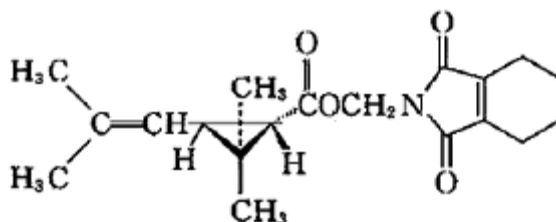# CIPAC STATUS REPORT

22/09/2018

0989   d-tetramethrin

Not allocated

CIPAC methods published in:

CIPAC

CIPAC   60th meeting, June 2016 in Tokyo

d-tetramethrin by Mr Quibai Jiang (5040, 5041)

Mr Jiang presented the results of a small scale trial on the determination of the active ingredient content and isomer ratio of the diastereomers of d-tetramethrin in technical material. d-tetramethrin is a mixture of the isomers (1*R-trans*, *R*), (1*R-trans*, *S*), (1*R-cis*, *R*) and (1*R-cis*, *S*) of tetramethrin in an approximate ratio of 4:4:1:1. In practice the *trans* isomer range is 75-85 % and the *cis* isomer range is 15-25 %.

d-tetramethrin was determined by gas chromatography with internal standardization and the isomer ratio of the diastereomers was determined by normal phase high performance liquid chromatography using a CHIRALPAK® AY-H, 250 × 4.6 mm (i.d.), 5 µm column, hexane/ethanol/diethylamine, 930/70/1 (v/v) mobile phase, detection wavelength 230 nm.

Three TC samples were sent to three independent laboratories. During the trial the participants conducted duplicate determinations on two different days with duplicate injections for each sample.

The statistical evaluation was carried out according to the CIPAC guidelines. The results were tested for outliers firstly using Cochran's test on the within laboratory variance and then using Grubbs test on laboratory means to test the between laboratory variance. The tests were carried out at the alpha level of 0.01 for outliers and 0.05 for stragglers.

After the initial evaluation the calculated RSD_R fulfilled the Horwitz criteria for two technical materials. One straggler occurred, using Cochrans' test (for TC-A material, in case of laboratory 2).

On the basis of the results, Mr Jiang proposed to proceed to a large scale collaborative study.

The following comments were received from the meeting:

➢ For reasons of safety would it be possible to use heptane in the mobile phase instead of hexane? Were there any tests carried out with heptane? The answer was that this option was not checked.

➢ In the presentation it was mentioned that the isomer ratio is roughly 4:4:1:1, but on the slide presented this ratio could not been observed. The answer was that the figure on slide 7 was for the technical material and not for the pure standard.

➢ Does the technical material have an ISO common name? The answer was that they are going to apply for an ISO common name.

Closed Meeting:

The problem with the isomer ratio was clarified. The Company should apply for ISO common name.

It was proposed to go for full scale collaborative study with the modification of using heptane instead of hexane.

CIPAC   61th meeting, June 2017 in Rome

Mr Quibai Jiang presented the results of a full scale trial on the determination of the active ingredient content and isomer ratio of the diastereomers of d-tetramethrin in technical material.   d-tetramethrin is a mixture of the isomers (1R-trans, R), (1R-trans, S), (1R-cis, R) and (1R-cis, S) of tetramethrin in an approximate ratio of 4:4:1:1. In practice the trans isomer range is 75-85   % and the cis isomer range is 15-25 %.

d-tetramethrin was determined by gas chromatography/flame ionization detection, with internal standardization. The isomer ratio of the diastereomers was determined by normal phase high performance liquid chromatography using a CHIRALPAK® AY-H HPLC column, 250×4.6 mm (i.d.), 5 µm particle size; n-heptane-ethanol-diethylamine, 930+70+1 (v/v) mobile phase, and detection wavelength of 230 nm.

Five TC samples were sent to fourteen laboratories. During the trial the participants conducted duplicate determinations on two different days with duplicate injections for each sample.

The statistical evaluation was carried out according to the CIPAC guidelines. The results were tested for outliers, firstly using Cochran's test on the within laboratory variance and then using Grubbs test on laboratory means to test the between laboratory variance. The tests were carried out at the alpha level of 0.01 for outliers and 0.05 for stragglers.

During the check of the GC results, three Cochran outliers occurred. After eliminating these data, no other outliers were found.

After the evaluation, the calculated RSDR fulfilled the Horwitz's criteria for all technical materials.

Based on these results, the organizers recommended that the d-tetramethrin method should progress to
a fully validated CIPAC method.

The following comments were received from the meeting:
- Mr Bura drew the attention on the problem of the anonymity of the participating laboratories and asked that in the future the organisers should respect this demand.
- One participant asked whether it was necessary to use dichloromethane as solvent.
- Mr Bura showed that even without eliminating the outliers the results of the measurements met the
Horwitz's criteria.
- The remarks about the quality of the results of laboratory 5 in the conclusion of the presentation were based on an assumption, not on actual data.

Closed Meeting:
A full scale collaborative trial was presented, the method was accepted as provisional.
(One remark was received: To contact ISO for ISO common name.)
The capillary GC method (CIPAC/5101) using internal standard for the determination of d-tetramethrin in TC formulations and the chiral phase HPLC method (CIPAC/5101) for the determination of the isomer ratio of d-tetramethrin in TC formulations were accepted as **provisional** CIPAC methods.

CIPAC   62nd meeting, June 2018 in Panama City

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.

Decision: The capillary GC method (CIPAC/5101) using internal standard for the determination of dtetramethrin in TC formulations and the chiral phase HPLC method (CIPAC/5101) for the determination of the isomer ratio of d-tetramethrin in TC formulations were accepted as **full** CIPAC methods.