Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 305 nm).

Column: A stainless steel column 4.6 mm in inside diameter and 25 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 40°C.

Mobile phase: Dissolve 4.8 g of potassium dihydrogenphosphate, 5.4 g of disodium hydrogenphosphate 12-water and 1.0 g of tetra *n*-butyl ammonium bromide in water to make 1000 mL. To 870 mL of this solution add 130 mL of acetonitrile.

Flow rate: Adjust the flow rate so that the retention time of faropenem is about 11 minutes.

System suitability—

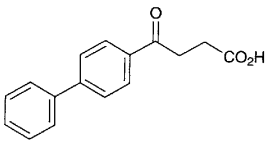
System performance: When the procedure is run with 20 μL of the standard solution under the above operating conditions, the internal standard and faropenem are eluted in this order with the resolution between these peaks being not less than 1.5.

System repeatability: When the test is repeated 6 times with 20 μL of the standard solution under the above operating conditions, the relative standard deviation of the ratios of the peak area of faropenem to that of the internal standard is not more than 1.0%.

Containers and storage Containers—Tight containers.

Fenbufen

フェンブフェン



$C_{16}H_{14}O_3$: 254.28

4-(Biphenyl-4-yl)-4-oxobutanoic acid [*36330-85-5*]

Fenbufen, when dried, contains not less than 98.0% of $C_{16}H_{14}O_3$.

Description Fenbufen occurs as a white crystalline powder.

It has a bitter taste.

It is sparingly soluble in acetone, slightly soluble in methanol, in ethanol (95) and in diethyl ether, and practically insoluble in water.

Melting point: about 188°C (with decomposition).

Identification (1) Determine the absorption spectrum of a solution of Fenbufen in ethanol (95) (1 in 200,000) 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 Fenbufen, 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.

Purity (1) Heavy metals—Take 2.0 g of Fenbufen, add 2 mL of sulfuric acid, and carbonize by gentle heating, proceed according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 10 ppm).

(2) Arsenic—Prepare the test solution with 1.0 g of Fenbufen according to Method 3, and perform the test using Apparatus B (not more than 2 ppm).

(3) Related substances—Dissolve 0.1 g of Fenbufen in 20 mL of acetone, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add acetone 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 10 μL each of the sample solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of dichloromethane, methanol and water (80:20:3) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): 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 0.3% (1 g, 105°C, 3 hours).

Residue on ignition Not more than 0.10% (1 g).

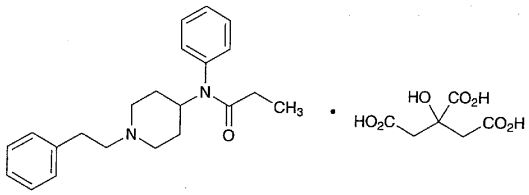
Assay Weigh accurately about 0.2 g of Fenbufen, previously dried, dissolve in 100 mL of ethanol (99.5), and titrate with 0.1 mol/L potassium hydroxide-ethanol VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L potassium hydroxide-ethanol VS
= 25.429 mg of $C_{16}H_{14}O_3$

Containers and storage Containers—Tight containers.

Fentanyl Citrate

クエン酸フェンタニル



$C_{22}H_{28}N_2O.C_6H_8O_7$: 528.59

N-(1-Phenethylpiperidin-4-yl)-*N*-phenylpropionamide monocitrate [*990-73-8*]

Fentanyl Citrate contains not less than 98.0% of $C_{22}H_{28}N_2O.C_6H_8O_7$, calculated on the dried basis.

Description Fentanyl Citrate occurs as white crystals or crystalline powder.

It is freely soluble in methanol and in acetic acid (100), sparingly soluble in water and in ethanol (95), and very slightly soluble in diethyl ether.

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 μL 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 $C_{22}H_{28}N_2.C_6H_8O_7$

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

Ferrous Sulfate

硫酸鉄

$FeSO_4.7H_2O$: 278.01

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

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 $FeSO_4.7H_2O$

Containers and storage Containers—Tight containers.