

CIPAC

COLLABORATIVE INTERNATIONAL PESTICIDES ANALYTICAL COUNCIL
LIMITED

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

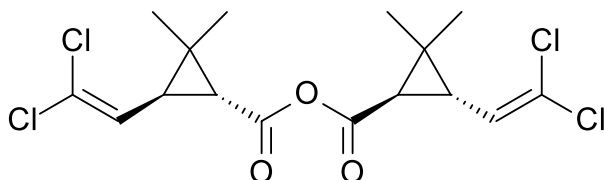
CIPAC Free relevant impurities methods:

Methods for relevant impurities becoming more and more important in the quality control of TK/TC and FAO-specifications. In order to meet an urgent need for methods to characterize TK/TC in a.i. and formulations, CIPAC provides selected methods as download. By downloading these methods, you accept the following conditions of use.

Terms of Use:

- these methods are provided subject to changes without prior notice
- the copyright of these methods is with CIPAC
- CIPAC declines any responsibility when using these methods

TRANSFLUTHRIN
PAA Impurity, 1R-trans-permethric acid anhydride

<i>Report Name</i>	1R-trans-permethric acid anhydride
<i>Chemical name</i>	(1R,3S)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylic anhydride (IUPAC) cyclopropanecarboxylic acid, 3-(2,2-dichloroethenyl)-2,2-dimethyl-, 1,1'-anhydride, (1R,1'R,3S,3'S)- (CA; Registry 1664359-57-2)
<i>Structural formula</i>	
<i>Empirical Formula</i>	C ₁₆ H ₁₈ Cl ₄ O ₃
<i>RMM</i>	400.1

TRANSFLUTHRIN 741

See CIPAC K, page 121

TRANSFLUTHRIN TECHNICAL *741/TC/M/-

1 Sampling. Take at least 25 g. Do not heat the sample to liquefy; mechanically homogenize solidified samples instead. Use samples which are liquid at room temperature as such.

2 Identity tests.

2.1 HPLC. Use the HPLC method described below. The relative retention time of 1*R*-trans permethric acid anhydride in the sample solution should not deviate by more than 4 % from that of the calibration solution.

2.2 UV spectroscopy. Record the UV spectrum during the HPLC determination. The UV spectrum obtained from the sample and reference item should not differ significantly. (Fig. 1)

4 1*R*-trans permethric acid anhydride.

OUTLINE OF THE METHOD.

1*R*-trans permethric acid anhydride contents are determined (ppm) by reversed phase high performance liquid chromatography using UV detection at 210 nm and external standard calibration.

* CIPAC method 2018. Based on a method supplied by Bayer Crop Science, Germany

REAGENTS AND EQUIPMENT

1R-trans permethric acid anhydride reference standard of known content

Acetonitrile (HPLC grade)

Phosphoric acid (HPLC grade, 85 – 90 %)

Purified water (HPLC grade)

Eluent A: phosphoric acid 0.01 mol/l

Eluent B: acetonitrile

APPARATUS

New generation high performance liquid chromatograph equipped with an injection system and a sensitive UV spectrophotometric detector operating at 210 nm.

Liquid chromatography column, stainless steel, 100 x 4.6 (i.d.) mm, packed with Zorbax SB C8, 1.8 µm or equivalent with the same selectivity.

Electronic integrator or data system.

Ultrasonic bath.

Polypropylene injection vials

PROCEDURE

(a) *Operating conditions (typical):*

Flow rate: 2.0 ml/min

Column temperature: 70 °C

Injection volume: 3 µL

Detector wavelength: 210 nm

Gradient:

time (min)	% A	% B
0.0	40	60
2.0	40	60
8.0	5	95
8.1	0	100
12.0	0	100
12.1	40	60
14.0	40	60

Retention time: approx. 4.6 min for 1*R*-trans permethric acid
anhydride
Total run time: approx. 14 min

(b) Equilibration of the system. Pump sufficient mobile phase through the column to equilibrate the system. Inject 3 μL portions of the calibration solution C1 and repeat the injections until retention times and peak areas deviate by less than $\pm 1\%$ from the mean for three successive injections.

