PYRIPROXYFEN

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Method Extension of CIPAC 715/TC/M/-

Determination of Pyriproxyfen
in Permethrin/Pyriproxyfen LN

by
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1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. Use the HPLC method below. The relative retention time of pyriproxyfen with respect to the internal standard for the sample solution should not deviate by more than 2% from that for the calibration solution (Fig. 1).

2.2 UV spectrum. Use the HPLC method below and record the spectrum of the pyriproxyfen peak with a diode array detector. The spectrum obtained from the sample solution should not differ significantly from that of the calibration solution (Fig. 2).

3 Pyriproxyfen

OUTLINE OF METHOD Pyriproxyfen is determined by reversed phase high performance liquid chromatography using UV detection at 254 nm and dicyclohexyl phthalate as internal standard.

REAGENTS

Heptane

1-Propanol

Acetonitrile HPLC grade

Water HPLC grade

Pyriproxyfen standard of known purity. Store refrigerated.

Dicyclohexyl phthalate internal standard. Must not show any peaks with the same retention time as pyriproxyfen and permethrin.

Internal standard solution. Dissolve dicyclohexyl phthalate (5.0 g) in 1-propanol (200 ml). Ensure that a sufficient quantity of this solution is prepared for all samples and calibration standards to be analysed.

Calibration solution. Homogenise the pyriproxyfen standard. When it is a waxy or partly waxy solid homogenise it by warming it to melting and by stirring. Weigh in duplicate (to the nearest 0.1 mg) 90 to 110 mg (s mg) of pyriproxyfen standard into a vial or a stoppered flask (200 ml). Add by pipette to each vial or flask internal standard solution (10.0 ml) and by measuring cylinder acetonitrile (90 ml). Mix well (solutions C_A and C_B).

APPARATUS

High performance liquid chromatograph equipped with a detector suitable for operation at 254 nm, a constant temperature column compartment and an injector capable of delivering 10 µl

Column 250 mm x 4.6 mm (i.d.), stainless steel, packed with Nucleosil C_{18} (5 µm), or equivalent
Electric integrator or data system
Water bath
Rotary evaporator

PROCEDURE

(a) Liquid chromatographic conditions (typical):

Column	stainless steel, 250 x 4.6 mm (i.d.), packed with Nucleosil C_{18} (5 µm), or equivalent.
Mobile phase	acetonitrile – water, 700 + 350 (v/v)
Column temperature	40°C
Flow rate	1.0 ml/min
Detector wavelength	254 nm
Injection volume	10 µl
Retention times	pyriproxyfen: about 17 min dicyclohexyl phthalate: about 25 min

(b) Linearity check. Before conducting the analysis check the linearity of the detector response by injecting 10 µl portions of solutions with pyriproxyfen concentrations 0.5, 1 and 2 times that of the calibration solution.

(c) System equilibration. Inject 10 µl portions of calibration solution C_A until the response factors obtained for two consecutive injections differ by less than 1.0%. Then inject a 10 µl portion of calibration solution C_B. The response factor for this solution should not deviate by more than 1.0% from that for the first calibration solution, otherwise prepare new calibration solutions.

(d) Preparation of sample solution. Clean a pair of scissors with acetone before use. Cut the sample with the scissors into 5 – 10 mm squares. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) sufficient sample to contain 18 to 22 mg (*w* mg) of pyriproxyfen into a vial or stoppered flask (100 ml). Add by pipette internal standard solution (2.0 ml) and by measuring cylinder heptane (48 ml). Place the vial or stoppered flask in a water bath (85 – 90°C) for 45 min. Shake the vial or stoppered flask once or twice during the extraction. After extraction, cool it to room temperature. Transfer by pipette the solution (10.0 ml) into a round-bottom flask (50 ml). Evaporate the solution *in vacuo*, add by pipette acetonitrile (4.0 ml) and dissolve completely (solutions S_A and S_B).

