

CIPAC

COLLABORATIVE INTERNATIONAL PESTICIDES ANALYTICAL COUNCIL LIMITED

Commission Internationale des Méthodes d'Analyse des Pesticides (CIMAP)

Long lasting insecticidal nets (LN) are becoming more and more important in the control and prevention of diseases like malaria. In order to meet an urgent need for methods to characterise LN, CIPAC provides selected methods as download. By downloading these methods you accept the following conditions of use:

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DETERMINATION OF deltamethrin in PermaNet ® BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

1. Sampling.

A sample is defined as one finished bed net taken randomly from a batch of bed net. Sub sample by cutting 18 pieces of size 100cm² from a net either randomly from the whole net or at recommended positions as in appendix 1. Sampling as recommended in appendix 1 leaves sufficient material for other physical properties tests including Dimensional Stability, Bursting Strength, Weight...etc. Cut 18 pieces into half, one portion is used for Deltamethrin content analysis and the other is used for washing test if required.

The portion used for Deltamethrin content analysis is cut into piece of less than 2cmx2cm each. Mix well.

Sample shall be separately packed in aluminium foil and kept out of direct sunlight at room temperature or lower.

2. Identity tests.

Use the HPLC method below. The retention time of Deltamethrin in the sample solution should not deviate by more than 15 s from that of the calibration solution if column oven is available.

3. Active ingredient.

OUTLINE OF METHOD

The sample is extracted in a mixture of iso-octane and 1,4- dioxane.

The Deltamethrin content is determined by normal phase high performance liquid chromatography using dipropyl phthalate as internal standard and detection at 236 nm.

SCOPE

This method is used for Deltamethrin determination in net sample, before and after washing.

REAGENTS

- Iso Octane, HPLC grade <Merck, Riedel De Haen, Prolabo>
 - 1,4 Dioxan, HPLC grade. Add 0.15% (v/v) water before use <Merck, Riedel De Haen, Prolabo>
 - Deltamethrin, neat standard, Dr. Ehrenstorfer Laboratories
 - Dipropyl Phthalate, Pestanal, Sigma Aldrich
 - Water, HPLC grade or higher <Merck, Riedel De Haen, Prolabo>
- (a) Extraction solvent (ES): iso octan + 1,4 dioxane = 80+20
(b) Mobile Phase (MP): iso octan + 1,4 dioxane = 95+5
(c) Internal Standard solution (IS): 0.5 mg/ml of dipropyl phthalate in extraction solvent.

(d) Deltamethrin standard solution 0.6mg/ml (DS).

Weight 0.03 g (to the nearest of 0.01 mg) of Deltamethrin neat standard, quantitatively transfer to 50 volumetric flask, dissolve completely with extraction solvent, keep regulated by water bath at 20oC, fill up to mark with extraction solvent.

APPARATUS

- Shaker
- Ultrasonic bath.
- HPLC, equipped with pump, auto-injector, column oven (optional) and UV detector. Guard Column (Supelguard Si). Analytical Column Supelco Si 5um, 150x4.6 or Lichrosorb Si60, 5um, 150x4.6.

PROCEDURE

(a) Operating conditions

- Mobile Phase: iso octan + 1,4 dioxane = 95+5
- Flow rate 1.3ml/min, isocratic
- Guard Column Supelguard Si
- Analytical Column Lichrosorb Si60, 5um, 150x4.6
- Column temperature 40oC if column oven is available, or room temperature
- Inject Volume 5ul
- Wavelength 236nm.
- Run time 6-8minute.

(b) Preparation of calibration curve

Into a series of clean 20ml PTFE liner screw cap vials, add accordingly as table below. Filter through 0.45um syringe filter before use.

Code	IS	DS	ES	Delta(mg)	Total Vol
C1	1ml	0.5 ml	13.5 ml	0.30	15ml
C2	1ml	0.7 ml	13.2 ml	0.42	15ml
C3	1ml	0.9 ml	12.8 ml	0.54	15ml
C4	1ml	1.1 ml	12.4 ml	0.66	15ml
C5	1ml	1.3 ml	12.1 ml	0.78	15ml

- All standards should be kept in the refrigerator if not in use in WELL-TIGHTEN, paraffin film sealed cap.

(c) Calibration

Daily calibrate HPLC system with full series of 05 standards, beginning with the lowest level standard. The correlation coefficient should not be less than 0.99 over this range.

(d) Preparation of sample

Weigh (to the nearest 0.1 mg) into an Erlenmeyer or a screw cap neutral glass bottle (50ml) sufficient sample to contain about 0.5 mg of Deltamethrin. For 75D, 100D and 150D netting sample, suitable weight (w g) is 0.3g, 0.4 and 0.6 g respectively. Add by pipette 1.0 ml internal standard solution. Add 14 ml extract solvent. Replace the cap closely.

Put the bottles into the ultrasonic bath, setting temperature 80oC, running time 15min. Vigorously shake the bottle using the shaker in 30 min at room temperature, speed of shaking is at level of 150-200 beats per minute.

Using on syringe membrane filter with pore size of 0.45um or finer, filter c.a. 1ml of extract solution into clean amber. Sample shall be injected within 24 hours since extraction, for longer waiting time the vial should be kept in a refrigerator.

