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Ferrous Sulfate

$FeSO_4 \cdot 7H_2O$ 278.01
$FeSO_4$ 151.91
Sulfuric acid, iron(2+) salt (1:1), heptahydrate;
Iron(2+) sulfate (1:1) heptahydrate CAS RN®: 7782-63-0; UNII: 39R4TAN1VT.
Anhydrous [7720-78-7].

DEFINITION

Ferrous Sulfate contains an amount of anhydrous ferrous sulfate ($FeSO_4$) equivalent to NLT 99.5% and NMT 104.5% of ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$).

IDENTIFICATION

• **A. Identification Tests—General, *Iron, Ferrous Salts*⟨191⟩ and *Sulfate*⟨191⟩:** Meets the requirements

ASSAY

• **Procedure**

Sample: 1 g of Ferrous Sulfate

Blank: Proceed as in the *Analysis* without the *Sample*.

Titrimetric system

(See *Titrimetry* ⟨541⟩.)

Mode: Direct titration

Titrant: 0.1 N ceric sulfate VS

Endpoint detection: Visual

Analysis: Dissolve the *Sample* in a mixture of 25 mL of 2 N sulfuric acid and 25 mL of freshly boiled and cooled water. Add orthophenanthroline TS, and immediately titrate with *Titrant*. Perform a blank determination.

Calculate the percentage of ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$) in the *Sample* taken:

$$\text{Result} = \{[(V_S - V_B) \times N \times F]/W\} \times 100$$

V_S = *Titrant* volume consumed by the *Sample* (mL)

V_B = *Titrant* volume consumed by the *Blank* (mL)

N = *Titrant* normality (mEq/mL)

F = equivalency factor, 278.0 mg/mEq

W = *Sample* weight (mg)

Acceptance criteria: 99.5%–104.5% of ferrous sulfate heptahydrate

IMPURITIES

Change to read:

• ▲*Arsenic* ⟨211⟩, *Procedures, Procedure 1*▲ (CN 1-Jun-2023)

Test preparation: Transfer 1 g of Ferrous Sulfate to a round-bottomed, 100-mL flask fitted with a glass joint, and add 40 mL of sulfuric acid (1 in 4) and 2 mL of 300 mg/mL potassium bromide solution. Immediately connect the flask to a condenser having a matching glass joint and a reservoir with a water jacket that is cooled by ice water. Heat the flask gently over a low flame until the solid dissolves, then distill until 25 mL of distillate collects in the reservoir. Transfer this distillate to the arsine generator flask, and wash the condenser and the reservoir with several small portions of water, adding the washings to the generator flask. Swirl to mix, add bromine TS until the color of the solution is slightly yellow, and dilute with water to 35 mL.

Acceptance criteria: NMT 3 ppm

• **Lead**

[Note—For the preparation of all aqueous solutions and for the rinsing of glassware before use, use water that has been passed through a strong-acid, strong-base, mixed-bed ion-exchange resin. Select all reagents to have as low a content of lead as practicable, and store all

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reagent solutions in containers of borosilicate glass. Cleanse glassware before use by soaking in warm nitric acid (1 in 2) for 30 min and by rinsing with deionized water.]

Ascorbic acid–sodium iodide solution: 100 mg/mL of ascorbic acid and 192.5 mg/mL of sodium iodide

Trioctylphosphine oxide solution: 50 mg/mL of trioctylphosphine oxide in 4-methyl-2-pentanone. [**Caution—**This solution causes irritation. Avoid contact with eyes, skin, and clothing. Take special precautions in disposing of unused portions of solutions to which this reagent is added.]

Standard solution: Transfer 5.0 mL of lead nitrate stock solution TS to a 100-mL volumetric flask. Dilute with water to volume, and mix. Transfer 2.0 mL of the resulting solution to a 50-mL volumetric flask. Add 10 mL of 9 N hydrochloric acid and 10 mL of water. Add 20 mL of *Ascorbic acid–sodium iodide solution* and 5.0 mL of *Trioctylphosphine oxide solution*, shake for 30 s, and allow to separate. Add water to bring the organic solvent layer into the neck of the flask, shake again, and allow to separate. The organic layer is the *Standard solution*, and it contains 2 µg/mL of lead.

