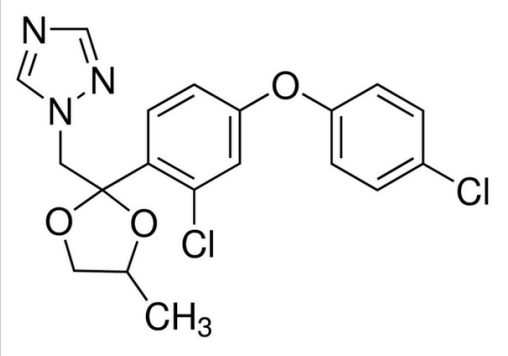
CIPAC STATUS REPORT

29.08.2023



0687 Difenoconazole

Allocated to DAPA

CIPAC methods published in:

CIPAC

CIPAC 65st meeting, June 2021 virtual

Difenoconazole by Mr Christian Mink (5179, 5180): Mr Christian Mink presented the results of a small scale collaborative trial for the determination of difenoconazole in TC, WG and EC formulation (5280/R) with a capillary GC method using a 60 m x 0.25 mm internal diameter DB-5 phase, with film thickness of 0.5 µm. Four participants took part in the DAPA collaborative trial. One laboratory had technical difficulties and was not CIPAC/5294/P 4 considered, 3 laboratories used 30 m column instead of 60 m. One laboratory conducted the trial twice (60 m and 30 m Column). All method deviations, noted by the participants, were deemed not to affect the analytical results significantly. The between laboratory experimental relative reproducibility standard deviation (% RSDR) was below the acceptance limit based on the Horwitz curve calculation for all samples tested, even without elimination of outliers or stragglers. The method simultaneously allows the determination of the ratio of the cis- and transdiastereomers of difenoconazole.
The organizers proposed the method to go for a CIPAC full scale collaborative trial.

The following comments were received from the meeting:
Mr Haustein noted that the HorRat value should be included in the full scale report.
Mr. Pigeon asked if differences in resolution were seen using 30 or 60 m long column and He or H_2 as eluent. Mr Mink answered that there weren't differences, however he would recommend the 60 m long column, as validation was done with this column.
Mr Garvey questioned the necessity of using the internal standard. The answer was that the position in the chromatogram of the internal standard would allow it, however the method was developed with the Istd.

Closed Meeting:
A small scale trial was presented and the method was proposed for a large scale collaborative trial with the remark to clearly define in the method description the column length, the eluent and the internal standard.

CIPAC 66th meeting, June 2022 virtual

Difenoconazole by Mr Christian Mink (5324, 5325)

Mr Christian Mink presented the results of a full scale collaborative trial for the determination of difenoconazole by GC-FID on a DB-5 or equivalent phase with hydrogen as carrier gas, and internal standard quantitation in two TCs, one WG and two EC formulations. 23 laboratories participated and 21 have returned their results in time. Seven laboratories used H_2, 11 used He, four used N_2 and two laboratories changed the stationary phase. Including the data of all participants the HorRat values ranged from 0.5-1.4, whereas after elimination of two outliers/ stragglers the HorRat values ranged from 0.5-1.0. A breakdown of the data set to evaluate whether the use of different carrier gasses was of influence to the results showed that when using H_2 or He the HorRat values were acceptable, even without removing outliers or stragglers suggesting that He could be added as an alternative carrier gas to H_2.

Mr Mink also investigated whether the proposed method could be used to investigate the isomeric ratio between the *cis* and *trans* isomers of difenoconazole in two TC samples. It was concluded that after elimination of one outlier the data were consistent with HorRat values ranging from 0.4-1.0 (n=14) indicating that the method was fit for purpose.

Mr Mink considered the method to be suitably validated and recommended this method to be accepted as a provisional CIPAC method.

The following comments were received from the meeting:

- Mr Hänel remarked that the aim of CIPAC trials is to validate an analytical method and not to show that the laboratory can analyse that active substance.
- Mr Bura noticed that there are two data sets, one using H as carrier gas and one using He. If the method is strictly followed, there are only 7 valid results, however there are 11 acceptable results using He as carrier gas. If the two data sets are considered together, the method description should allow the use of both gases.
- Mrs Nováková requested why no report was sent to the participants before the meeting. Mr Mink apologized, it was due to time constraints. Furthermore, she requested why a total run time of 44 minutes was given as the compounds of interest eluted before 20 minutes and no further peaks can be seen after 25 minutes. Mr Mink replied that an impurity elutes at around 45 min, in consequence, if the runtime is shortened, interference may appear with the impurity.
- Mr Pigeon asked whether a difference in efficacy was related to both isomers. This was not known.
- Mrs Tessier asked whether both isomers were truly baseline separated as it could not be assessed based on the presented chromatograms. This was confirmed, the isomer separation is needed, because of specifications in some countries.
- Mr Garvey asked whether the deviation of Lab 7 could be assigned to a deviating method. This was not the case as only He was used as carrier gas and no further method deviations were reported.

Closed Meeting:
Mrs Tessier requested whether baseline separation was achieved. Mr Patrian, seconded by Mr Pigeon, added that this was nearly achieved so the separation was sufficient.

Mr Bura requested what kind of dataset was accepted as several carrier gases have been applied. The data from 15 participants (H_2 and He based) were accepted.

Mr Garvey requested what the actual method was as many variations were present, therefore, a stricter description was required. Ms Vinke, seconded by Mr Pigeon, replied that both H_2 and He

were carrier gases leading to acceptable results, only N_2-based analytical results were not acceptable.

The method can be promoted to a **provisional CIPAC method**. considering the data sets using hydrogen or helium as eluent gas with the need for a stricter description of the method.

CIPAC 67th meeting, June 2023 Braunschweig

Decision

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 considering the data sets using hydrogen or helium as eluent gas with a stricter description of the method.