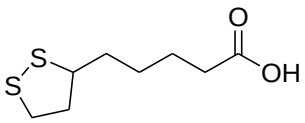
Alpha Lipoic Acid



$C_8H_{14}O_2S_2$ Mol. Wt. 206.3

Alpha Lipoic Acid is 1,2-Dithiolane-3-pentanoic acid; 1,2-Dithiolane-3-valeric acid.

Alpha Lipoic Acid contains not less than 99.0 per cent and not more than 101.0 per cent of $C_8H_{14}O_2S_2$ calculated on the dried basis.

Category. Nutraceutical.

Description. A light yellow to yellow powder.

Identification

A. Determine by infrared absorption spectrophotometry (2.4.6). Compare the spectrum with that obtained with *alpha lipoic acid RS* or with the reference spectrum of alpha lipoic acid.

B. In the Assay, the principal peak in the chromatogram obtained with the test solution corresponds to the peak in the chromatogram obtained with reference solution (a).

Tests

Melting range (2.4.21). 60° to 62°.

Specific optical rotaion (2.4.22). -1.0° to +1.0°, determined in 5.0 per cent w/v solution in *ethanol*.

Related substances. A. Determine by liquid chromatography (2.4.14).

Test solution. Dissolve 0.1 g of the substance under examination in the mobile phase and dilute to 100.0 ml with the mobile phase.

Reference solution (a). A 0.1 cent w/v solution of *alpha lipoic acid RS* in the mobile phase.

Reference solution (b). Dilute 1.0 ml of reference solution (a) to 100.0 ml with the mobile phase. Further dilute 1.0 ml of the solution to 10.0 ml with the mobile phase.

Chromatographic system

- a stainless steel column 25 cm x 4.6 mm, packed with octadecylsilane bonded to porous silica (5 µm),
- mobile phase: a mixture of 46 volumes of 0.068 per cent w/v solution of *potassium dihydrogen phosphate*, 58 volumes of *methanol* and 9 volumes of *acetonitrile*, adjusted to pH 3.0 with *orthophosphoric acid*,
- flow rate: 1.2 ml per minute,
- spectrophotometer set at 215 nm,
- injection volume: 20 µl.

Inject reference solution (b). The test is not valid unless the signal-to-noise ratio is not less than 10 and the relative standard deviation for replicate injections is not more than 10.0 per cent.

Inject the test solution. The area of any secondary peak is not more than 1.0 per cent and the sum of areas of all the secondary peaks is not more than 2.0 per cent, calculated by area normalization.

B. Determine by thin-layer chromatography (2.4.17), coating the plate with *silica gel G*.

Note- Carry out the test using low actinic glassware.

Mobile phase. A mixture of 40 volumes of *1-propanol,* 40 volumes of *ethyl acetate,* 10 volumes of *water* and 5 volumes of *ammonia*.

Test solution. Dissolve 0.4 g of the substance under examination in 10.0 ml of *dimethylformamide*.

Reference solution. A 4.0 per cent w/v solution of *alpha lipoic acid RS* in *dimethylformamide.*

Apply to the plate 5µl of each solution. Allow the mobile phase to rise 10 cm. Dry the plate in air, until the ammonia disappears completely, heat at 50° for 20 minutes allow to cool and place in iodine vapour saturated chamber until spots are visible. The R_f values for the spot alpha lipoic acid is, 0.25 - 0.30 and for polymeric lipoic acid is 0. Any secondary spot in the chromatogram obtained with the test solution is not more intense than the spot at R_f 0 in the chromatogram obtained with the reference solution.

Loss on drying (2.4.19). Not more than 0.2 per cent, by drying under vacuum at 40° for 3 hours.

Sulphated ash (2.3.18). Not more than 0.1 per cent.

Assay. Determine by liquid chromatography (2.4.14), as described under Related substances,

Inject reference solution (a). The test is not valid unless the column efficiency is not less than 10000 theoretical plates, the tailing factor is not more than 2.0 and the relative standard deviation for replicate injections is not more than 2.0 per cent.

Inject reference solution (a) and the test solution.

Calculate the content of $C_8H_{14}O_2S_2$.

Storage. Store protected from moisture, at a temperature not exceeding 30°.

Solubility (2.4.26): Soluble in *ethanol*, very slightly soluble in *water*.