

Sulfinpyrazone Tablets

スルフィンピラゾン錠

Sulfinpyrazone Tablets contain not less than 93% and not more than 107% of the labeled amount of sulfinpyrazone ($C_{23}H_{20}N_2O_3S$; 404.48).

Method of preparation Prepare as directed under Tablets, with Sulfinpyrazone.

Identification (1) Weigh a portion of powdered Sulfinpyrazone Tablets, equivalent to 2 mg of Sulfinpyrazone according to the labeled amount, add 1 mL of acetic acid (100), and shake. To this solution add 1 mL of palladium (II) chloride TS and 2 mL of chloroform, and shake: a yellow color develops in the chloroform layer.

(2) Determine the absorption spectrum of the sample solution obtained in the Assay as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 257 nm and 261 nm.

Dissolution test Perform the test with 1 tablet of Sulfinpyrazone Tablets at 50 revolutions per minute according to Method 2 under the Dissolution Test, using 900 mL of the 2nd fluid under the Disintegration Test as the test solution. Take 20 mL or more of the dissolved solution 45 minutes after starting the test, and filter through a membrane filter with pore size of not more than $0.8 \mu\text{m}$. Discard the first 10 mL of the filtrate, pipet 2 mL of the subsequent, add the 2nd fluid to make exactly 20 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.01 g of Sulfinpyrazone Reference Standard, previously dried at 105°C for 2 hours, dissolve in the 2nd fluid to make exactly 100 mL. Pipet 10 mL of this solution, add the 2nd fluid to make exactly 100 mL, and use this solution as the standard solution. Determine the absorbances, A_T and A_S , of the sample solution and the standard solution at 259 nm as directed under the Ultraviolet-visible Spectrophotometry.

The dissolution rate of Sulfinpyrazone Tablets in 45 minutes is not less than 80%.

Dissolution rate (%) with respect to the labeled amount of sulfinpyrazone ($C_{23}H_{20}N_2O_3S$)

$$= W_S \times \frac{A_T}{A_S} \times 9$$

W_S : Amount (mg) of Sulfinpyrazone Reference Standard.

Assay Weigh accurately, and powder not less than 20 Sulfinpyrazone Tablets. Weigh accurately a portion of the powder, equivalent to about 0.05 g of sulfinpyrazone ($C_{23}H_{20}N_2O_3S$), add 25 mL of methanol, and shake for 15 minutes. To this solution add 50 mL of sodium hydroxide TS, shake, cool, and add water to make exactly 200 mL. After centrifuging, pipet 4 mL of the supernatant liquid, add water to make exactly 100 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.05 g of Sulfinpyrazone Reference Standard, previously dried at 105°C for 2 hours, dissolve in 25 mL of methanol, add 50 mL of sodium hydroxide TS, and cool. To this solution add water to make exactly 200 mL, pipet 4 mL of this solution, add water to make exactly 100 mL, and use this solution as the standard solution. Determine the absorbances, A_T and

A_S , of these solutions at 260 nm as directed under the Ultraviolet-visible Spectrophotometry.

Amount (mg) of sulfinpyrazone ($C_{23}H_{20}N_2O_3S$)
= amount (mg) of Sulfinpyrazone
Reference Standard

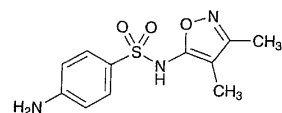
$$\times \frac{A_T}{A_S}$$

Containers and storage Containers—Well-closed containers.

Sulfisoxazole

Sulfafurazole

スルフィソキサゾール



$C_{11}H_{13}N_3O_3S$: 267.30

4-Amino-*N*-(3,4-dimethylisoxazol-5-yl)benzenesulfonamide [127-69-5]

Sulfisoxazole, when dried, contains not less than 99.0% of $C_{11}H_{13}N_3O_3S$.

Description Sulfisoxazole occurs as white crystals or crystalline powder. It is odorless, and has a slightly bitter taste.

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

It dissolves in dilute hydrochloric acid, in sodium hydroxide TS and in ammonia TS.

It is gradually colored by light.

Identification (1) Dissolve 0.01 g of Sulfisoxazole in 1 mL of dilute hydrochloric acid and 4 mL of water: the solution responds to the Qualitative Tests for primary aromatic amines.

(2) Dissolve 0.02 g of Sulfisoxazole in 5 mL of water and 1 mL of *n*-butylamine, add 2 to 3 drops of copper (II) sulfate TS, and shake well. Add 5 mL of chloroform, shake, and allow to stand: a blue-green color develops in the chloroform layer.

(3) Dissolve 0.01 g of Sulfisoxazole in 1 mL of pyridine, add 2 drops of copper (II) sulfate TS, and shake. Add 3 mL of water and 5 mL of chloroform, shake, and allow to stand: a light yellow-brown color develops in the chloroform layer.

(4) To 0.5 g of Sulfisoxazole add 2 mL of acetic acid (100), dissolve by heating under a reflux condenser, add 1 mL of acetic anhydride, and boil for 10 minutes. Add 10 mL of water, cool, and alkalize with about 7 mL of a solution of sodium hydroxide (3 in 10). Filter, if necessary, immediately acidify by adding acetic acid (100) dropwise, collect the produced precipitate, recrystallize from methanol, and dry at 105°C for 1 hour: the crystals melt between 208°C and 210°C .

Melting point 192 – 196°C (with decomposition).