(c) Calibration solution. Ensure temperature equilibration for all dilution steps. Weigh (to the nearest 0.1 mg) approximately 10 mg of the reference item into a volumetric flask (100 ml). Add 20 ml of acetonitrile and place the flask in an ultrasonic bath for 5 minutes. Make up to volume with acetonitrile. Transfer 1 ml of this solution into a separate volumetric flask (100 ml) and make up to volume with acetonitrile (Calibration solution C1). (Fig. 2)

(d) Sample preparation. Ensure temperature equilibration for all dilution steps. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) 100 mg of Transfluthrin into a volumetric flask (10 mL). Add 5 mL acetonitrile and place the flasks in an ultrasonic bath for 5 min. Make up to the mark with acetonitrile (Sample solutions S1, S2). (spiked sample solution Fig. 3)

(e) Determination. Inject in duplicate each calibration and sample solution and bracket a series of sample solution injections by injections of the calibration solution as follows: calibration solution, sample solution, calibration solution, ... (C1, S1, S2, C1, ...).

Do not use the calibration solution vials for more than two consecutive injections. Prepare fresh fillings from the stock solution instead. Determine the peak areas of 1*R*-trans-permethric acid anhydride.

(f) *Calculation.* Calculate the response factors from the calibration solution bracketing the injections of the sample solutions. Average the response factors of the calibration solutions preceding and following the sample solution injections. These must agree within $\pm 2\%$ of the average otherwise repeat the determination. Calculate the content of the sample solutions.

$$f_i = \frac{s \cdot P}{H_s}$$

$$\text{content} = \frac{H_w \cdot f}{w}$$

Where:

f_i = single response factor

f = average response factor

H_s = peak area of 1*R*-trans-permethric acid anhydride standard in the calibration solution

H_w = peak area of 1*R*-trans-permethric acid anhydride in the sample solution

s = weight of the 1*R*-trans-permethric acid anhydride standard in the calibration solution (mg/10 mL)

w = weight of the sample (mg/10 mL)

P = purity of the 1*R*-trans-permethric acid anhydride standard (g/kg)

