Quality standards

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

Deltamethrin

General Notices

C₂₂H₁₉Br₂NO₃ 505.2 52918-63-5

Action and use

Insecticide (veterinary).

Preparation

Deltamethrin Pour-on

DEFINITION

Deltamethrin is (S)- α -cyano-3-phenoxybenzyl-(1R,3R)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxylate. It contains not less than 97.0% and not more than 101.0% of $C_{22}H_{10}Br_2NO_3$.

CHARACTERISTICS

A white to buff-coloured, crystalline powder.

Insoluble in water, soluble in ethanol (96%) and in acetone.

IDENTIFICATION

A. The infrared absorption spectrum, Appendix II A, is concordant with the reference spectrum of deltamethrin (RSV 47).

B. In the test for Related substances, the principal spot in the chromatogram obtained with solution (2) corresponds to that in the chromatogram obtained with solution (5).

TESTS

Specific optical rotation

In a 4% w/v solution in toluene, +55.5 to +61.5, Appendix V F.

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Becisthemic acid chloride

Not more than 0.2% when determined by the following method. Dissolve 2 g in 100 ml of <u>methanol</u> with moderate heating, if necessary, and cool. Titrate with <u>0.02M potassium hydroxide VS</u> using a solution containing 0.8% w/v of <u>dimethyl yellow</u> and 0.08% w/v of <u>methylene blue</u> in <u>methanol</u> as indicator to a green end point. Each ml of <u>0.02M potassium hydroxide VS</u> is equivalent to 6.329 mg of becisthemic acid chloride, C₈H_oBr₂CIO.

Becisthemic acid and becisthemic anhydride

Not more than 1% in total when determined by the following methods.

Becisthemic acid

Dissolve 2 g in 100 ml of <u>ethanol</u> (96%) with moderate heating. Cool in an ice-bath and immediately titrate with <u>0.02M</u> <u>sodium hydroxide VS</u> using a 1% w/v solution of <u>1-naphtholbenzein</u> in <u>ethanol</u> (96%) solution as indicator to a green end point. Correct the volume of titrant for any contribution due to the becisthemic acid chloride content using the following expression:

where V = titration volume obtained in the becisthemic acid chloride test,

P₁ = weight of sample used in the becisthemic chloride test,

 P_2 = weight of sample used in this test.

Each ml of 0.02m sodium hydroxide VS is equivalent to 5.959 mg of becisthemic acid, C₈H₁₀Br₂O₂.

Becisthemic anhydride

To 1.0 g add 10 ml of 0.01M <u>aniline</u> in <u>cyclohexane</u> and 10 ml of <u>glacial acetic acid</u>. Stopper the flask and allow to stand at room temperature for 1 hour. Titrate with 0.01M <u>perchloric acid VS</u> using <u>crystal violet solution</u> as indicator. Repeat the procedure omitting the substance being examined. Correct the volume of titrant for any contribution due to twice the becisthemic acid chloride content calculated using the above formula. Each ml of 0.01M <u>perchloric acid VS</u> is equivalent to 5.779 mg of becisthemic anhydride, $C_{16}H_{18}Br_4O_3$.

Related substances

Carry out the method for thin-layer chromatography, Appendix III A, using the following solutions in toluene.

- (1) 2.0% w/v of the substance being examined.
- (2) 0.5% w/v of the substance being examined.
- (3) 0.020% w/v of the substance being examined.
- (4) 0.010% w/v of the substance being examined.
- (5) 0.5% w/v of <u>deltamethrin BPCRS</u>.

CHROMATOGRAPHIC CONDITIONS

- (a) Use a silica gel F_{254} precoated plate (Merck silica gel 60 F_{254} plates are suitable).
- (b) Use the mobile phase as described below.
- (c) Apply 10 µl of each solution.
- (d) Develop the plate to 15 cm.
- (e) After removal of the plate, dry in air and examine under <u>ultraviolet light (254 nm)</u>.

MOBILE PHASE

20 volumes of di-isopropyl ether and 80 volumes of hexane.

LIMITS

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Any <u>secondary spot</u> in the chromatogram obtained with solution (1) is not more intense than the spot in the chromatogram obtained with solution (3) (1%) and not more than two such spots are more intense than the spot in the chromatogram obtained with solution (4) (0.5%).

ASSAY

Carry out the method for liquid chromatography, Appendix III D, using the following solutions in the mobile phase.

- (1) 0.1% w/v of the substance being examined.
- (2) 0.1% w/v of deltamethrin BPCRS.
- (3) 0.1% w/v of deltamethrin impurity standard BPCRS.

CHROMATOGRAPHIC CONDITIONS

- (a) Use a stainless steel column (25 cm \times 4.6 mm) packed with <u>silica gel for chromatography</u> (5 μ m) (Zorbax Sil is suitable).
- (b) Use isocratic elution and the mobile phase described below.
- (c) Use a flow rate of 1.3 ml per minute.
- (d) Use an ambient column temperature.
- (e) Use a detection wavelength of 278 nm.
- (f) Inject 20 µl of each solution.

MOBILE PHASE

0.04 volume of propan-2-ol, 2 volumes of acetonitrile, 10 volumes of dichloromethane and 100 volumes of hexane.

SYSTEM SUITABILITY

The test is not valid unless, in the chromatogram obtained with solution (3), a peak due to (*R*)-deltamethrin appears immediately before the principal peak, as indicated in the reference chromatogram supplied with <u>deltamethrin impurity</u> <u>standard BPCRS</u>.

DETERMINATION OF CONTENT

Calculate the content of C₂₂H₁₉Br₂NO₃ using the declared content of C₂₂H₁₉Br₂NO₃ in <u>deltamethrin BPCRS</u>.

IMPURITIES

The impurities limited by the requirements of this monograph include:

- Becisthemic acid,
- Becisthemic anhydride,
- Becisthemic acid chloride.