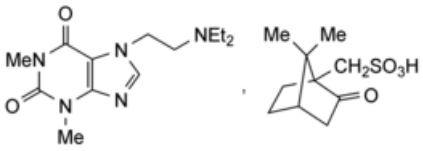
Edition: BP 2025 (Ph. Eur. 11.6 update)

Etamiphylline Camsilate

General Notices



$C_{13}H_{21}N_5O_2,C_{10}H_{16}O_4S$ 511.6 *19326-29-5*

Action and use

Non-selective phosphodiesterase inhibitor (xanthine); treatment of reversible airways obstruction.

Preparations

Etamiphylline Injection

Etamiphylline Tablets

DEFINITION

Etamiphylline Camsilate is 7-(2-diethylaminoethyl)theophylline camphorsulfonate. It contains not less than 98.0% and not more than 102.0% of $C_{13}H_{21}N_5O_2,C_{10}H_{16}O_4S$, calculated with reference to the dried substance, when determined by both methods described under the Assay.

CHARACTERISTICS

A white or almost white powder.

Very soluble in *water*; soluble in *chloroform* and in *ethanol (96%)*; very slightly soluble in *ether*.

IDENTIFICATION

A. The *light absorption*, Appendix II B, in the range 230 to 350 nm of a 0.004% w/v solution exhibits a maximum only at 274 nm. The *absorbance* at 274 nm is about 0.70.
B. The *infrared absorption spectrum*, Appendix II A, is concordant with the *reference spectrum* of etamiphylline camsilate *(RSV 51)*.
C. Fuse 0.1 g with a pellet of *sodium hydroxide*, dissolve in *water* and neutralise with *hydrochloric acid*. The resulting solution yields reaction A characteristic of *sulfates*, Appendix VI.

TESTS

Melting point

202° to 206°, Appendix V A.

Acidity

pH of a 10% w/v solution, 3.9 to 5.4, Appendix V L.

Free etamiphylline

Not more than 2.0% w/w when determined by Method I for *non-aqueous titration*, Appendix VIII A, using 2 g dissolved in 75 mL of *acetic anhydride* and determining the end point potentiometrically. Each mL of *0.1M perchloric acid VS* is equivalent to 27.93 mg of free etamiphylline.

Related substances

Carry out the method for *thin-layer chromatography*, Appendix III A, using *silica gel* HF_{254} as the coating substance and a mixture of 80 volumes of *chloroform*, 20 volumes of *ethanol (96%)* and 1 volume of 13.5M *ammonia* as the mobile phase. Apply separately to the plate 10 µL of each of two solutions of the substance being examined in *water* containing (1) 4.0% w/v and (2) 0.0080% w/v. After removal of the plate, allow it to dry in air and examine under *ultraviolet light (254 nm)*. Any *secondary spot* in the chromatogram obtained with solution (1) is not more intense than the spot in the chromatogram obtained with solution (2) (0.2%).

Loss on drying

When dried to constant weight at 105°, loses not more than 0.5% of its weight. Use 1 g.

Sulfated ash

Not more than 0.2%, Appendix IX A.

ASSAY

For camphorsulfonic acid

Dissolve 1 g in 25 mL of *methanol*, previously neutralised with *1M sodium hydroxide VS*, and titrate with *0.1M sodium hydroxide VS* using *thymol blue solution* as indicator. Each mL of *0.1M sodium hydroxide VS* is equivalent to 51.16 mg of $C_{13}H_{21}N_5O_2$,$C_{10}H_{16}O_4S$.

For etamiphylline

Dissolve 0.15 g in 20 mL of *2M hydrochloric acid*, add 12 mL of a 5% w/v solution of *silicotungstic acid* and allow to stand for 5 hours. Filter, wash the residue with *2M hydrochloric acid* until the filtrate yields no precipitate with a 1% w/v solution of *quinine hydrochloride* and dry at 110°. Each g of residue is equivalent to 0.2830 g of $C_{13}H_{21}N_5O_2$,$C_{10}H_{16}O_4S$.