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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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ALPHA-CYPERMETHRIN

454

CIPAC 4508/R, method extension for LN

by

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ALPHA-CYPERMETHRIN LONG LASTING INSECTICIDAL NETS
454/LN/(M)/-

1 Sampling. Take net samples as described in this paper

2 Identity test.

2.1 GLC. As for *alpha-cypermethrin technical* 454/TC/(M)2.1

3 Alpha-cypermethrin. As for *alpha-cypermethrin technical* 454/TC/(M)3, except: *Calibration solution* (REAGENTS), *Preparation of sample* and *Determination* (PROCEDURE)

OUTLINE OF METHOD. As for *alpha-cypermethrin* 454/TC/(M)3

REAGENTS. As for *alpha-cypermethrin* 454/TC/(M)3, except,

Calibration solutions.
Weigh 50 mg *alpha-cypermethrin* (to the nearest 0.1 mg) in a volumetric flask (25 ml). Fill to the mark with THF. Place the flask in an ultrasonic bath for 10 min. After temperature equilibration pipette 1.50 ml, 3.00 ml and 4.50 ml of this solution into three volumetric flasks (50 ml). Add 0.5 ml of internal standard solution (dioctyl phthalate, 1% in acetone) and fill up each to the mark with THF.
These solutions are used as calibration solutions A (C_A), B (C_B) and C (C_C).
Transfer 200 µl out of each flask into separate GC vials. Add one drop of citric acid in each case and seal the vials. Place the vials into the sample tray (cooled down to 15 °C) of the GC apparatus. Note: Citric acid is added to stop epimerisation of *alpha-cypermethrin* in solution.

Description of the calibration solutions:
C_A : concentration of approximately 3.0 mg *alpha-cypermethrin* in 50 ml THF.
C_B : concentration of approximately 6.0 mg *alpha-cypermethrin* in 50 ml THF.
C_C : concentration of approximately 9.0 mg *alpha-cypermethrin* in 50 ml THF.

APPARATUS. As for *alpha-cypermethrin* 454/TC/(M)3

Gas chromatograph with flame ionization detection (e.g., HP 6890 Plus)
Automatic sample injector (e.g., HP series 7683) equipped with a sample tray which is cooled down to 15 °C.
Capillary column fused silica (e.g., DB–1) 30 m x 0.32 mm, film thickness of 0.25 µm.
Electronic data evaluation system (e.g., HP ChemStation)
Ultrasonic bath
Usual laboratory equipment, e.g., electronic balance and standard laboratory glassware.

PROCEDURE.

(a) Operating conditions (typical). As for *alpha-cypermethrin* 454/TC/(M)3

(b) Preparation of the samples.

(i) Determination via the water / detergent extraction process

First step: Quantification of the adherent contents of *alpha-cypermethrin* on the surface of the fibers.

(1) Procedure done by manufacturer.
Place 2 g of impregnated net in a beaker (1 l). Add soap (commercially available bar soap or as flakes, 2 g/l) and water (500 ml). The washing is done for 10 min. at 30 °C on a shaker with 155 movements/min. Directly after the washing, the wash liquor is acidified with 10 ml/l acetic acid 30% in order to prevent hydrolysis of the extracted *alpha-cypermethrin* (note: there is no difference in hydrolysis in neutral water compared to the wash liquor after 10 min. at 30 °C). Transfer 150 ml of the washing liquid into a separation funnel (500 ml). The extraction procedure is done twice with 100 ml ethyl acetate each by shaking twice for 30 seconds. Both portions of ethyl acetate are combined together in a flask (250 ml). Ethyl acetate is evaporated in a rotary evaporator. The flask is stoppered and sent to the analytical laboratory.

(2) Procedure done by analytical laboratory.
Fill up the flask with THF (50 ml). Add 0.5 ml of internal standard solution (dioctyl phthalate, 1% in acetone). Transfer 200 µl of sample out of the flask into a separate GC vial. Add 1 drop of citric acid into the vial.

Second step: Quantification of the total contents of *alpha-cypermethrin.*

Weigh (to the nearest 0.1 mg) 1 g of the impregnated net into a volumetric flask (100 ml) and add THF (50 ml). Place this flask in a refluxing apparatus. Heat up the flask in the apparatus and reflux at approximately 90 °C (oil bath temperature). Sampling will be done after 5 min refluxing. Add 0.5 ml of internal standard solution (dioctyl phthalate, 1% in acetone). Transfer 200 µl of the sample into a separate GC vial. Add 1 drop of citric acid into the vial.

(ii) Determination via the n-hexane extraction process

First step: Quantification of the adherent contents of *alpha-cypermethrin* on the surface of the fibers.

Weigh (to the nearest 0.1 mg) 1 g of the impregnated net into a volumetric flask (100 ml). Add n-hexane (50 ml). Shake the flask gently.
Sampling will be done after about 10 seconds.
Remove the net without adherent drops of n-hexane. Add 0.5 ml of internal standard solution (dioctyl phthalate, 1% in acetone). Transfer 200 µl of the sample into a separate GC vial. Add 1 drop of citric acid into the vial.

Second step: Quantification of the total contents of *alpha-cypermethrin*.

Transfer the net (without adherent drops of n-hexane) out of the flask into another volumetric flask (100 ml) and add THF (50 ml). Place this flask in a refluxing apparatus. Heat up the flask in the apparatus and reflux at approximately 90 °C. Sampling will be done after 5 min refluxing.). Add 0.5 ml of internal standard solution (dioctyl phthalate, 1% in acetone). Transfer 200 µl of the sample into a separate GC vial. Add 1 drop of citric acid into the vial.

(c) System equilibration. As for *alpha-cypermethrin* 454/TC/(M)3

(d) Determination. Each calibration solution C_i and each sample solution S_j is injected twice. The following sequence is advised:
C_A, C_A, C_B, C_B, C_C, C_C, S_A, S_A, S_B, S_B, S_C, S_C, ...

(e) Calculation. As for *alpha-cypermethrin* 454/TC/(M)3 and see test of linearity

$$\text{concentration} = \frac{f \times H_W}{I_q \times w}$$

where:
- f = response factor
- H_W = total peak area of alpha-cypermethrin (cis I + II) in the sample solution
- I_q = peak area of the internal standard in the sample solution
- w = mass of sample taken

(f) Surface concentration and release index

(i) Determination

The determination of the *alpha-cypermethrin* content is done via the n-hexane extraction process from the fibers.

Each calibration solution C_i and each sample solution S_j is injected twice. The following sequence is advised: C_A, C_A, C_B, C_B, C_C, C_C, S_{A1}, S_{A1}, S_{A2}, S_{A2}, S_{A3}, S_{A3}, S_{B1}, S_{B1}, ...

S_A = refers to first rinse; S_A mean value of S_{A1}, S_{A2}, S_{A3}
S_B = refers to second rinse; S_B mean value of S_{B1}, S_{B2}, S_{B3}
S_C = refers to third rinse; S_C mean value of S_{C1}, S_{C2}, S_{C3}
where as $_{1,2,3}$ = net samples

(ii) Calculation of the release index

Calculate the mean value of sample solutions S_C and S_B by the equations described in (e) and the release index for each piece of netting.

Release index = $1 - (S_C / S_B)$, where

S_B = refers to second rinse; S_B mean value of S_{B1}, S_{B2}, S_{B3}
S_C = refers to third rinse; S_C mean value of S_{C1}, S_{C2}, S_{C3}