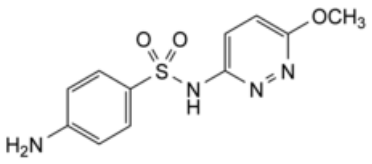
Edition: BP 2025 (Ph. Eur. 11.6 update)

Sulfametoxypyridazine

General Notices

(Sulfamethoxypyridazine for Veterinary Use, Ph. Eur. monograph 0638)



$C_{11}H_{12}N_4O_3S$ 280.3 *80-35-3*

Action and use

Sulfonamide antibacterial.

Ph Eur

DEFINITION

4-Amino-*N*-(6-methoxypyridazin-3-yl)benzene-1-sulfonamide.

Content

99.0 per cent to 101.0 per cent (dried substance).

CHARACTERS

Appearance

White or slightly yellowish, crystalline powder, colouring slowly on exposure to light.

Solubility

Practically insoluble in water, sparingly soluble in acetone, slightly soluble in ethanol (96 per cent), very slightly soluble in methylene chloride. It dissolves in solutions of alkali hydroxides and in dilute mineral acids.

mp

About 180 °C, with decomposition.

IDENTIFICATION

First identification: A, B.

Second identification: B, C, D.

A. Infrared absorption spectrophotometry (2.2.24).

Comparison sulfamethoxypyridazine CRS.

B. Examine the chromatograms obtained in the test for related substances. The principal spot in the chromatogram obtained with test solution (b) is similar in position and size to the principal spot in the chromatogram obtained with reference solution (a).
C. Dissolve 0.5 g in 1 mL of a 40 per cent *V/V* solution of sulfuric acid R, heating gently. Continue heating until a crystalline precipitate appears (about 2 min). Cool and add 10 mL of dilute sodium hydroxide solution R. Cool again, add 25 mL of ether R and shake the solution for 5 min. Separate the upper layer, dry over anhydrous sodium sulfate R and filter. Evaporate the ether by heating in a water-bath. An oily residue is obtained which becomes crystalline on cooling; if necessary, scratch the wall of the container with a glass rod. The residue melts (2.2.14) at 102 °C to 106 °C.
D. Dissolve about 5 mg in 10 mL of a 103 g/L solution of hydrochloric acid R. Dilute 1 mL of the solution to 10 mL with water R. The solution, without further acidification, gives the reaction of primary aromatic amines (2.3.1).

TESTS

Appearance of solution

The solution is clear (2.2.1) and not more intensely coloured than reference solution Y_4 or BY_4 (2.2.2, Method II).

Dissolve 1.0 g in a mixture of 10 mL of a 40 g/L solution of sodium hydroxide R and 15 mL of water R.

Acidity

To 1.25 g, finely powdered, add 25 mL of carbon dioxide-free water R. Heat at 70 °C for 5 min. Cool in iced water for about 15 min and filter. To 20 mL of the filtrate add 0.1 mL of bromothymol blue solution R1. Not more than 0.5 mL of 0.1 M sodium hydroxide is required to change the colour of the indicator.

Related substances

Thin-layer chromatography (2.2.27).

Test solution (a) Dissolve 0.10 g of the substance to be examined in acetone R and dilute to 5 mL with the same solvent.

Test solution (b) Dilute 1 mL of test solution (a) to 10 mL with acetone R.

Reference solution (a) Dissolve 20 mg of sulfamethoxypyridazine CRS in acetone R and dilute to 10 mL with the same solvent.

Reference solution (b) Dilute 2.5 mL of test solution (b) to 50 mL with acetone R.

Plate TLC silica gel GF_{254} plate R.

Mobile phase dilute ammonia R1, water R, 2-propanol R, ethyl acetate R (1:9:30:50 *V/V/V/V*).

Application 5 µL.

Development Over a path of 15 cm.

Drying At 100-105 °C.

Detection Examine in ultraviolet light at 254 nm.

Results Any spot in the chromatogram obtained with test solution (a), apart from the principal spot, is not more intense than the spot in the chromatogram obtained with reference solution (b) (0.5 per cent).

Loss on drying (2.2.32)

Maximum 0.5 per cent, determined on 1.000 g by drying in an oven at 105 °C.

Sulfated ash (*2.4.14*)

Maximum 0.1 per cent, determined on 1.0 g.

ASSAY

Carry out the determination of primary aromatic amino-nitrogen (*2.5.8*), using 0.250 g, determining the end-point electrometrically.

1 mL of *0.1 M sodium nitrite* is equivalent to 28.03 mg of $C_{11}H_{12}N_4O_3S$.

STORAGE

Protected from light.

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