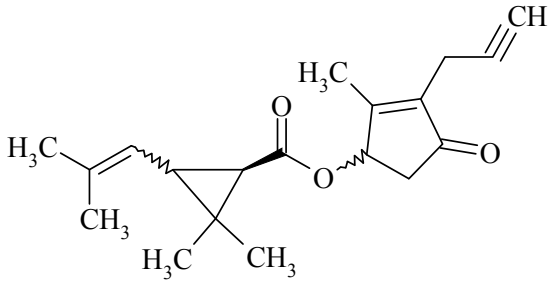
CIPAC STATUS REPORT

04/08/2019



0743 Prallethrin

Allocated to J

CIPAC methods published in:

CIPAC L, p. 110

CIPAC 46th meeting, June 2002 in Rome

Dr Furuta presented a report of the small-scale study of the capillary GC method for the determination of prallethrin in technical material. The compound is a full racemic mixture and hence the identity test does not require consideration of isomer ratios. It was agreed the method could go to full collaborative study.

CIPAC 48th meeting, June 2004 in Brno

Ms. Furuta presented the results of a collaborative study on two technical materials, and three liquid vaporizers (LV) using capillary GC, FID and triphenyl phosphate as internal standard. Twelve laboratories participated in the study. None of the outliers or strugglers were eliminated. JAPAC proposed the method to be accepted as provisional CIPAC method. The stereoselective identity test for prallethrin is based on the hydrolytic cleavage of the ester bond and analysis of the resulting acid and alcohol by two different chiral HPLC-columns. Mr. Müller asked if the isomer ratio of the alcohol and acid moiety had been un-changed during hydrolysis. The reply was that it was checked and that the configuration does not change.

Decision The capillary GC method (CIPAC/4363) for determination of prallethrin in TC and LV formulations was accepted as **provisional** CIPAC method. The HPLC method as a quantitative identity test remains tentative.

CIPAC 49th meeting, June 2005 in Utrecht

Ms R. Furuta presented an amended analytical method for the determination of the stereoisomer composition of prallethrin. Additional validation data showed the reliability of the method, the absence of racemisation was confirmed and explanations for the different saponification procedures were given in the test. The identity test was considered acceptable.
Decision The capillary GC method (CIPAC/4363) for determination of prallethrin in TC and LV formulations was accepted as **full** CIPAC method. The HPLC method (CIPAC/4435) for the determination of stereoisomer composition was accepted as a quantitative identity test.

CIPAC 61th meeting, June 2017 in Rome

Mr John Dawson presented the **extension of method** CIPAC 743/LV/M/- total prallethrin content, to the UL formulation type, which contains prallethrin, with a few modifications. This report was prepared to demonstrate the validity of the extension of the CIPAC 743/LV/M/- for total prallethrin to UL formulations.

In order to apply the CIPAC 743/LV/M/- methodology to the formulation of interest, (CMP123-004), the following modifications were applied:

- ➢ Detector temperature changed from 270 ºC to 320 ºC

- ➢ Column oven temperature changed from 245 ºC isothermal, to 50 ºC for 0.5 min then 40 ºC/ min up to 240 ºC for 15min. This was necessary in order to ensure complete separation of formulation components from the prallethrin peak.

- ➢ Injection port temperature changed from 270 ºC to 275 ºC

- ➢ Carrier gas flow changed from 35 cm/s to 41.541 cm/s (flow rate 2 ml/min)

Furthermore, due to a combination of the large sample weight required, and other ingredients of the formulation, separation of the internal standard triphenyl phosphate in the sample solution was not achievable. The calculations to determine the amount of prallethrin content were changed to external standard quantitation.
The data shown demonstrated that the method is linear, specific, and has acceptable precision (repeatability, r). Therefore, the modified method was considered appropriate for the determination of total prallethrin in a UL formulation and the extension of CIPAC 743/LV/M/- to UL formulations was proposed by Clarke International.

The following comments were received from the meeting:

- ➢ Observations were made by several participants of the meeting regarding the use of internal standard and different conditions in the method. It was a controversial issue whether these changes were minor or major changes. Finally, it was considered that there were so many changes that the method could be considered as an extension of the existing method.

Closed Meeting:
The following comments were received from the meeting:
Observations were made regarding the necessity to use of external standardisation instead of internal standardisation, different temperature program. It was a controversial issue whether these changes were minor or major changes. Finally, it was considered as a major change and the meeting did not agree to consider it as a method extension. As a consequence, the study has to be repeated with a proper internal standard and in at least two different laboratories.

CIPAC 62nd meeting, June 2018 in Panama City

Prallethrin method extension to UL by Mr Kevin King (5163, 5164)
Mr King presented the results of a validation study in which the extension of the CIPAC method for total prallethrin LV (743/LV/M) to an ultra-low volume liquid (UL) formulation type was investigated. The proposed method, based on capillary gas chromatography using flame ionization detection and triphenyl phosphate (TPP) as internal standard, was identical to the original method with exception of the inlet and detector temperature which were changed 270ºC to 325ºC. This was considered to be a minor modification. During the study it became clear that a matrix effect caused suppression of the prallethrin signal with approx. 25%. Correct quantification was obtained by using standard addition methodology. An additional identity check can be performed with the aid of GC-MS on an Agilent HP-5ms (5%-phenyl)-methylpolysiloxane: 30m x 250µm x 0.25µm).
The validation study was performed by two participating laboratories (five replicates) and resulted in an overall RSDR of 2.28% which is well within the Horwitz criteria (HorRat 0.54), and proven linearity.
The organizers propose to extend CIPAC 743/LV/M/- to UL formulations.

No comments were received from the meeting.

Closed Meeting:
The requested additional dataset was presented, therefore the method extension can be promoted to
a **provisional** method.

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CIPAC 63rd meeting, June 2019 in Braunschweig

Closed Meeting:
At the previous meeting, the method was accepted as provisional. No further comments were received. The method can be promoted to a **full CIPAC method.**