Sample solution: To a 50-mL volumetric flask add 1.0 g of Ferrous Sulfate, 10 mL of 9 N hydrochloric acid, 10 mL of water, 20 mL of *Ascorbic acid–sodium iodide solution*, and 5.0 mL of *Trioctylphosphine oxide solution*. Shake for 30 s, and allow to separate. Add water to bring the organic solvent layer into the neck of the flask, shake again, and allow to separate. The organic layer is the *Sample solution*.

Blank: To a 50-mL volumetric flask add 10 mL of 9 N hydrochloric acid, 10 mL of water, 20 mL of *Ascorbic acid–sodium iodide solution*, and 5.0 mL of *Trioctylphosphine oxide solution*. Shake for 30 s, and allow to separate. Add water to bring the organic solvent layer into the neck of the flask, shake again, and allow to separate. The organic layer is the *Blank*, and it contains 0 µg/mL of lead.

Instrumental conditions

(See *Atomic Absorption Spectroscopy (852)*.)

Mode: Atomic absorption spectrophotometry

Analytical wavelength: 283.3 nm

Lamp: Lead hollow-cathode

Flame: Air–acetylene

System suitability

Samples: *Standard solution* and *Blank*

Suitability requirements: The absorbance of the *Standard solution* and the absorbance of the *Blank* are significantly different.

Analysis

Samples: *Standard solution*, *Sample solution*, and *Blank*

Concomitantly determine the absorbances of the *Blank*, *Standard solution*, and the *Sample solution*. Use the *Blank* to set the instrument to zero.

Acceptance criteria: The absorbance of the *Sample solution* does not exceed that of the *Standard solution* (NMT 10 ppm).

Change to read:

• **Mercury**

[Note—Carry out this test in subdued light, because mercuric dithizonate is light sensitive.]

Hydroxylamine hydrochloride solution, Standard mercury solution, Dithizone extraction solution, and **Diluted dithizone extraction solution:** ▲Proceed as directed in *Mercury (261), Procedures, Procedure 1*.▲ (CN 1-Jun-2023)

Control solution: Mix 3.0 mL of *Standard mercury solution*, 30 mL of dilute nitric acid (1 in 10), 5 mL of 250 mg/mL of sodium citrate solution, and 1 mL of *Hydroxylamine hydrochloride solution*.

Test preparation: Dissolve 1 g of Ferrous Sulfate in 30 mL of dilute nitric acid (1 in 10) with the aid of heat, on a steam bath. Cool quickly by immersing in an ice bath, and pass through a fine-porosity filter that previously has been washed with dilute nitric acid (1 in 10) and water. To the filtrate add 20 mL of 250 mg/mL sodium citrate solution and 1 mL of *Hydroxylamine hydrochloride solution*.

Analysis: Adjust the *Control solution* to a pH of 1.8 with ammonium hydroxide and the *Sample solution* to a pH of 1.8 with sulfuric acid. Separately transfer the solutions to separators. Treat the *Sample solution* and the *Control solution* in parallel as follows.

Extract with two 5-mL portions of *Dithizone extraction solution* and 5 mL of chloroform, pooling the chloroform extracts in a second separator. Add 10 mL of dilute hydrochloric acid (1 in 2), shake, allow the layers to separate, and discard the chloroform layer. Wash the acid extract with 3 mL of chloroform, and discard the washing. Add 0.1 mL of 20 mg/mL of edetate disodium solution and 2 mL of 6 N acetic acid, mix, and add slowly 5 mL of ammonium hydroxide. Close the separator, cool it under cold running water, and dry its outer surface. Remove the stopper, and pour the contents into a beaker. Adjust the *Sample solution* and the *Control solution* to a pH of 1.8 in the same manner as before, and return the solutions to their respective separators. Add 5.0 mL of *Diluted dithizone extraction solution*, shake vigorously, and allow the layers to separate. Using *Diluted dithizone extraction solution* as a color blank, compare the colors developed in the chloroform layers of the *Sample solution* and the *Control solution*.

Acceptance criteria: The color developed by the *Sample solution* is not more intense than that developed by the *Control solution* (NMT 3 µg/g).

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers.

• **Labeling:** Label it to indicate that it is not to be used if it is coated with brownish yellow basic ferric sulfate.

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Topic/Question	Contact	Expert Committee
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