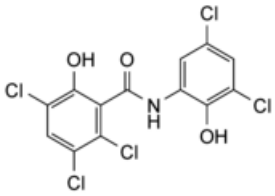
Edition: BP 2025 (Ph. Eur. 11.6 update)

Oxyclozanide

General Notices



$C_{13}H_6Cl_5NO_3$ 401.5 *2277-92-1*

Action and use

Antihelminthic.

Preparation

Oxyclozanide Oral Suspension

DEFINITION

Oxyclozanide is 3,3′,5,5′,6-pentachloro-2′-hydroxysalicylanilide. It contains not less than 98.0% and not more than 101.0% of $C_{13}H_6Cl_5NO_3$, calculated with reference to the dried substance.

CHARACTERISTICS

A pale cream or cream coloured powder.

Very slightly soluble in *water*; freely soluble in *acetone*; soluble in *ethanol (96%)*; slightly soluble in *chloroform*.

IDENTIFICATION

A. The *infrared absorption spectrum*, Appendix II A, is concordant with the *reference spectrum* of oxyclozanide *(RSV 33)*.
B. The *light absorption*, Appendix II B, in the range 250 to 350 nm of a 0.003% w/v solution in *acidified methanol* exhibits a maximum only at 300 nm. The *absorbance* at the maximum is about 0.76, Appendix II B.
C. *Melting point*, about 208°, Appendix V A.

TESTS

Ionisable chlorine

Dissolve 2 g in 100 mL of *methanol*, add 10 mL of 1.5M *nitric acid* and titrate with *0.1M silver nitrate VS* determining the end point potentiometrically. Not more than 1.4 mL is required (0.25%).

Related substances

Carry out the method for *liquid chromatography*, Appendix III D, using the following solutions.

(1) 0.1% w/v of the substance being examined prepared by dissolving it in a suitable volume of *methanol* and slowly diluting with *water* containing 0.1% v/v *orthophosphoric acid* to give a solution containing about the same ratio of methanol to water as the mobile phase.
(2) Dilute 1 volume of solution (1) to 100 volumes with the mobile phase.

CHROMATOGRAPHIC CONDITIONS

(a) Use a stainless steel column (20 cm × 4.6 mm) packed with *octadecylsilyl silica gel for chromatography* (5 µm) (Hypersil ODS is suitable).
(b) Use isocratic elution and the mobile phase described below.
(c) Use a flow rate of 2 mL per minute.
(d) Use an ambient column temperature.
(e) Use a detection wavelength of 300 nm.
(f) Inject 20 µL of each solution.

MOBILE PHASE

A mixture of *methanol* and *water* containing 0.1% v/v of *orthophosphoric acid* (a mixture of 38 volumes of water and 62 volumes of methanol is usually suitable).

LIMITS

In the chromatogram obtained with solution (1):

the area of any *secondary peak* with a retention time less than that of the principal peak is not greater than one third of the area of the principal peak in the chromatogram obtained with solution (2) (0.3%);

the area of any *secondary peak* with a retention time greater than that of the principal peak is not greater than the area of the principal peak in the chromatogram obtained with solution (2) (1%).

Loss on drying

When dried to constant weight at 60° at a pressure not exceeding 0.7 kPa, loses not more than 1.0% of its weight. Use 1 g.

Sulfated ash

Not more than 0.2%, Appendix IX A.

ASSAY

Dissolve 0.25 g in 75 mL of *anhydrous pyridine* and pass a current of *nitrogen* through the solution for 5 minutes. Carry out Method II for *non-aqueous titration*, Appendix VIII A, maintaining a current of *nitrogen* through the solution throughout the titration, using 0.1M *tetrabutylammonium hydroxide VS* as titrant and determining the end point potentiometrically. Each mL of 0.1M *tetrabutylammonium hydroxide VS* is equivalent to 20.07 mg of $C_{13}H_6Cl_5NO_3$.