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**TRANSFLUTHRIN**

**741**

**TRANSFLUTHRIN Technical**

As for transfluthrin technical **741**/TC/(M)/- (CIPAC K, *p* 121) but add:

**4 Transfluthrin and its enantiomer**

# OUTLINE OF METHOD The sample is dissolved in hexane and the contents of transfluthrin (1*R*-trans) and its enantiomer (1*S*-trans) are determined by high performance liquid chromatography on a Phenomenex LUX Cellulose-1 column with detection at 230 nm (in combination with CIPAC 741/TC/(M)/-, section 3d). The method should replace the enantiomeric purity method under section 3.2 (CIPAC L, *p* 128).

# REAGENTS

# *Hexane* HPLC grade

*Propan-2-ol* HPLC grade

*Diluting solvent* hexane

*Eluent* hexane - propan-2-ol, 97 + 3 (v/v)

*Transfluthrin* 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 transfluthrin stereoisomers and to ensure the correct assignment of the transfluthrin enantiomer (1*S*-trans) 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 230 nm

*Column* stainless steel, 250 × 4.6 mm (i.d.) packed with Phenomenex LUX Cellulose‑1, 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-1

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

*Mobile phase* hexane - propan-2-ol, 97 + 3 (v/v)

*Flow rate* 1.0 ml/min

*Column temperature* 25 °C

*Injection volume* 2 µl

*Detector wavelength* 230 nm

*Relative retention times*

|  |  |  |
| --- | --- | --- |
|  | transfluthrin  stereoisomer | rel. retention time |
|  |  |  |
|  | 1*S*-cis | 0.74 |
|  | 1*S*-trans | 0.79 |
| transfluthrin | 1*R*-trans | 1.00 |
|  | 1*S*-cis | 1.07 |

*(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 transfluthrin. 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 transfluthrin enantiomer (see Fig. xx and the relative retention times given above). Measure the peak areas and determine the transfluthrin to transfluthrin enantiomer 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 the peak areas of transfluthrin (1*R*-trans) and its enantiomer (1*S*-tran*s*) and calculate the percentage of each peak. (The detector response of each enantiomer is considered to be the same). Calculate the content of transfluthrin and its enantiomer using the following formulae:

Content of transfluthrin (1*R*-trans)  g/kg

Content of the transfluthrin enantiomer (1*S*-trans)  g/kg

where:

*A* = transfluthrin content (1*R*/*S*-trans) obtained under *Section* **3**(*d)* (g/kg)

*B* = area percentage of the transfluthrin peak (%)

*C* = area percentage of the transfluthrin enantiomer peak (%)

0.0

0.5

1.0

1.5

2.0

2.5

3.0

3.5

4.0

4.5

5.0

5.5

6.0

6.5

7.0

7.5

8.0

8.5

9.0

9.5

10.0

10.5

11.0

11.5

12.0

-20

0

13

25

38

50

63

75

88

100

113

125

138

150

163

180

UV\_VIS\_1

mAU

min

1 - BCS-CV15694 (1S-cis) - 4.263

2 - BCS-CV15695 (1S-trans) - 4.517

3 - **AE 0035474 (1R-trans)** - 5.727

4 - AE 1371433 (1R-cis) - 6.123

WVL:235 nm

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