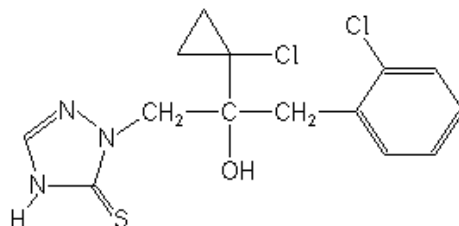


CIPAC STATUS REPORT

16/09/2020



0745 Prothioconazole

Allocated to DAPA

CIPAC methods published in:

CIPAC Handbook P

CIPAC 61th meeting, June 2017 in Rome

Mr Friedhelm Schulz presented the results of a small scale collaborative trial carried out by DAPA with 8 laboratories on one TC and four different formulation samples (EC (250 g/L prothioconazole); FS (100 g/L prothioconazole); FS (250 g/L penycuron + 8 g/L prothioconazole) and SC (480 g/L prothioconazole).

The homogenized samples containing prothioconazole were dissolved in solvent mixture of acetonitrile/purified water followed by active ingredient determination using gradient reversed phase high performance liquid chromatography with UV detection at 254 nm and external standard calibration.

After the evaluation, the calculated RSD_R fulfilled the Horwitz's and HorRat criteria for all analysed samples.

Based on these results, the organizers recommended that the prothioconazole method should progress to a full scale CIPAC trial.

The following comments were received from the meeting:

- It was asked if sonication for 50 min was necessary and in which cases a shorter sonication time would be enough? The answer was that for this type of formulation the relatively long sonication time was needed.
- Some participants to the trial filtered, other centrifuged the samples. Was the centrifugation necessary? Both methods can be used.

Closed Meeting:

The method was proposed for a full scale collaborative study

CIPAC 62nd meeting, June 2018 in Panama City

Prothioconazole by Mr Friedhelm Schulz (5159, 5160)

Mr Friedhelm Schulz presented the results of a full scale collaborative trial for prothioconazole in one TC and four different formulation samples: one emulsifiable concentrate (EC 250), two flowable concentrates for seed treatment (FS 100 and FS 258), and one suspension concentrate (SC 480) with 20 participants from America, Asia, and Europe.

The samples were dissolved in an acetonitrile/water solvent mixture followed by active ingredient determination using HPLC based chromatography on an Agilent Zorbax Extend C18, 50 mm x 4.6 mm, particle size 3,5 µm HPLC-column, a 10 mM phosphoric acid in water/acetonitrile-tetrahydrofuran-methanol (50-25-25) gradient, UV detection at 254 nm and external standard calibration.

CIPAC STATUS REPORT

16/09/2020

The participants reported several deviations, mainly related to the use of different reversed phase HPLC columns. Also deviations of the prescribed flow rate, injection volume and the use of filtration instead of centrifugation were reported.

Statistical evaluation revealed one Grubb's outlier in FS 100, one in FS 258, and one in SC 480.

Horwitz criteria were met for all five samples when the outliers were removed. However incorporating the outlier data resulted in RSDR values which were above the Horwitz criteria for FS

100, FS 258, and SC 480. After removal of outlier data HorRat values of 0.43, 0.56, 0.74, 0.65, and 0.74 were reported for TC, EC 250, FS 100, FS 258, and SC 480 respectively.

The organizers recommended that the method should be accepted as a provisional CIPAC method in technical samples as well as EC-, FS- and SC-formulations.

The following comments were received from the meeting:

- Mr Manso asked how many potential participants have reacted and how many labs were selected. Mr Schulz replied that 40 potential participants have reacted, that 25 were selected for participation and that results of 20 participants were received. The selection of participants was based on a "first come-first served" procedure. Mr Hänel added that CIPAC has no guideline for the selection of participants in CIPAC trials. However a proposal from DAPA regarding this subject is on the agenda of this meeting at 6.3.
- Mr Garvey asked whether the outliers could be traced back to deviations from the method. The answer was that the outliers had deviations from the method.

Closed Meeting:

A full scale trial was presented and the method can be promoted to a **provisional** CIPAC method. However, a comment on the applicability of filtration instead of centrifugation should be added.

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**.

CIPAC 64th meeting, June 2020 virtual (Geneva, Corona)

The reversed phase HPLC method (ESI-MS/MS in MRM mode) for the determination of the relevant impurity prothioconazole-desthio in prothioconazole TC, SC, EC and FS formulations (CIPAC/5251) was noticed and **adopted**.