C.A.S. number 7720-78-7

Chemical formula $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$

Formula weight 278.02

Assay Not less than 99.5% and not more than 104.5%

DESCRIPTION Pale, bluish or whitish green, odourless crystals, crystalline powder or granules.

Effloresces in dry air. In moist air it oxidizes readily to form brownish yellow basic ferric sulfate

FUNCTIONAL USES Nutrient supplement

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) One g dissolves in 1.5 ml of water at 25° and in 0.5 ml of boiling water; insoluble in ethanol

Acidity (Vol. 4) A 1 in 10 solution is acid to litmus

Test for iron (Vol. 4) Passes test

Test for sulfate (Vol. 4) Passes test

PURITY

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 $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$.