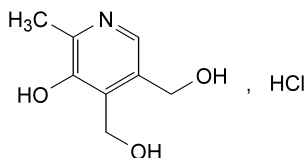


01/2008:0245
corrected 6.0

PYRIDOXINE HYDROCHLORIDE

Pyridoxini hydrochloridum

C₈H₁₂ClNO₃
[58-56-0]M_r 205.6

DEFINITION

Pyridoxine hydrochloride contains not less than 99.0 per cent and not more than the equivalent of 101.0 per cent of (5-hydroxy-6-methylpyridine-3,4-diyl)dimethanol hydrochloride, calculated with reference to the dried substance.

CHARACTERS

A white or almost white, crystalline powder, freely soluble in water, slightly soluble in alcohol.

It melts at about 205 °C, with decomposition.

IDENTIFICATION

First identification: B, D.

Second identification: A, C, D.

- A. Dilute 1.0 ml of solution S (see Tests) to 50.0 ml with 0.1 M hydrochloric acid (solution A). Dilute 1.0 ml of solution A to 100.0 ml with 0.1 M hydrochloric acid. Examined between 250 nm and 350 nm (2.2.25), the solution shows an absorption maximum at 288 nm to 296 nm. The specific absorbance at the maximum is 425 to 445. Dilute 1.0 ml of solution A to 100.0 ml with a mixture of equal volumes of 0.025 M potassium dihydrogen phosphate solution and 0.025 M disodium hydrogen phosphate solution (2.2.3). Examined between 220 nm and 350 nm, the solution shows 2 absorption maxima, at 248 nm to 256 nm and at 320 nm to 327 nm. The specific absorbances at the maxima are 175 to 195 and 345 to 365, respectively.
- B. Examine by infrared absorption spectrophotometry (2.2.24), comparing with the spectrum obtained with pyridoxine hydrochloride CRS.
- C. 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, colour and size to the principal spot in the chromatogram obtained with reference solution (a).
- D. Solution S gives reaction (a) of chlorides (2.3.1).

TESTS

Solution S. Dissolve 2.50 g in carbon dioxide-free water R and dilute to 50.0 ml with the same solvent.

Appearance of solution. Solution S is clear (2.2.1) and not more intensely coloured than reference solution Y₇ (2.2.2, Method II).

pH (2.2.3). The pH of solution S is 2.4 to 3.0.

Related substances. Examine by thin-layer chromatography (2.2.27), using a TLC silica gel G plate R.

Test solution (a). Dissolve 1.0 g of the substance to be examined in water R and dilute to 10 ml with the same solvent.

Test solution (b). Dilute 1 ml of test solution (a) to 10 ml with water R.

Reference solution (a). Dissolve 0.10 g of pyridoxine hydrochloride CRS in water R and dilute to 10 ml with the same solvent.

Reference solution (b). Dilute 2.5 ml of test solution (a) to 100 ml with water R. Dilute 1 ml of this solution to 10 ml with water R.

Apply to the plate 2 µl of each solution. Develop in an unsaturated tank over a path of 15 cm using a mixture of 9 volumes of concentrated ammonia R, 13 volumes of methylene chloride R, 13 volumes of tetrahydrofuran R and 65 volumes of acetone R. Allow the plate to dry in air and spray with a 50 g/l solution of sodium carbonate R in a mixture of 30 volumes of alcohol R and 70 volumes of water R. Dry the plate in a current of air, spray with a 1 g/l solution of dichloroquinonechlorimide R in alcohol R and examine the chromatograms immediately. 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) (0.25 per cent). Disregard any spots remaining on the starting line.

Heavy metals (2.4.8). 12 ml of solution S complies with limit test A (20 ppm). Prepare the standard using lead standard solution (1 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.

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

ASSAY

In order to avoid overheating in the reaction medium, mix thoroughly throughout and stop the titration immediately after the end-point has been reached.

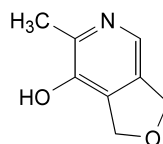
Dissolve 0.150 g in 5 ml of anhydrous formic acid R. Add 50 ml of acetic anhydride R. Titrate with 0.1 M perchloric acid, determining the end-point potentiometrically (2.2.20). Carry out a blank titration.

1 ml of 0.1 M perchloric acid is equivalent to 20.56 mg of C₈H₁₂ClNO₃.

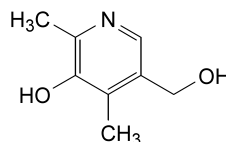
STORAGE

Store protected from light.

IMPURITIES



A. 6-methyl-1,3-dihydrofuro[3,4-c]pyridin-7-ol,



B. 5-(hydroxymethyl)-2,4-dimethylpyridin-3-ol.