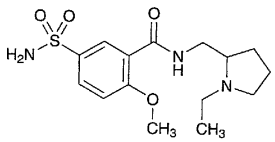
Containers and storage Containers—Well-closed containers.

Sulpiride

スルピリド



$C_{15}H_{23}N_3O_4S$: 341.43
N-(1-Ethylpyrolidin-2-ylmethyl)-2-methoxy-5-sulfamoylbenzamide [*15676-16-1*]

Sulpiride, when dried, contains not less than 98.5% of $C_{15}H_{23}N_3O_4S$.

Description Sulpiride is a white, crystalline powder. It is odorless.

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

It is soluble in dilute hydrochloric acid and in 0.05 mol/L sulfuric acid TS.

Melting point: 175 - 182°C (with decomposition).

Identification (1) Dissolve 0.01 g of Sulpiride in 5 mL of dilute hydrochloric acid and 20 mL of water. To 5 mL of this solution add 1 mL of Dragendorff's TS: a reddish orange precipitate is produced.

(2) To 0.5 g of Sulpiride add 3 mL of sodium hydroxide solution (3 in 10), and heat: the gas evolved changes moistened red litmus paper to blue.

(3) Dissolve 0.1 g of Sulpiride in 0.05 mol/L sulfuric acid to make 100 mL. Dilute 5 mL of the solution with water to make 100 mL. Determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry, using water as the blank, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wavelengths.

Purity (1) Clarity of solution—Dissolve 2.0 g of Sulpiride in 7 mL of dilute acetic acid, and add water to make 20 mL: the solution is clear. Perform the test as directed under the Ultraviolet-visible Spectrophotometry, using water as the blank: the absorbance at a wavelength of 450 nm does not exceed 0.020.

(2) Heavy metals—Proceed with 2.0 g of Sulpiride as directed under Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 10 ppm).

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

(4) Related substances—Dissolve 0.050 g of Sulpiride in 10 mL of methanol, and use this solution as the sample solution. Dilute 1 mL of the sample solution, accurately measured, with methanol to make exactly 100 mL. Dilute 5 mL of this solution, accurately measured, with methanol to make exactly 10 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 20 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 1-butanol, water and acetic acid (100) (4:2:1) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): spots other than the principal spot from the sample solution have no more color than the spot from the standard solution. When the plate is exposed to iodine vapor for 30 minutes, the spots other than the principal spot from the sample solution have no more color than the spot from the standard solution.

Loss on drying Not more than 0.5% (1 g, 105°C, 3 hours).

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

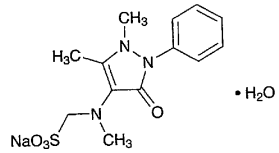
Assay Dissolve about 0.4 g of Sulpiride, previously dried and accurately weighed, in 80 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS (indicator: 2 drops of crystal violet TS) until the color of the solution changes from violet through blue to bluish green. Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 34.143 mg of $C_{15}H_{23}N_3O_4S$

Containers and storage Containers—Well-closed containers.

Sulpyrine

スルピリン



$C_{13}H_{16}N_3NaO_4S.H_2O$: 351.35
Monosodium [(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)methylamino]methanesulfonate monohydrate [*5907-38-0*]

Sulpyrine contains not less than 98.5% of $C_{13}H_{16}N_3NaO_4S$ (mol. wt.: 333.34), calculated on the dried basis.

Description Sulpyrine occurs as white to light yellow crystals or crystalline powder. It is odorless, and has a bitter taste.

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

It is colored by light.

Identification (1) Add 2 drops of dilute sulfuric acid and 1 mL of chlorinated lime TS to 3 mL of a solution of Sulpyrine (1 in 15): a deep blue color develops at first, but the color immediately turns red, then gradually changes to yellow.

(2) Boil 5 mL of a solution of Sulpyrine (1 in 25) with 3 mL of dilute hydrochloric acid: the odor of sulfur dioxide is perceptible at first, and on further boiling, the odor of formaldehyde is perceptible.

(3) A solution of Sulpyrine (1 in 10) responds to the Qualitative Tests for sodium salt.

Purity (1) Clarity of solution, and acidity or alkalinity—Dissolve 1.0 g of Sulpyrine in 10 mL of water: the solution is clear and neutral.

(2) Sulfate—Dissolve 0.20 g of Sulpyrine in 0.05 mol/L hydrochloric acid VS to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution with 0.50 mL of 0.005 mol/L sulfuric acid VS and 0.05 mol/L hydrochloric acid VS to make 50 mL (not more than 0.120%).

