Iron (*2.4.9*): maximum 30 ppm.

In a separating funnel, dissolve 0.33 g in 10 ml of *dilute hydrochloric acid R*. Shake with 3 quantities, each of 10 ml, of *methyl isobutyl ketone R1*, shaking for 3 min each time. To the combined organic layers add 10 ml of *water R* and shake for 3 min. The aqueous layer complies with the test.

Heavy metals (*2.4.8*): maximum 10 ppm.

12 ml of solution S complies with test A. Prepare the reference solution using *lead standard solution (1 ppm Pb) R*.

Loss on drying (*2.2.32*): maximum 0.5 per cent, determined on 1.000 g by drying in an oven at 60 °C for 3 h.

Sulphated ash (*2.4.14*): maximum 0.1 per cent, determined on 1.0 g.

ASSAY

Dissolve 80.0 mg in 3 ml of *anhydrous formic acid R*. Add 50 ml of *anhydrous acetic acid 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 10.31 mg of $C_8H_{18}N_2O_4$.

STORAGE

Protected from light.

IMPURITIES

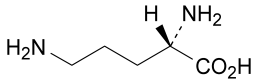
Specified impurities: A, B, C, D, E, F.

A. aspartic acid,

B. glutamic acid,

C. alanine,

D. valine,



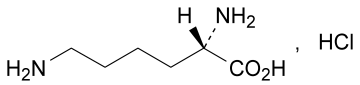
E. (2*S*)-2,5-diaminopentanoic acid (ornithine),

F. arginine.

01/2008:0930
corrected 6.0

LYSINE HYDROCHLORIDE

Lysini hydrochloridum



$C_6H_{15}ClN_2O_2$ M_r 182.7
[657-27-2]

DEFINITION

Lysine hydrochloride contains not less than 98.5 per cent and not more than the equivalent of 101.0 per cent of (*S*)-2,6-diaminohexanoic acid hydrochloride, calculated with reference to the dried substance.

CHARACTERS

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

IDENTIFICATION

First identification: A, B, E.

Second identification: A, C, D, E.

A. It complies with the test for specific optical rotation (see Tests).

B. Examine by infrared absorption spectrophotometry (*2.2.24*), comparing with the spectrum obtained with *lysine hydrochloride CRS*. Examine the substances prepared as discs. If the spectra obtained show differences, dissolve the substance to be examined and the reference substance separately in the minimum volume of *water R*, evaporate to dryness at 60 °C, and record new spectra using the residues.

C. Examine the chromatograms obtained in the test for ninhydrin-positive 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. To 0.1 ml of solution S (see Tests) add 2 ml of *water R* and 1 ml of a 50 g/l solution of *phosphomolybdic acid R*. A yellowish-white precipitate is formed.

E. To 0.1 ml of solution S add 2 ml of *water R*. The solution gives reaction (a) of chlorides (*2.3.1*).

TESTS

Solution S. Dissolve 5.0 g in *carbon dioxide-free water R* prepared from *distilled 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 B_7 or GY_7 (*2.2.2, Method II*).

Specific optical rotation (*2.2.7*). Dissolve 2.00 g in *hydrochloric acid R1* and dilute to 25.0 ml with the same acid. The specific optical rotation is + 21.0 to + 22.5, calculated with reference to the dried substance.

Ninhydrin-positive substances. Examine by thin-layer chromatography (*2.2.27*), using a *TLC silica gel plate R*.

Test solution (a). Dissolve 0.10 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 50 ml with *water R*.

Reference solution (a). Dissolve 10 mg of *lysine hydrochloride CRS* in *water R* and dilute to 50 ml with the same solvent.

Reference solution (b). Dilute 5 ml of test solution (b) to 20 ml with *water R*.

Reference solution (c). Dissolve 10 mg of *lysine hydrochloride CRS* and 10 mg of *arginine CRS* in *water R* and dilute to 25 ml with the same solvent.

Apply separately to the plate 5 µl of each solution. Develop over a path of 15 cm using a mixture of 30 volumes of *concentrated ammonia R* and 70 volumes of *2-propanol R*. Dry the plate at 100 °C to 105 °C until the ammonia disappears completely. Spray with *ninhydrin solution R* and heat at 100 °C to 105 °C for 15 min. 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). The test is not valid unless the chromatogram obtained with reference solution (c) shows two clearly separated principal spots.

Sulphates (*2.4.13*). Dilute 5 ml of solution S to 15 ml with *distilled water R*. The solution complies with the limit test for sulphates (300 ppm).

Ammonium (*2.4.1*). 50 mg complies with limit test B for ammonium (200 ppm). Prepare the standard using 0.1 ml of *ammonium standard solution (100 ppm NH_4) R*.

Iron (*2.4.9*). In a separating funnel, dissolve 0.33 g in 10 ml of *dilute hydrochloric acid R*. Shake with three quantities, each of 10 ml, of *methyl isobutyl ketone R1*, shaking for 3 min each time. To the combined organic layers add 10 ml of *water R* and shake for 3 min. The aqueous layer complies with the limit test for iron (30 ppm).

Heavy metals (*2.4.8*). 12 ml of solution S complies with limit test A for heavy metals (10 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

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

1 ml of *0.1 M perchloric acid* is equivalent to 18.27 mg of $C_6H_{15}ClN_2O_2$.

STORAGE

Store protected from light.