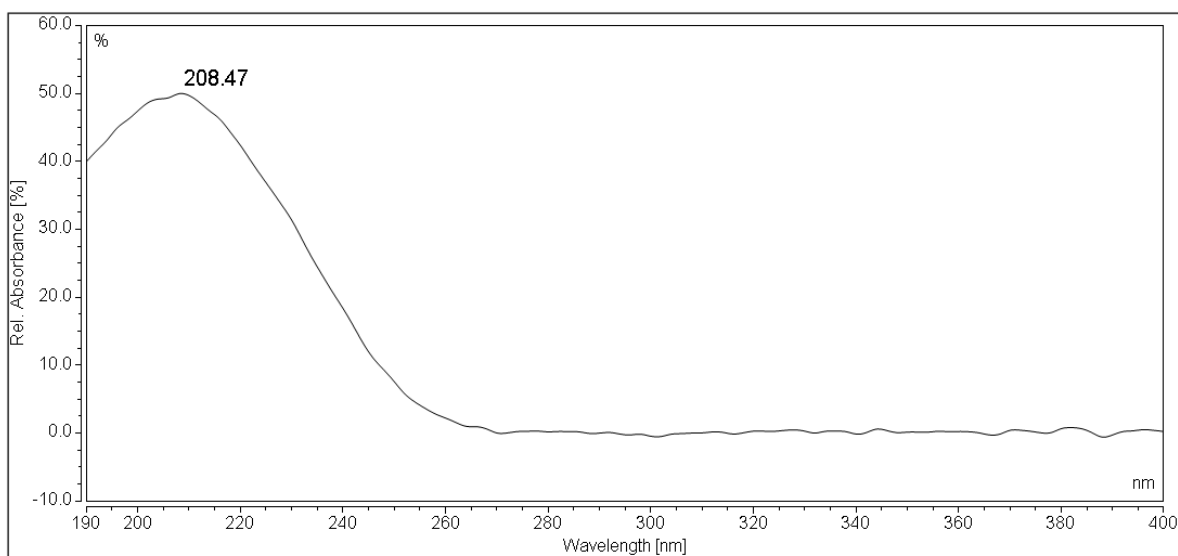


Fig. 1 UV-Spectrum of 1*R*-trans-permethric acid anhydride

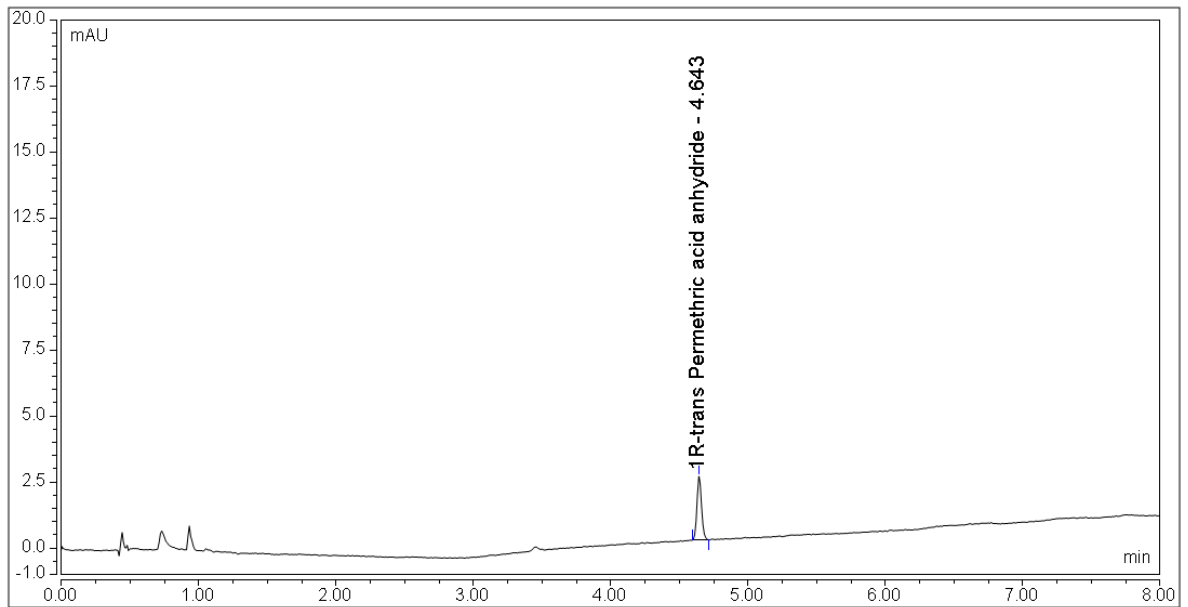


Fig. 2 Chromatogram of 1R-trans-permethric acid anhydride analytical standard

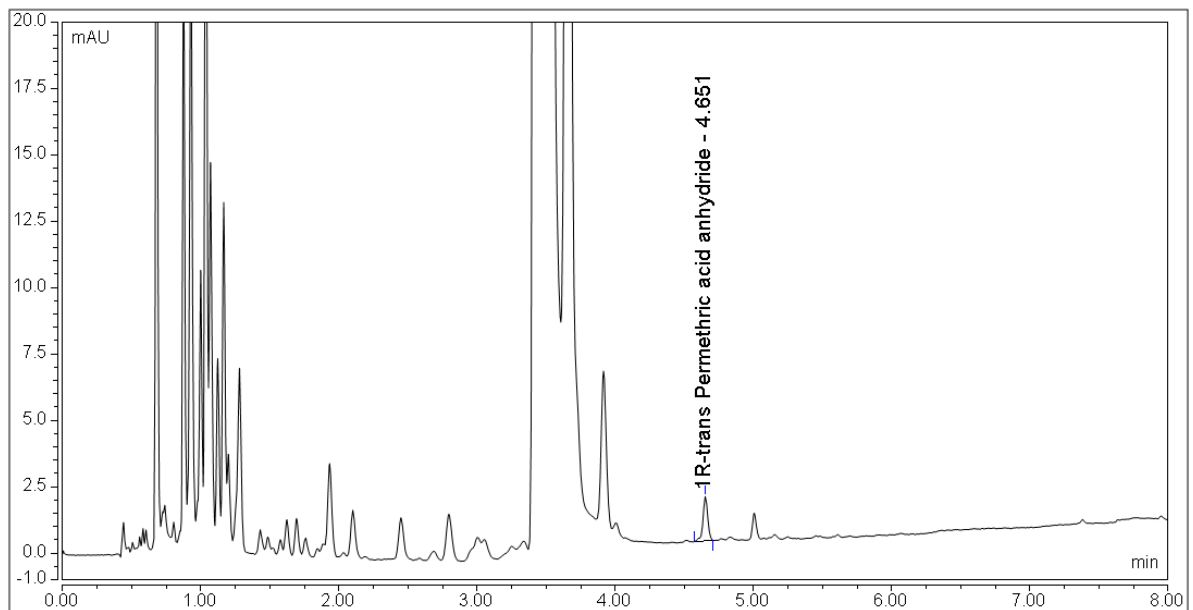


Fig. 3 Chromatogram of a spiked technical Transfluthrin batch (Content of 1R-trans-permethric acid anhydride of approx. 70 ppm)