(g) Determination

Inject sample with HPLC configured as in (a).

$$\text{Deltamethrin content (g/kg)} = a/w$$

a = Deltamethrin reading from the analysis in mg

w = mass of sample taken (g)

Note: Conventional unit of active ingredient content (deltamethrin) for netting is mg/m². The conversion from g/kg to mg/m² is made as follow:

$$\text{Deltamethrin content (mg/m}^2\text{)} = \text{Deltamethrin content (g/kg)} \times D$$

D= weight per square metre.

D of typical polyester netting:

75 Denier netting, D = 30g/m².

100 Denier netting, D = 40g/m².

150 Denier netting, D = 60g/m².

METHOD VALIDATION SUMMARY

Precision

Analyze 05 replicates RSD is 1.8%.

Accuracy

Recovery of 04 Laboratory Synthetic samples is in the range of 97% to 101%, averaged at 99.3%

Linearity

Over the range of Deltamethrin from 0.30 to 0.78 mg, the correlation coefficient R² is observed not less than 0.995.

Figure 1: Chromatogram Of Calibration Solution

TYPICAL STANDARD CHROMATOGRAPH

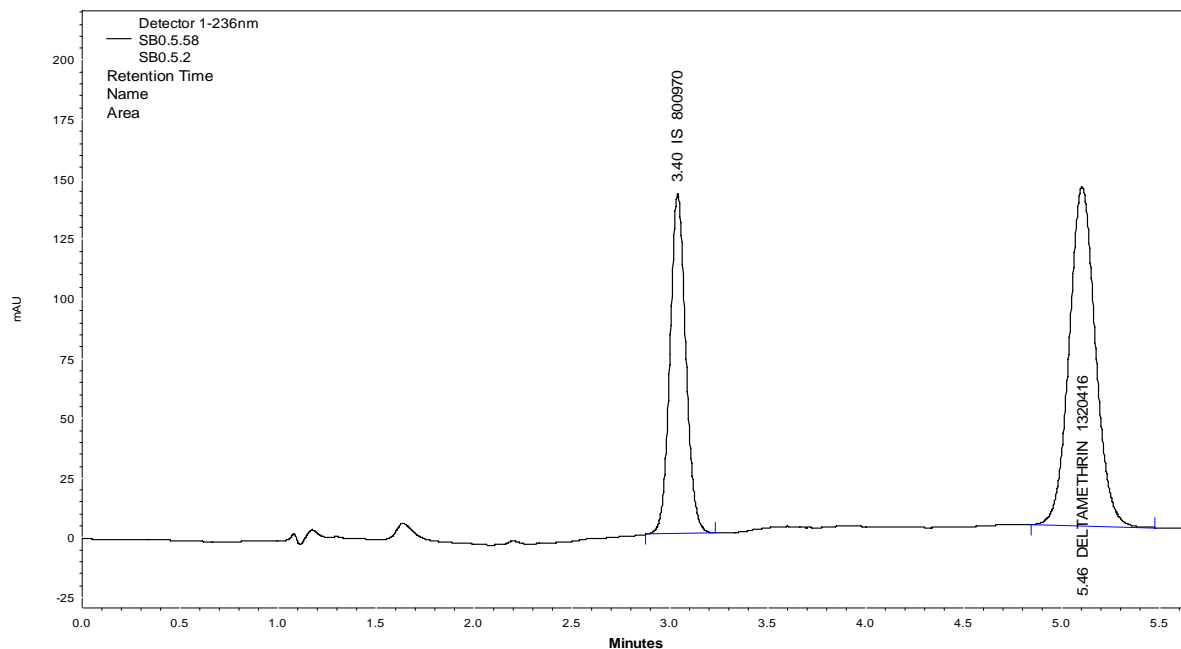
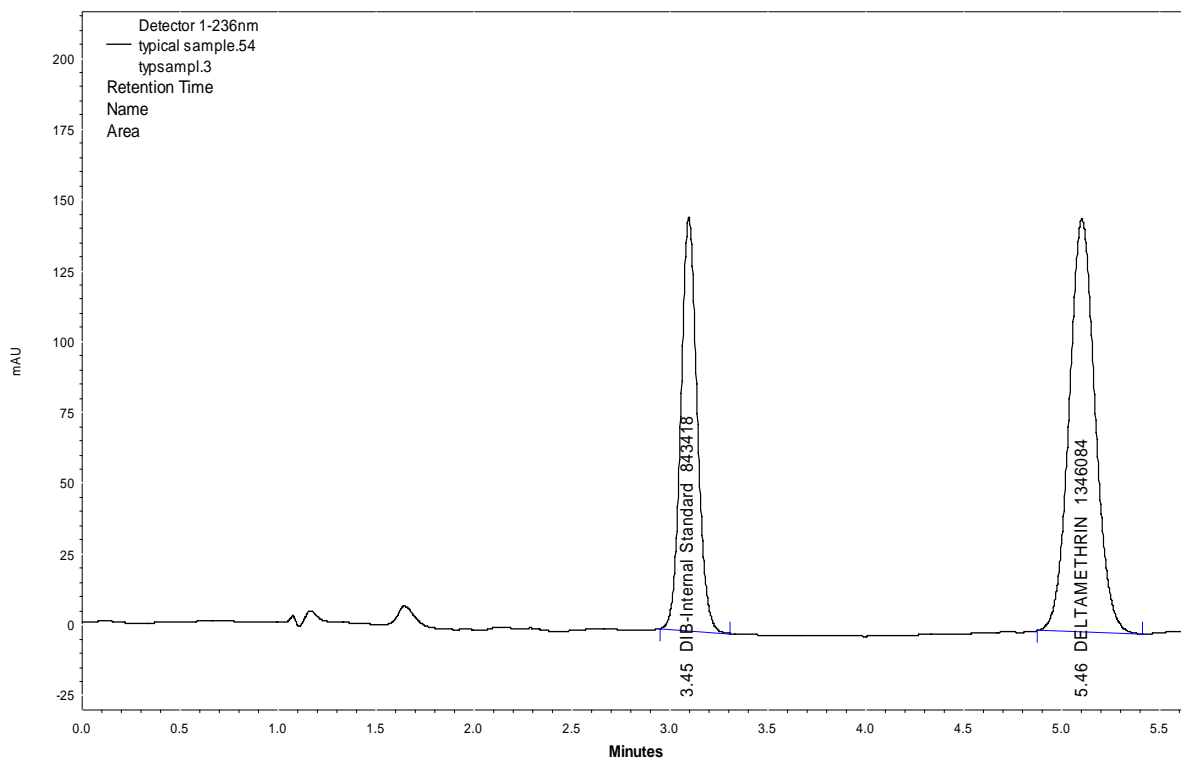
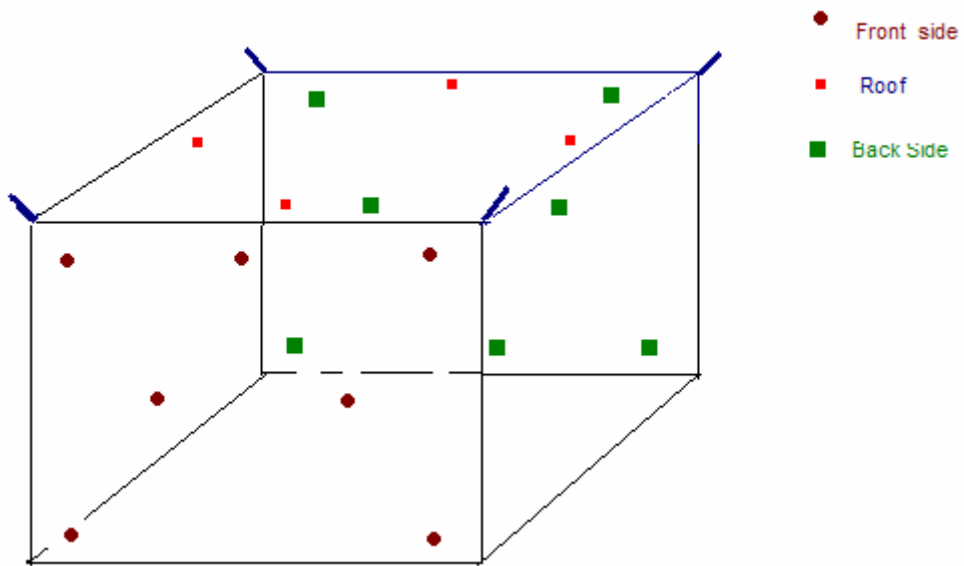


