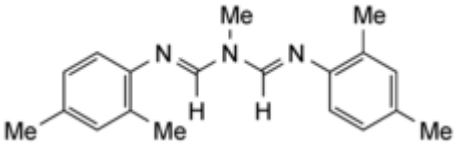
British Pharmacopoeia | Quality standards

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

Amitraz

General Notices



$C_{19}H_{23}N_3$ 293.4 *33089-61-1*

Action and use

Topical parasiticide; acaricide.

Preparation

Amitraz Dip Concentrate (Liquid)

DEFINITION

Amitraz is *N*-methylbis (2,4-xylyliminomethyl) amine. It contains not less than 97.0% and not more than 101.0% of $C_{19}H_{23}N_3$, calculated with reference to the anhydrous substance.

CHARACTERISTICS

A white to buff powder.

Practically insoluble in *water*; decomposes slowly in *ethanol (96%)*; freely soluble in *acetone*.

IDENTIFICATION

The *infrared absorption spectrum*, Appendix II A, is concordant with the *reference spectrum* of amitraz *(RSV 04)*.

TESTS

Related substances

Carry out the method for *gas chromatography*, Appendix III B, using the following solutions.

0.010% w/v of *2,4-dimethylaniline*, 0.20% w/v of *form-2',4'-xylidide BPCRS* and 0.20% w/v of *N,N'-bis(2,4-xylyl)formamidine BPCRS* in *methyl acetate* (solution A)

Disperse 30 mg of *N-methyl-N'-(2,4-xylyl)formamidine hydrochloride BPCRS* in 5 mL of *methyl acetate*, add about 32 mg of *triethylamine*, mix with the aid of ultrasound for 2 minutes, filter, wash the filter with a small amount of *methyl acetate* and add sufficient *methyl acetate* to the combined filtrate and washings to produce 25 mL (solution B) (about 0.1% w/v of *N-methyl-N'-(2,4-xylyl)formamidine)*.

(1) 5.0% w/v solution of the substance being examined in *methyl acetate*.
(2) A mixture of equal volumes of solution A and solution B.

CHROMATOGRAPHIC CONDITIONS

(a) Use a *fused silica capillary column* (10 m × 0.53 mm) bonded with a film (5 µm) of *poly [methyl(95)phenyl(5)]siloxane* (Chrompack CP-SIL 8 CB is suitable).
(b) Use *helium* as the carrier gas at 12 mL per minute.
(c) Use gradient conditions at an initial temperature of 125°, maintained at 125° for 5 minutes, increasing linearly to 270° at a rate of 5° per minute and maintained at 270° for 15 minutes.
(d) Use an inlet temperature of 230°.
(e) Use a flame ionisation detector at a temperature of 300°.
(f) Inject 1 µL of each of solutions (1) and (2).

In the chromatogram obtained with solution (2) the peaks following the solvent peak, in order of emergence, are due to 2,4-dimethylaniline, form-2′,4′-xylidide, N-methyl-N′-(2,4-xylyl)formamidine and N,N′-bis(2,4-xylyl)formamidine.

LIMITS

In the chromatogram obtained with solution (1):

the area of any peak corresponding to 2,4-dimethylaniline, form-2′,4′-xylidide, *N*-methyl-*N*′-(2,4-xylyl)formamidine and *N,N*′-bis(2,4-xylyl)formamidine is not greater than the area of the corresponding peak in the chromatogram obtained with solution (2) (0.1%, 2%, 1% and 2% respectively);

the area of any other secondary peak is not greater than the area of the peak due to 2,4-dimethylaniline in the chromatogram obtained with solution (2) (0.1%).

Water

Not more than 0.1% w/w, Appendix IX C, Method IA. Use 5 g and a mixture of equal volumes of *chloroform* and *2-chloroethanol* in place of *anhydrous methanol*.

Sulfated ash

Not more than 0.2%, Appendix IX A.

ASSAY

Carry out the method for *gas chromatography*, Appendix III B, using the following solutions.

Prepare a 2% v/v solution of *squalane* (internal standard) in *methyl acetate* (solution C).

(1) 0.15 g of the substance being examined in sufficient *methyl acetate* to produce 30 mL.
(2) 0.15 g of the substance being examined in 10 mL of solution C and add sufficient *methyl acetate* to produce 30 mL.
(3) 1.50% w/v solution of *amitraz BPCRS* in solution C and dilute 1 volume of this solution to 3 volumes with *methyl acetate*.

CHROMATOGRAPHIC CONDITIONS

(a) Use a fused silica capillary column (15 m × 0.53 mm) coated with a 1.5 µm film of methyl silicone gum (Chrompack CP-Sil 5 CB is suitable).
(b) Use *helium* as the carrier gas at 12 mL per minute.
(c) Use isothermal conditions maintained at 220°.
(d) Use an inlet temperature of 230°.
(e) Use a flame ionisation detector at a temperature of 300°.
(f) Inject 1 µL of each solution.

SYSTEM SUITABILITY

The assay is not valid unless, in the chromatogram obtained with solution (3), the *resolution factor* between the peaks corresponding to squalane and amitraz is at least 3.0.

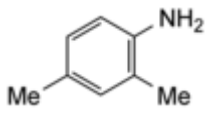
LIMITS

Calculate the content of $C_{19}H_{23}N_3$ from the chromatograms obtained using the declared content of $C_{19}H_{23}N_3$ in *amitraz BPCRS*.

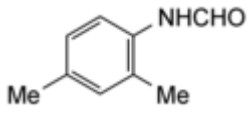
STORAGE

Amitraz should be kept in a well-closed container, which may contain paraformaldehyde, packed in separate sachets as a stabiliser.

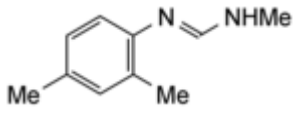
IMPURITIES



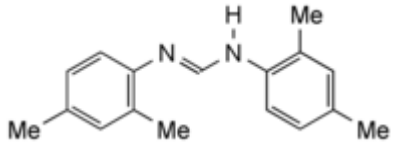
A. 2,4-dimethylaniline (2,4-xylidine),



B. form-2′,4′-xylidide,



C. *N*-methyl-*N*′-(2,4-xylyl)formamidine,



D. *N*,*N*′-bis(2,4-xylyl)formamidine.