

Sulfamethoxazole, when dried, contains not less than 99.0% of $C_{10}H_{11}N_3O_3S$.

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

It is very soluble in *N,N*-dimethylformamide, sparingly soluble in ethanol (95), slightly soluble in diethyl ether, and very slightly soluble in water.

It dissolves in sodium hydroxide TS.

It is gradually colored by light.

Identification Determine the infrared absorption spectrum of Sulfamethoxazole, 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.

Melting point 169 – 172°C

Purity (1) Clarity and color of solution—Dissolve 1.0 g of Sulfamethoxazole in 5 mL of sodium hydroxide TS, and add 20 mL of water: the solution is clear and colorless.

(2) Acid—To 1.0 g of Sulfamethoxazole add 50 mL of water, heat at 70°C for 5 minutes, allow to stand in ice water for 1 hour, and filter. To 25 mL of the filtrate add 2 drops of methyl red TS and 0.60 mL of 0.1 mol/L sodium hydroxide VS: a yellow color develops.

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

(5) Related substances—Dissolve 0.20 g of Sulfamethoxazole in 10 mL of a solution of ammonia solution (28) in methanol (1 in 50), and use this solution as the sample solution. Pipet 1 mL of the sample solution, add a solution of ammonia solution (28) in methanol (1 in 50) to make exactly 10 mL. Pipet 1 mL of this solution, add a solution of ammonia solution (28) in methanol (1 in 50) to make exactly 20 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5 μ L 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 ethyl acetate, acetonitrile and diluted ammonia solution (28) (7 in 100) (10:8:1) 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 0.5% (1 g, 105°C, 4 hours).

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

Assay Weigh accurately about 0.4 g of Sulfamethoxazole, previously dried, dissolve in 30 mL of *N,N*-dimethylformamide, add 10 mL of water, and titrate with 0.1 mol/L sodium hydroxide VS until a light blue color is produced (indicator: 0.5 mL of thymolphthalein TS). Separately, perform a blank determination in the same manner with a mixture of 30 mL of *N,N*-dimethylformamide and 26 mL of water, and make any necessary correction.

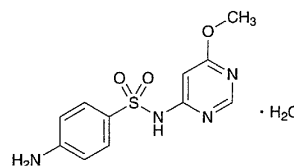
Each mL of 0.1 mol/L sodium hydroxide VS
= 25.328 mg of $C_{10}H_{11}N_3O_3S$

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Sulfamonomethoxine

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$C_{11}H_{12}N_4O_3S \cdot H_2O$: 298.32

4-Amino-*N*-(6-methoxypyrimidin-4-yl)benzenesulfonamide monohydrate [1220-83-3, anhydride]

Sulfamonomethoxine, when dried, contains not less than 99.0% of $C_{11}H_{12}N_4O_3S$ (mol. wt.: 280.31).

Description Sulfamonomethoxine occurs as white to pale yellow crystals, granules or crystalline powder. It is odorless.

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

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

It is gradually colored by light.

Identification Determine the infrared absorption spectrum of Sulfamonomethoxine 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.

Melting point 204 – 206°C

Purity (1) Clarity and color of solution—Dissolve 1.0 g of Sulfamonomethoxine in 5 mL of sodium hydroxide TS and 20 mL of water: the solution is clear and colorless to pale yellow. Dissolve 0.5 g of Sulfamonomethoxine in 5 mL of sodium hydroxide TS, and heat: no turbidity is produced. After cooling, add 5 mL of acetone: the solution is clear.

(2) Heavy metals—Proceed with 1.0 g of Sulfamonomethoxine 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).

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

(4) Related substances—Dissolve 0.02 g of Sulfamonomethoxine in ethanol (95) to make 10 mL, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add ethanol (95) to make exactly 200 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 with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of 1-butanol and ammonia solution (28) (4:1) 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 4.5 – 6.5% (1 g, 105°C, 4 hours).

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

Assay Weigh accurately about 0.5 g of Sulfinpyrazone, previously dried, dissolve in 5 mL of hydrochloric acid and 50 mL of water, add 10 mL of a solution of potassium bromide (3 in 10), cool below 15°C, and titrate with 0.1 mol/L sodium nitrite VS according to the potentiometric titration method or the amperometric titration method under the Endpoint Detection Methods in Titrimetry.

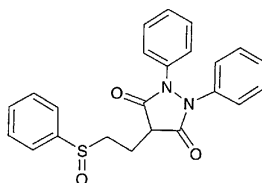
Each mL of 0.1 mol/L sodium nitrite VS
= 28.031 mg of $\text{C}_{11}\text{H}_{12}\text{N}_4\text{O}_3\text{S}$

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Sulfinpyrazone

スルフィンピラゾン



$\text{C}_{23}\text{H}_{20}\text{N}_2\text{O}_3\text{S}$: 404.48
1,2-Diphenyl-4-(2-phenylsulfinylethyl)pyrazolidine-3,5-dione [57-96-5]

Sulfinpyrazone, when dried, contains not less than 98.5% of $\text{C}_{23}\text{H}_{20}\text{N}_2\text{O}_3\text{S}$.

Description Sulfinpyrazone occurs as a white to pale yellowish white powder. It is odorless, and has a bitter taste.

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

It dissolves in sodium hydroxide TS.

Melting point: about 138°C (with decomposition).

Identification (1) Dissolve 2 mg of Sulfinpyrazone in 1 mL of acetic acid (100), 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 a solution of Sulfinpyrazone in 0.01 mol/L sodium hydroxide TS (1 in 100,000) as directed under the Ultraviolet-visible Spec-

trophotometry, and compare the spectrum with the Reference Spectrum or the spectrum of a solution of Sulfinpyrazone Reference Standard prepared in the same manner as the sample solution: both spectra exhibit similar intensities of absorption at the same wavelengths.

(3) Determine the infrared absorption spectrum of Sulfinpyrazone, 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) Clarity and color of solution—Dissolve 0.5 g of Sulfinpyrazone in 10 mL of acetone: the solution is clear and colorless. Dissolve 0.5 g of Sulfinpyrazone in 10 mL of sodium hydroxide TS: the solution is clear and colorless.

(2) Heavy metals—Proceed with 2.0 g of Sulfinpyrazone 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).

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

(4) Related substances—Dissolve 0.10 g of Sulfinpyrazone in 5 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 (1). Pipet 1 mL of the sample solution, add acetone to make exactly 200 mL, and use this solution as the standard solution (2). Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot rapidly 5 μL each of the sample solution and the standard solutions (1) and (2) on a plate of silica gel with fluorescent indicator for thin-layer chromatography under a stream of nitrogen. Develop the plate with a mixture of chloroform and acetic acid (100) (4:1) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the most intense spot other than the principal spot from the sample solution is not more intense than the spot from the standard solution (1), and the spots other than the principal and above spots from the sample solution are not more intense than the spot from the standard solution (2).

Loss on drying Not more than 0.5% (1 g, 105°C, 2 hours).

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

Assay Weigh accurately about 0.5 g of Sulfinpyrazone, previously dried, dissolve in 40 mL of acetone, add 40 mL of water, and titrate with 0.1 mol/L sodium hydroxide VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L sodium hydroxide VS
= 40.45 mg of $\text{C}_{23}\text{H}_{20}\text{N}_2\text{O}_3\text{S}$

Containers and storage Containers—Well-closed containers.