Purity (1) Clarity and color of solution—Dissolve 1.0 g of Sulfisoxazole in 5 mL of sodium hydroxide TS and 20 mL of water: the solution is clear and colorless to pale yellow.

(2) Acid—To 1.0 g of Sulfisoxazole add 50 mL of water, warm at 70°C for 5 minutes, allow to stand in an ice bath for 1 hour, and filter. To 25 mL of the filtrate add 2 drops of methyl red TS and 0.20 mL of 0.1 mol/L sodium hydroxide VS: a yellow color develops.

(3) Heavy metals—Proceed with 1.0 g of Sulfisoxazole 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).

Loss on drying Not more than 0.5% (2 g, 105°C, 4 hours).

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

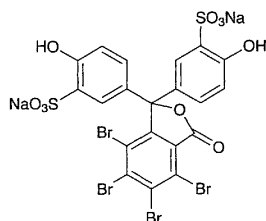
Assay Weigh accurately about 1 g of Sulfisoxazole, previously dried, dissolve in 50 mL of methanol by warming, cool and titrate with 0.2 mol/L sodium hydroxide VS (indicator: 3 drops of phenolphthalein TS). Perform a blank determination using a mixture of 50 mL of methanol and 18 mL of water, and make any necessary correction.

$$\begin{aligned} \text{Each mL of 0.2 mol/L sodium hydroxide VS} \\ = 53.46 \text{ mg of } C_{11}H_{13}N_3O_3S \end{aligned}$$

Containers and storage Containers—Well-closed containers.

Sulfobromophthalein Sodium

スルホブロモフタレインナトリウム



$C_{20}H_8Br_4Na_2O_{10}S_2$: 838.00

Disodium 5,5'-(4,5,6,7-tetrabromo-1,3-dihydro-3-oxo-isobenzofuranylidene)bis(2-hydroxybenzenesulfonate) [71-67-0]

Sulfobromophthalein Sodium, when dried, contains not less than 96.0% and not more than 104.0% of $C_{20}H_8Br_4Na_2O_{10}S_2$.

Description Sulfobromophthalein Sodium occurs as a white, crystalline powder. It is odorless.

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

It is hygroscopic.

Identification (1) Dissolve 0.02 g of Sulfobromophthalein Sodium in 10 mL of water, and add 1 mL of sodium carbonate TS: a blue-purple color is produced. Add 1 mL of dilute hydrochloric acid to the solution: the color of

the solution disappears.

(2) Transfer 0.2 g of Sulfobromophthalein Sodium to a porcelain crucible, mix well with 0.5 g of anhydrous sodium carbonate, and ignite until the mixture is charred. After cooling, add 15 mL of hot water to the residue, heat for 5 minutes on a water bath, filter, and render the filtrate slightly acid with hydrochloric acid: the solution responds to the Qualitative Tests for bromide, and the Qualitative Tests (1) and (2) for sulfate.

(3) Sulfobromophthalein Sodium responds to the Qualitative Tests (1) for sodium salt.

pH The pH of a solution of 1.0 g of Sulfobromophthalein Sodium in 20 mL of water is between 4.0 and 5.5.

Purity (1) Clarity and color of solution—Dissolve 0.5 g of Sulfobromophthalein Sodium in 10 mL of water: the solution is clear and colorless to pale yellow.

(2) Chloride—Perform the test with 2.0 g of Sulfobromophthalein Sodium. Prepare the control solution with 0.10 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.002%).

(3) Sulfate—To 10 mL of a solution of Sulfobromophthalein Sodium (1 in 500) add 5 drops of dilute hydrochloric acid, heat to boil, and add 1 mL of hot barium chloride TS: the solution is clear when observed 1 minute after the addition of the barium chloride TS.

(4) Calcium—Weigh accurately about 5 g of Sulfobromophthalein Sodium, transfer to a porcelain dish, heat gently to char, and heat strongly between 700°C and 750°C until the residue is incinerated. After cooling, add 10 mL of dilute hydrochloric acid, and heat for 5 minutes on a water bath. Transfer the contents to a flask with 50 mL of water, and add 5 mL of 8 mol/L potassium hydroxide TS and 0.1 g of NN indicator. Titrate with 0.01 mol/L disodium dihydrogen ethylenediamine tetraacetate VS until the red-purple color of the solution changes to blue.

$$\begin{aligned} \text{Each mL of 0.01 mol/L disodium dihydrogen} \\ \text{ethylenediamine tetraacetate VS} \\ = 0.4008 \text{ mg of Ca} \end{aligned}$$

The content of calcium (Ca: 40.08) is not more than 0.05%.

(5) Heavy metals—Proceed with 1.0 g of Sulfobromophthalein Sodium 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).

(6) Arsenic—Transfer 0.65 g of Sulfobromophthalein Sodium to a crucible, add 10 mL of a solution of magnesium nitrate hexahydrate in ethanol (95) (1 in 50), fire to burn, then heat gently until the residue is incinerated. If any carbon remains, moisten the residue with a small amount of nitric acid, and incinerate again by ignition. After cooling, add 10 mL of dilute sulfuric acid, and heat until white fumes are evolved. After cooling, add 5 mL of water to the residue, and perform the test using Apparatus B with this solution as the test solution (not more than 3.1 ppm).

Loss on drying Not more than 5.0% (0.5 g, 105°C, 3 hours).

Residue on ignition 14.0 – 19.0% (after drying, 0.5 g, 700 – 750°C).

Assay Dissolve about 0.1 g of Sulfobromophthalein Sodium, previously dried and accurately weighed, in water to