(e) Determination. Inject in duplicate 10 µl portions of each sample solution bracketing them by injections of the calibration solutions as follows; calibration solution C_A, sample solution S_A, sample solution S_A, calibration solution C_B, sample solution S_B, sample solution S_B, calibration solution C_A, and so on. Measure the relevant peak areas. Average the values of the duplicate sample injections. Calculate the mean value of the response factors of the calibration solution bracketing two sample solutions and use this value to calculate the pyriproxyfen concentration of the bracketed samples.

$$f_i = \frac{I_r \times s \times P}{H_s \times 5}$$

$$\text{Pyriproxyfen content} = \frac{f \times H_w}{I_q \times w} \text{ g/kg}$$

where:

f_i = individual response factor
f = mean response factor
H_s = peak area of pyriproxyfen in the calibration solution
H_w = peak area of pyriproxyfen in the sample solution
I_r = peak area of the internal standard in the calibration solution
I_q = peak area of the internal standard in the sample solution
s = mass of pyriproxyfen standard in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of pyriproxyfen standard (g/kg)

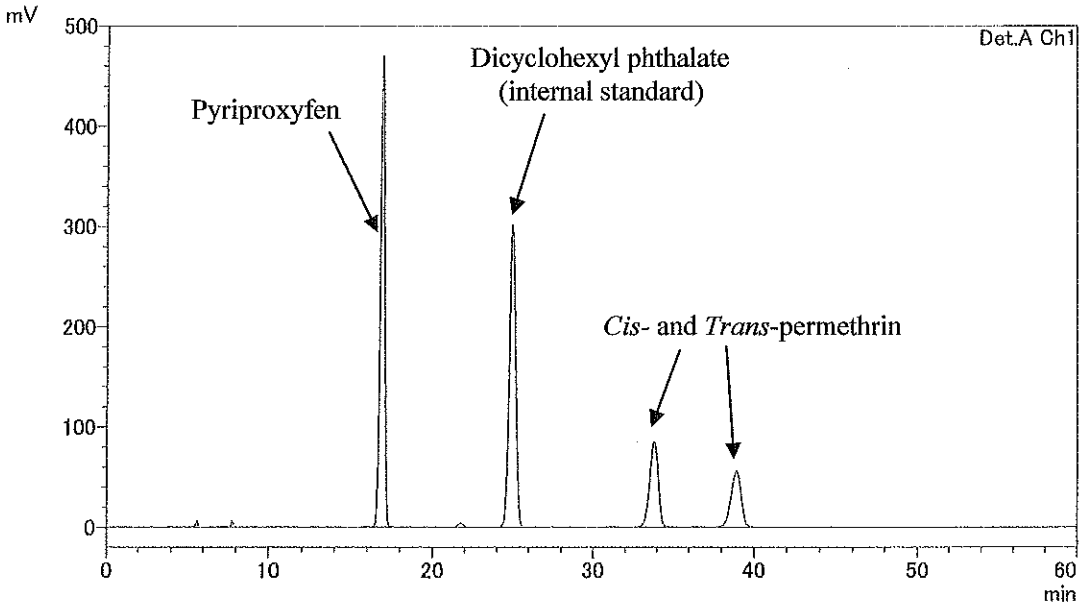

Fig. 1 Chromatogram of permethrin/pyriproxyfen LN

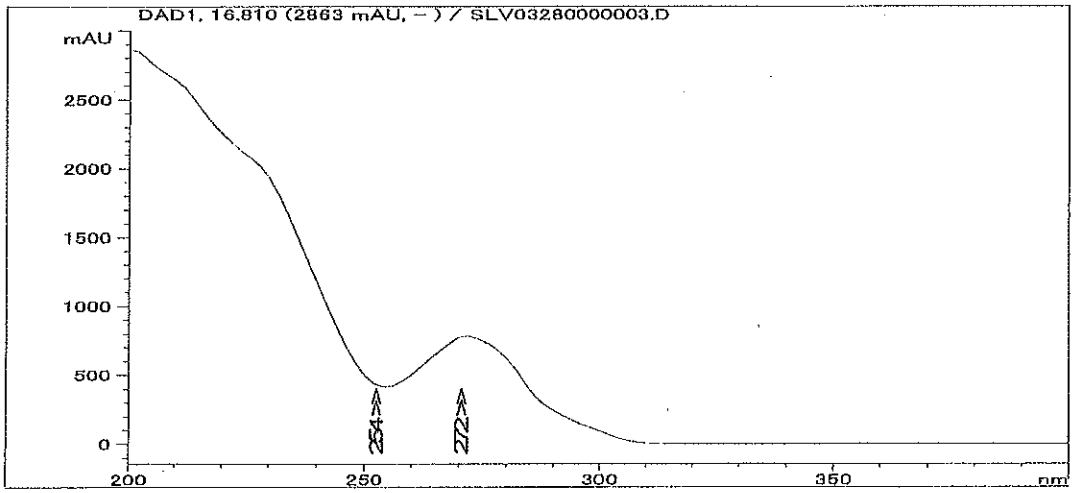

Fig. 2 UV spectrum of pyriproxyfen