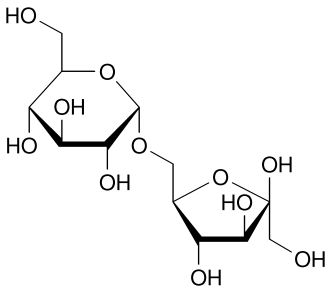
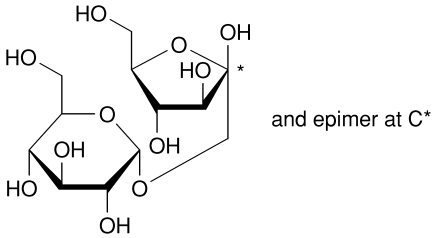


A. 6-*O*-α-D-glucopyranosyl-β-D-*arabino*-hex-2-ulofuranose (isomaltulose),

B. mannitol,

C. sorbitol,

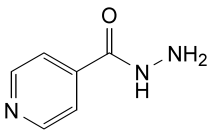


D. 1-*O*-α-D-glucopyranosyl-D-*arabino*-hex-2-ulofuranose (trehalulose).

01/2008:0146
corrected 6.0

ISONIAZID

Isoniazidum



$C_6H_7N_3O$ M_r 137.1
[54-85-3]

DEFINITION

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

CHARACTERS

A white or almost white, crystalline powder or colourless crystals, freely soluble in water, sparingly soluble in alcohol.

IDENTIFICATION

First identification: A, B.

Second identification: A, C.

A. Melting point (*2.2.14*): 170 °C to 174 °C.

B. Examine by infrared absorption spectrophotometry (*2.2.24*), comparing with the spectrum obtained with *isoniazid CRS*.

C. Dissolve 0.1 g in 2 ml of *water R* and add 10 ml of a warm 10 g/l solution of *vanillin R*. Allow to stand and scratch the wall of the test tube with a glass rod. A yellow precipitate is formed, which, after recrystallisation from 5 ml of *alcohol (70 per cent V/V) R* and drying at 100 °C to 105 °C, melts (*2.2.14*) at 226 °C to 231 °C.

TESTS

Solution S. Dissolve 2.5 g in *carbon dioxide-free water R* and dilute to 50 ml with the same solvent.

Appearance of solution. Solution S is clear (*2.2.1*) and not more intensely coloured than reference solution BY_7 (*2.2.2, Method II*).

pH (*2.2.3*). The pH of solution S is 6.0 to 8.0.

Hydrazine and related substances. Examine by thin-layer chromatography (*2.2.27*), using *silica gel* GF_{254} *R* as the coating substance.

Test solution. Dissolve 1.0 g of the substance to be examined in a mixture of equal volumes of *acetone R* and *water R* and dilute to 10.0 ml with the same mixture of solvents.

Reference solution. Dissolve 50.0 mg of *hydrazine sulphate R* in 50 ml of *water R* and dilute to 100.0 ml with *acetone R*. To 10.0 ml of this solution add 0.2 ml of the test solution and dilute to 100.0 ml with a mixture of equal volumes of *acetone R* and *water R*.

Apply separately to the plate 5 µl of each solution and develop over a path of 15 cm using a mixture of 10 volumes of *water R*, 20 volumes of *acetone R*, 20 volumes of *methanol R* and 50 volumes of *ethyl acetate R*. Allow the plate to dry in air and examine in ultraviolet light at 254 nm. Any spot in the chromatogram obtained with the test solution, apart from the principal spot, is not more intense than the spot in the chromatogram obtained with the reference solution (0.2 per cent). Spray the plate with *dimethylaminobenzaldehyde solution R1*. Examine in daylight. An additional spot, corresponding to hydrazine, appears in the chromatogram obtained with the reference solution. Any corresponding spot in the chromatogram obtained with the test solution is not more intense than the spot corresponding to hydrazine in the chromatogram obtained with the reference solution (0.05 per cent).

Heavy metals (*2.4.8*). 2.0 g complies with limit test C for heavy metals (10 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 1.00 g by drying in an oven at 105 °C.

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

ASSAY

Dissolve 0.250 g in *water R* and dilute to 100.0 ml with the same solvent. To 20.0 ml of the solution add 100 ml of *water R*, 20 ml of *hydrochloric acid R*, 0.2 g of *potassium bromide R* and 0.05 ml of *methyl red solution R*. Titrate dropwise with *0.0167 M potassium bromate*, shaking continuously, until the red colour disappears.

1 ml of *0.0167 M potassium bromate* is equivalent to 3.429 mg of $C_6H_7N_3O$.