

**Identification** (1) Dissolve 0.05 g of Fentanyl Citrate in 10 mL of 0.1 mol/L hydrochloric acid TS and ethanol (95) to make 100 mL. Determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wavelengths.

(2) Determine the infrared absorption spectrum of Fentanyl Citrate, previously dried, as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

(3) A solution of Fentanyl Citrate (1 in 100) responds to the Qualitative Tests (1) for citrate.

**pH** Dissolve 0.10 g of Fentanyl Citrate in 10 mL of water: the pH of this solution is between 3.0 and 5.0.

**Melting point** 150 – 154°C

**Purity** (1) Heavy metals—Proceed with 0.5 g of Fentanyl Citrate according to Method 2, and perform the test. Prepare the control solution with 1.0 mL of Standard Lead Solution (not more than 20 ppm).

(2) Related substances—Dissolve 0.10 g of Fentanyl Citrate in 5 mL of methanol, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add methanol to make exactly 100 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5  $\mu$ L each of the sample solution and the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of 1-butanol, water and acetic acid (100) (3:1:1) to a distance of about 10 cm, and air-dry the plate. Spray evenly Dragendorff's TS for spraying on the plate: the spots other than the principal spot from the sample solution are not more intense than the spot from the standard solution.

**Loss on drying** Not more than 0.5% (0.2 g, in vacuum, silica gel, 60°C, 2 hours).

**Residue on ignition** Not more than 0.2% (0.5 g).

**Assay** Weigh accurately about 0.075 g of Fentanyl Citrate, dissolve in 50 mL of acetic acid (100), and titrate with 0.02 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.02 mol/L perchloric acid VS  
= 10.572 mg of  $C_{22}H_{28}N_2 \cdot C_6H_8O_7$

**Containers and storage** Containers—Tight containers.  
Storage—Light-resistant.

**Description** Ferrous Sulfate occurs as pale green crystals or crystalline powder. It is odorless, and has an astringent taste.

It is freely soluble in water, and practically insoluble in ethanol (95) and in diethyl ether.

It is efflorescent in dry air, and its surface becomes yellowish brown in moist air.

**Identification** A solution of Ferrous Sulfate (1 in 10) responds to the Qualitative Tests for ferrous salt and for sulfate.

**Purity** (1) Clarity of solution—Dissolve 1.0 g of Ferrous Sulfate in 20 mL of water and 1 mL of dilute sulfuric acid: the solution is clear.

(2) Acid—To 5.0 g of powdered Ferrous Sulfate add 50 mL of ethanol (95), shake well for 2 minutes, and filter the mixture. To 25 mL of the filtrate add 50 mL of water, 3 drops of bromothymol blue TS and 0.5 mL of dilute sodium hydroxide TS: a blue color develops.

(3) Heavy metals—Take 1.0 g of Ferrous Sulfate in a porcelain dish, add 3 mL of aqua regia, and dissolve. Then evaporate on a water bath to dryness. To the residue add 5 mL of 6 mol/L hydrochloric acid TS, and dissolve. Transfer this solution to a separator. Wash the porcelain dish with two 5-mL portions of 6 mol/L hydrochloric acid TS, and combine the washings and the solution in the separator. Pour two 40-mL portions and one 20-mL portion of diethyl ether in the separator, shaking each time to mix. Allow to stand, and discard each separated diethyl ether layer. To the aqueous layer add 0.05 g of hydroxylammonium chloride, dissolve, and heat on a water bath for 10 minutes. Cool, adjust the solution to a pH of 3 to 4 by dropping strong ammonia solution, add water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution as follows: take 2.5 mL of Standard Lead Solution in a porcelain dish, add 3 mL of aqua regia, and proceed as directed for the preparation of the test solution (not more than 25 ppm).

(4) Arsenic—Prepare the test solution with 1.0 g of Ferrous Sulfate according to Method 1, and perform the test using Apparatus B (not more than 2 ppm).

**Assay** Dissolve about 0.7 g of Ferrous Sulfate, accurately weighed, in a mixture of 20 mL of water and 20 mL of dilute sulfuric acid, add 2 mL of phosphoric acid, and immediately titrate with 0.02 mol/L potassium permanganate VS.

Each mL of 0.02 mol/L potassium permanganate VS  
= 27.802 mg of  $FeSO_4 \cdot 7H_2O$

**Containers and storage** Containers—Tight containers.

## Ferrous Sulfate

硫酸鉄

$FeSO_4 \cdot 7H_2O$ : 278.01

Ferrous Sulfate contains not less than 98.0% and not more than 104.0% of  $FeSO_4 \cdot 7H_2O$ .