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. Place the plate in a chamber filled with iodine vapor for 10 minutes: 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% (1 g, in vacuum, phosphorus (V) oxide, 50°C, 3 hours).

**Residue on ignition** Not more than 0.20% (1 g).

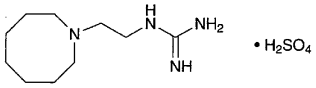
**Assay** Weigh accurately about 0.25 g of Guanabenz Acetate, previously dried, dissolve in 50 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 29.114 mg of $C_8H_8Cl_2N_4.C_2H_4O_2$

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

# Guanethidine Sulfate

硫酸グアネチジン



$C_{10}H_{22}N_4.H_2SO_4$: 296.39
1-[2-(Hexahydroazocin-1(2*H*)-yl)ethyl]guanidine monosulfate [*645-43-2*]

Guanethidine Sulfate, when dried, contains not less than 98.5% of $C_{10}H_{22}N_4.H_2SO_4$.

**Description** Guanethidine Sulfate occurs as white crystals or crystalline powder. It is odorless or has a slight, characteristic odor and a bitter taste.

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

Melting point: 251 – 256°C (an evacuated sealed capillary tube, with decomposition).

**Identification** (1) To 4 mL of a solution of Guanethidine Sulfate (1 in 4000) add 2 mL of 1-naphthol TS, 1 mL of diacetyl TS and 15 mL of water, and allow to stand for 30 minutes: a red color develops.

(2) Determine the infrared absorption spectrum of Guanethidine Sulfate, 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 Guanethidine Sulfate (1 in 10) responds to the Qualitative Tests for sulfate.

**pH** Dissolve 1.0 g of Guanethidine Sulfate in 50 mL of water: the pH of the solution is between 4.7 and 5.7.

**Purity** (1) Clarity and color of solution—Dissolve 1.0 g of Guanethidine Sulfate in 50 mL of water: the solution is clear and colorless.

(2) Methylisothiourea sulfate—Dissolve 2.0 g of Guanethidine Sulfate in 80 mL of sodium hydroxide TS, and allow to stand for 10 minutes. Add 60 mL of hydrochloric acid, 2 g of sodium bromide and water to make 200 mL. Then, to this solution add 0.70 mL of 1/60 mol/L potassium bromate VS and 2 mL of zinc iodide-starch paste TS: a blue color develops.

(3) Heavy metals—Proceed with 2.0 g of Guanethidine Sulfate according to Method 4, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 10 ppm).

**Loss on drying** Not more than 0.5% (1 g, 105°C, 4 hours).

**Residue on ignition** Not more than 0.20% (1 g).

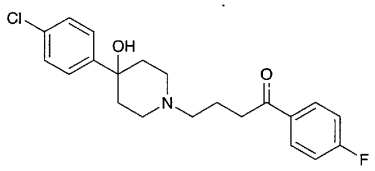
**Assay** Weigh accurately about 0.5 g of Guanethidine Sulfate, previously dried, dissolve in 2 mL of formic acid, add 70 mL of a mixture of acetic anhydride and acetic acid (100) (6:1), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 29.639 mg of $C_{10}H_{22}N_4.H_2SO_4$

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

# Haloperidol

ハロペリドール



$C_{21}H_{23}ClFNO_2$: 375.86
4-[4-(4-Chlorophenyl)-4-hydroxypiperidin-1-yl]-1-(4-fluorophenyl)butan-1-one [*52-86-8*]

Haloperidol, when dried, contains not less than 99.0% of $C_{21}H_{23}ClFNO_2$.

**Description** Haloperidol occurs as white to pale yellow crystals or powder. It is odorless.

It is freely soluble in acetic acid (100), soluble in chloroform, sparingly soluble in methanol and in ethanol (95), slightly soluble in 2-propanol and in diethyl ether, and practically insoluble in water.

**Identification** (1) Transfer 0.02 g of Haloperidol and 0.05 g of sodium to a test tube, and heat gradually and cautiously to ignite. Cool, add 0.5 mL of methanol and 5 mL of water, and heat to boil. Filter the solution, acidify the filtrate with 2 to 3 drops of hydrochloric acid, then add 2 drops of zirconyl-alizarin red S TS: the red-purple color of the test solution disappears, and a pale yellow color develops.

(2) Dissolve 0.1 g of Haloperidol in 30 mL of diluted