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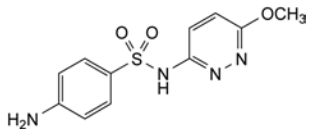
SULFAMETHOXYPYRIDAZINE FOR VETERINARY USE

Sulfamethoxypyridazinum ad usum veterinarium



$C_{11}H_{12}N_4O_3S$ M_r 280.3
[80-35-3]

DEFINITION

Sulfamethoxypyridazine for veterinary use contains not less than 99.0 per cent and not more than the equivalent of 101.0 per cent of 4-amino-*N*-(6-methoxypyridazin-3-yl)benzenesulfonamide, calculated with reference to the dried substance.

CHARACTERS

A white or slightly yellowish, crystalline powder, colouring slowly on exposure to light, practically insoluble in water, sparingly soluble in acetone, slightly soluble in alcohol, very slightly soluble in methylene chloride. It dissolves in solutions of alkali hydroxides and in dilute mineral acids.

It melts at about 180 °C, with decomposition.

IDENTIFICATION

First identification: A, B.

Second identification: B, C, D.

A. Examine by infrared absorption spectrophotometry (*2.2.24*), comparing with the spectrum obtained with *sulfamethoxypyridazine CRS*. Examine the substances prepared as discs.

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 ether 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 °Cto 106 °C.

D. Dissolve about 5 mg in 10 mL of *1 M hydrochloric acid*. 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. Dissolve 1.0 g in a mixture of 10 mL of *1 M sodium hydroxide* and 15 mL of *water R*. 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*).

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. Examine by thin layer chromatography (*2.2.27*), using *TLC silica gel GF_{254} plate R*.

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*.

Apply separately to the plate 5 µL of each solution. Develop over a path of 15 cm using a mixture of 1 volume of *dilute ammonia R1*, 9 volumes of *water R*, 30 volumes of *2-propanol R* and 50 volumes of *ethyl acetate R*. Dry the plate at 100-105 °C and examine in ultraviolet light at 254 nm. 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).

Heavy metals (*2.4.8*). 1.0 g complies with limit test D for heavy metals (20 ppm). Prepare the standard using 2 mL of *lead standard solution (10 ppm Pb) R*.

Loss on drying (*2.2.32*). Not more than 0.5 per cent, determined on 1.000 g by drying in an oven at 105 °C.

Sulfated ash (*2.4.14*). Not more than 0.1 per cent, determined on 1.0 g.

ASSAY

Carry out the assay of primary aromatic amino-nitrogen (*2.5.8*), using 0.2500 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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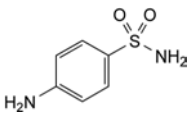
SULFANILAMIDE

Sulfanilamidum



$C_6H_8N_2O_2S$ M_r 172.2
[63-74-1]

DEFINITION

Sulfanilamide contains not less than 99.0 per cent and not more than the equivalent of 101.0 per cent of 4-aminobenzenesulfonamide, calculated with reference to the dried substance.

CHARACTERS

White or yellowish-white crystals or fine powder, slightly soluble in water, freely soluble in acetone, sparingly soluble in alcohol, practically insoluble in methylene chloride. It dissolves in solutions of alkali hydroxides and in dilute mineral acids.

IDENTIFICATION

First identification: B.

Second identification: A, C, D.

A. Melting point (*2.2.14*): 164.5 °C to 166.0 °C.