Figure 2: Chromatogram Of Sample

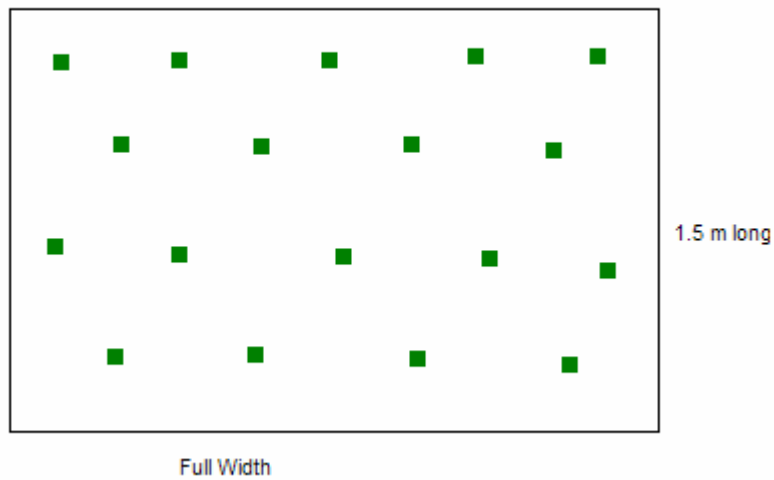
TYPICAL SAMPLE CHROMATOGRAPH



Appendix 1.
Rectangular net



From a roll or a piece of netting material



The position is at least far from any adjacent border 10 cm. Each piece is either roundel or square of 100cm².