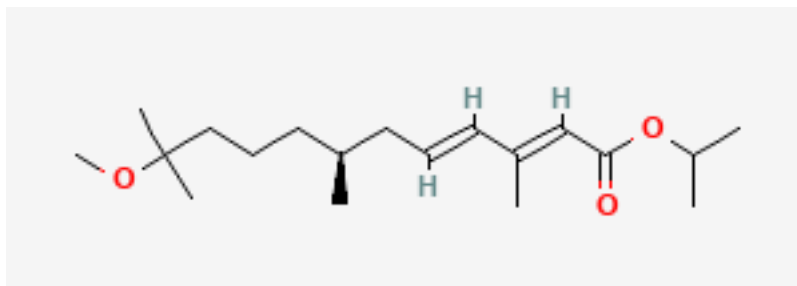


CIPAC STATUS REPORT

11.08.2024



1026 S-Methoprene

Allocated to CHIPAC

CIPAC 66th meeting, June 2022 virtual

S-methoprene by Ms Junhua Song (5307, 5308, 5309, 5310)

Ms Song presented the results of a small scale collaborative study for the determination of free (non-encapsulated) methoprene in three CS formulation by GC-FID on a capillary chemically bonded and cross-linked dimethylpolysiloxane stationary phase with internal standardization. Four laboratories were invited and all reported results in time. The extraction method consisted of an extraction step with *n*-hexane and a rotary evaporator as rolling apparatus. Each sample was analyzed in quadruplicate on two different days and no major method deviations were registered apart from laboratory four which was excluded from the dataset. After statistical evaluation of the data following the “Guidelines for CIPAC Collaborative Study Procedures for Assessment of Performance of Analytical Methods”, the HorRat values ranged from 2.8-6.4. This was attributed to the dynamic equilibrium between the inside and outside of the capsule, and the uncertainties during the extraction process. However, considering the fact that the purpose of the method is to monitor the content of free S-methoprene in CS formulations, as indicated in the proposed WHO specification “not greater than 5% of the total AI content”, it is believed that the method can fulfil its function.

Ms Song proposed to go forward to a full scale CIPAC collaborative trial.

The following comments were received from the meeting:

- Ms McNally asked whether there were any proposals for change from the participants or they just sent the results. The answer was that the participants didn't send proposals, they used the method as it was requested. The only comment was the amount of water needed for dispersion.
- Mr Pigeon remarked that the use of the rotary evaporator might be the cause of the deviations. He also asked whether other types of equipment were tested as replacement of the rotary evaporator. This was not the case. A complication when using a rotary evaporator is that solvent can evaporate. However, as the internal standard is added before using the rotary evaporator no influence on the quantitation procedure is expected. Ms Song answered that the rotary evaporator is a more common equipment than a roller and that there wasn't much evaporation during the extraction.
- Mr Pigeon asked whether the proposed method could also be applied for determination of the release rate. This was not tested.

CIPAC STATUS REPORT

11.08.2024

Closed meeting:

Ms Song presented also the results of a small scale collaborative study for the determination of the ratio of *R*- and *S*-methoprene in three TC samples by normal phase using a Chiralpak AD-H silica HPLC column with UV-detection at 254 nm. Four laboratories were invited and 3 reported results in time. The samples were dissolved in *n*-hexane and analyzed in quadruplicate on two different days. There weren't major method deviations registered apart from laboratory four which was excluded from the dataset. After statistical evaluation of the data following the "Guidelines for CIPAC Collaborative Study Procedures for Assessment of Performance of Analytical Methods", the HorRat values ranged from 0.15-0.16.

Ms Junhua Song recommended to go forward to a full scale CIPAC collaborative trial.

The following comments were received from the meeting:

- Mr Pigeon asked whether the method could be applied to formulated products. This was not tested and as the column is rather expensive this will not be proposed.
- Mr Pigeon asked if there is the intention also to develop a method for release rate. Ms Song will check with the company.

Closed meeting:

On the small scale collaborative study for the determination of the ratio of *R*- and *S*-methoprene in three TC samples Ms Vinke, Mr Pigeon, and Mr Patrian asked to clarify the aim of the method. Clarification was also asked why Turbovap equipment was used as horizontal rollers are described in the original CIPAC method. This should be clarified and Mr Pigeon will contact Ms Song.

CIPAC 67th meeting, June 2023 Braunschweig

***S*-Methoprene by Ms Junhua Song (5359, 5360)**

Ms Song presented the results of a full scale collaborative study for the determination of *S*-methoprene in three TC materials by normal phase HPLC. Twelve laboratories participated and nine laboratories from Europe and China reported results. The aim of the analysis was to distinguish *R*-methoprene from *S*-methoprene and to determine the *S*-methoprene content of TC material. The sample was dissolved in *n*-hexane and the ratio of *S*-methoprene is determined by normal phase HPLC on a Chiralpak AD-H silica column with detection at 254 nm. One laboratory reported that resolution of the peaks of interest could not be achieved and that retention times drifted during the run. However, the data of this laboratory were not rejected. The statistical evaluation was performed according to DIN ISO 5725 and the 'Guidelines for CIPAC Collaborative Study Procedure for Assessment of the Performance of Analytical Methods'. The results were in line with the requested HorRat ratios with 0.47, 0.55 and 0.51 respectively.

Ms Song proposed the method to be accepted as a provisional method.

The following comments were received from the meeting:

- Mr Patrian asked what the purpose of the analysis was as one laboratory clearly indicated that no separation could be achieved but the result was accepted for final evaluation. Is it to analyse *S*-methoprene as an identity, or to quantify *S*-methoprene? Ms Song answered that this was developed for the purpose of the FAO specification.
- Mr Grassi asked whether the current CIPAC method distinguished between the different isomers and Ms Song answered that the current method only is capable of analysing the total methoprene content.

CIPAC STATUS REPORT

11.08.2024

Closed meeting:

Mr Hänel suggested that a decision should be postponed as the results from Lab 9 should be discarded and statistics should be recalculated. If the recalculated results are within the relevant criteria the method can be promoted to **provisional** CIPAC method. Mr Hänel and Mr Bura will contact Ms Song.

CIPAC 68th meeting, June 2024 Wageningen

1026 S-methoprene

Closed meeting:

The normal phase HPLC method (CIPAC/5359) for the determination of S-methoprene in technical materials was accepted as **provisional** CIPAC method as after the elimination of the results of laboratory 9 the recalculated results were within the relevant criteria.