**CIPAC**

COLLABORATIVE INTERNATIONAL PESTICIDES ANALYTICAL COUNCIL LIMITED

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

**CIPAC Prepublished Methods:**

Adopted CIPAC methods are available on a per-method basis as pdf-file, as long as they are not yet published in a Handbook. CIPAC charges a nominal fee per method. The list on the order form is exhaustive, which means that methods which are not on the list are not available. The list of methods (see <http://www.cipac.org/prepubme.htm>) covers the time period of adoption of methods at the from going to press of Handbook O (summer 2012) to present (September 2014, after CIPAC Meeting in Liège, Belgium). For further details see prepublished order form.

**Terms of Use:**

By ordering one or more of these methods, the user accepts the conditions of use as follows:

* these are draft methods and are subject to revision without further notice
* these methods will appear in revised and edited form in CIPAC Handbook O presumably in the year 2015. The prepublished methods expire as soon as they appear in printed form in the CIPAC Handbook O.
* The CIPAC copyright and disclaimer (cf. CIPAC Handbook N, page ii) applies to all prepublished methods.

**PERMETHRIN**

**331**

**PERMETHRIN Technical and EW Formulation**

As for permethrin technical **331**/TC/M2/- (CIPAC M, *p* 155) but add:

**4 Enantiomeric purity of permethrin**

# OUTLINE OF METHOD The sample is dissolved in hexane and the contents of permethrin stereoisomers (1*R*-trans, 1*S*-trans, 1*R*-cis, 1*S*-cis) are determined by high performance liquid chromatography on a Phenomenex LUX Cellulose‑3 column with detection at 235 nm (in combination with CIPAC 331/TC/M2/-, section 3e).

# REAGENTS

# *Hexane* HPLC grade

*Propan-2-ol* HPLC grade

*Water* HPLC grade

*Diluting solvent* hexane

*Eluent* hexane - propan-2-ol, 95 + 5 (v/v) containing 0.1 % water each

*Permethrin* control sample: (to be adapted upon finalization). Store refrigerated.

*Control sample solution.* Prepare a suitable solution of the control sample in hexane. This solution is used only for a column efficacy check to demonstrate the separation of the permethrin stereoisomers and to ensure their correct assignment via the relative retention time (solution R).

# APPARATUS

# *High performance liquid chromatograph* equipped with an automatic loop injector and an UV spectrophotometric detector capable of measuring at 235 nm

*Column* stainless steel, 250 × 4.6 mm (i.d.) packed with Phenomenex LUX Cellulose‑3, 3 µm

*Pre-column* Phenomenex Security Guard with 4 × 3 mm cartridge

*Electronic integrator* or *data system*

# procedure

*(a) Operating conditions* (typical):

*Column* 250 × 4.6 mm (i.d.) packed with Phenomenex LUX Cellulose‑3

*Pre-column* Phenomenex Security Guard with 4 × 3 mm cartridge

*Mobile phase* hexane - propan-2-ol, 95 + 5 (v/v) containing 0.1 % water each

*Flow rate* 1.2 ml/min

*Column temperature* 40 °C (see: *(c)* *Performance check*)

*Injection volume* 2 µl

*Detector wavelength* 235 nm

*Relative retention times*

|  |  |
| --- | --- |
| permethrin  stereoisomer | typical retention times |
|  |  |
| 1*S*-cis | 4.3 |
| 1*R*-cis | 5.0 |
| 1*S*-trans | 5.3 |
| 1*R*-trans | 6.2 |

*(b) Preparation of sample*. Prepare samples in duplicate. Weigh (to the nearest 0.1 mg) into a volumetric flask (50 ml) 40 to 60 mg of permethrin. Dissolve, allow to attain room temperature, and make up to volume with diluting solvent (solutions S1 and S2). Keep the samples solutions at constant room temperature.

*(c)* *Performance check*. Make replicate injections of the control sample solution to check the pattern and the separation of permethrin enantiomer. Adjust the column temperature so that the typical retention times given above differ by less than 0.3 minutes (see Fig. xx and table of relative retention times above). Measure the peak areas and determine the peak area ratios. Repeat until the values for subsequent injections differ by less than 2 %.

*(d) Determination.* Inject duplicate aliquots of each sample solution S1 and S2 and measure the peak areas. Repeat the measurement of control sample solution (solution R) after a series of 5 sample runs and at the end of the sequence.

*(e) Calculation*. Determine for each injection the sum of peak areas of permethrin stereoisomers and calculate the percentage of each peak. (The detector response of each stereoisomer is considered to be the same). Calculate the content of permethrin stereoisomers using the following formulae:

Content of individual permethrin stereoisomer *x*  g/kg

where:

*A* = total content of permethrin stereoisomers (1*R*/*S*-trans + 1*R*/*S*-cis) obtained under *Section* **3**(*e)* (g/kg)

*B(x)* = area percentage of individual permethrin stereoisomer *x* peak (%)

0.0

1.0

2.0

3.0

4.0

5.0

6.0

7.0

8.0

9.0

10.0

11.0

12.0

13.0

14.0

15.0

-10

25

50

75

100

125

150

175

200

225

250

275

300

325

350

mAU

min

1 - 1*S*-cis - 4.347

2 - 1*R*-cis - 4.973

3 - 1*R*-trans - 5.330

4 - 5.797

5 - 1*S*-trans - 6.183

WVL:235 nm

**Fig.xx Chromatogram of the four permethrin stereoisomers**