

ver.

Gas: Combustible gas—Acetylene

Supporting gas—Air

Lamp: A silver hollow cathode lamp

Wavelength: 328.1 nm

Assay Weigh accurately about 0.1 g each of Sulfadiazine Silver and Sulfadiazine Silver Reference Standard, each previously dried, and add ammonia TS to make exactly 100 mL, respectively. Pipet 1 mL each of these solutions, add water to make exactly 100 mL, and use these solutions as the sample solution and the standard solution, respectively. Determine the absorbances, A_T and A_S , of the sample solution and the standard solution at 255 nm, using a solution, prepared with exactly 1 mL of ammonia TS and a sufficient water to make exactly 100 mL, as the blank.

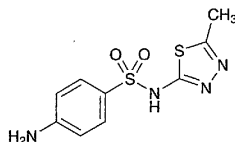
$$\begin{aligned} &\text{Amount (mg) of } C_{10}H_9AgN_4O_2S \\ &= \text{amount (mg) of Sulfadiazine Silver} \\ &\quad \text{Reference Standard} \\ &\quad \times \frac{A_T}{A_S} \end{aligned}$$

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Sulfamethizole

スルファメチゾール



$C_9H_{10}N_4O_2S_2$: 270.33
4-Amino-*N*-(5-methyl-1,3,4-thiadiazol-2-yl)-benzenesulfonamide [144-82-1]

Sulfamethizole, when dried, contains not less than 99.0% of $C_9H_{10}N_4O_2S_2$.

Description Sulfamethizole occurs as white to yellowish white crystals or crystalline powder. It is odorless.

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

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

It is gradually colored by light.

Identification Determine the infrared absorption spectrum of Sulfamethizole, 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 208 – 211°C

Purity (1) Clarity and color of solution—Dissolve 0.5 g of Sulfamethizole in 3 mL of sodium hydroxide TS and 20 mL of water: the solution is clear and colorless.

(2) Acid—To 1.0 g of Sulfamethizole add 50 mL of water, warm at 70°C for 5 minutes, allow to stand for 1 hour in an ice bath, 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 Sulfamethizole 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 Sulfamethizole according to Method 3, and perform the test using Apparatus B (not more than 2 ppm).

(5) Related substances—Dissolve 0.10 g of Sulfamethizole in acetone to make 10 mL, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add acetone to make exactly 50 mL, then pipet 5 mL of this solution, add acetone 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 and acetic acid (100) (20:1) 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 Sulfamethizole, 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.

$$\begin{aligned} &\text{Each mL of 0.1 mol/L sodium nitrite VS} \\ &= 27.034 \text{ mg of } C_9H_{10}N_4O_2S_2 \end{aligned}$$

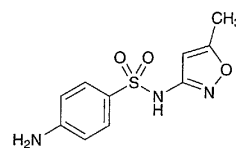
Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Sulfamethoxazole

Sulfisomezole

スルファメトキサゾール



$C_{10}H_{11}N_3O_3S$: 253.28
4-Amino-*N*-(5-methylisoxazol-3-yl)benzenesulfonamide [723-46-6]

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.

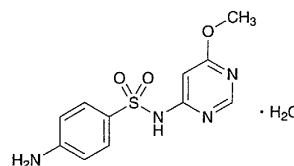
Each mL of 0.1 mol/L sodium hydroxide VS
= 25.328 mg of $C_{10}H_{11}N_3O_3S$

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Sulfamonomethoxine

スルファモノメトキシシ



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