

(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).

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

**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 ( $C_{13}H_{16}N_3NaO_4S \cdot H_2O$ : 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 ( $C_{13}H_{16}N_3NaO_4S \cdot H_2O$ ), 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 ( $C_{13}H_{16}N_3NaO_4S \cdot H_2O$ )  
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.