(3) Heavy metals—Proceed with 1.0 g of Sulpyrine according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(4) Merbuline—Transfer 0.10 g of Sulpyrine with 2 mL of water and 1 mL of dilute sulfuric acid into a flask, cover with a funnel, and boil gently for 15 minutes. Cool, add 2 mL of a solution of sodium acetate trihydrate (1 in 2) and water to make 5 mL, shake this solution with 5 mL of benzaldehyde-saturated solution, and allow to stand for 5 minutes: the solution is clear.

(5) Chloroform-soluble substances—Mix, by frequent shaking, 1.0 g of Sulpyrine and 10 mL of chloroform for 30 minutes. Collect the precipitate, wash with two 5-mL portions of chloroform, combine the washings with the filtrate, and evaporate on a water bath to dryness. Dry the residue at 105°C for 4 hours: the mass of the residue is not more than 5.0 mg.

Loss on drying Not more than 6.0% (1 g, 105°C, 4 hours).

Assay Weigh accurately about 0.25 g of Sulpyrine, dissolve in 100 mL of diluted hydrochloric acid (1 in 20), previously cooled below 10°C. Titrate immediately with 0.05 mol/L iodine VS while keeping the temperature between 5°C and 10°C, until the color of the solution remains blue upon shaking vigorously for 1 minute after the addition of 0.05 mol/L iodine VS (indicator: 1 mL of starch TS).

Each mL of 0.05 mol/L iodine VS
= 16.667 mg of $C_{13}H_{16}N_3NaO_4S$

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

Sulpyrine Injection

スルピリン注射液

Sulpyrine Injection is an aqueous solution for injection. It contains not less than 95% and not more than 105% of the labeled amount of sulpyrine ($C_{13}H_{16}N_3NaO_4S.H_2O$: 351.35).

Method of preparation Prepare as directed under Injections, with Sulpyrine.

Description Sulpyrine Injection is a clear, colorless or pale yellow liquid.
pH: 5.0 – 8.5

Identification (1) To a volume of Sulpyrine Injection, equivalent to 0.2 g of Sulpyrine according to the labeled amount, add water to make 3 mL, then add 2 drops of dilute sulfuric acid and 1 mL of chlorinated lime TS: a deep blue color develops at first, and the color immediately turns red and gradually changes to yellow.

(2) To a volume of Sulpyrine Injection, equivalent to 0.2 g of Sulpyrine according to the labeled amount, add water to make 5 mL, and boil this solution with 3 mL of dilute hydrochloric acid: the odor of sulfur dioxide is perceptible at first, and on further boiling the odor of formaldehyde is perceptible.

Assay Pipet 2 mL of Sulpyrine Injection, dilute with water to exactly 100 mL. Measure exactly a volume (V mL) of this solution, equivalent to about 0.05 g of sulpyrine ($C_{13}H_{16}N_3NaO_4S.H_2O$), and add water to make exactly 100 mL. Pipet 5 mL of this solution, add water to exactly 100 mL, and use this solution as the sample solution. Weigh accurately about 0.05 g of sulpyrine for assay (previously dry at 105°C for 4 hours, and weigh the loss on drying), and dissolve in water to make exactly 100 mL. Pipet 5 mL of this solution, add water to exactly 100 mL, and use this solution as the standard solution.

Pipet 2 mL each of the sample solution and the standard solution into separate 25-mL volumetric flasks, add 5 mL of ethanol (95), 2 mL of a solution of 4-dimethylaminocinnamaldehyde in ethanol (95) (1 in 250) and 2 mL of acetic acid (100) to each of these solutions, shake well, allow to stand for 15 minutes, and add water to exactly 25 mL. Perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry, using a solution prepared with 2 mL of water in the same manner as the blank. Determine the absorbances, A_T and A_S, of the subsequent solutions of the sample solution and the standard solution at 510 nm.

Amount (mg) of sulpyrine ($C_{13}H_{16}N_3NaO_4S.H_2O$) in 1 mL of Sulpyrine Injection
= amount (mg) of sulpyrine for assay, calculated on the dried basis

$$\times \frac{A_T}{A_S} \times \frac{50}{V} \times 1.0540$$

Containers and storage Containers—Hermetic containers, and colored containers may be used.

Storage—Light-resistant, and under nitrogen atmosphere.