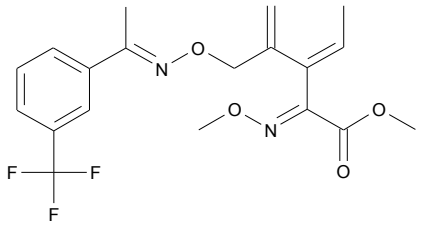
CIPAC STATUS REPORT

19.09.2021



0617 Trifloxystrobin

Allocated to D

CIPAC methods published in:

Not published

CIPAC 58th meeting, June 2014 in Liège

Mr Haustein presented the results of a **full scale** collaborative study on the determination of trifloxystrobin in technical material (TC), emulsifiable concentrate (EC), flowable concentrate for seed treatment (FS), suspension concentrate (SC), water dispersible granules (WG) and ready to use liquids (AL) using HPLC-UV, with a C18 column (temperature = 50°C), detection at 280 nm and external standard calibration.
Two samples of TC, 1 sample of AL, 1 sample of EC, 1 sample of FS, 1 sample of WG and 1 sample of SC were provided. 33 laboratories offered to participate, however the trial was limited to 24 participants due to the availability of samples for the study. The first 24 respondents were therefore chosen. Data were received from 22 laboratories within the requested timeframe.
The results of one participant were rejected as a number of major changes to the method were introduced:

- Use of a non-equivalent separation column
- Change of column-dimensions
- Change of flow-rate
- Change of injection volume

As a result retention time and peak-width changed significantly. Moreover, the laboratory remarked on additional retention time variation
The statistical evaluation was carried out according to the CIPAC guidelines. The following outliers and stragglers were identified: For the FS Lab 20 was identified as a Grubb's straggler
No other statistical outliers were identified. All results were initially included in the evaluation. In the initial evaluation the Horwitz criteria were met for all samples.
Mr Haustein concluded that the method is suitable and proposed the method be adopted as provisional by CIPAC.

The following comments were received from the meeting:

- Do you need to place the standard in an ultrasonic bath for 15 min to dissolve it? In our experience this is not necessary. Mr Haustein replied that this was to ensure that the sample was homogenous but agreed that it was not necessary.
- Why did you choose an injection volume of 3 µl – this may be too small for some laboratories? Mr Haustein replied that good repeatability can be achieved with a 3 µl injection volume without causing column saturation He also noted that some of the labs that participated had used 5 µl injection with no problems.
- Why did you choose detection at 280 nm when the absorbance maximum is approx. 250 nm? Particularly for the AL formulation this lead to a small peak as it was a much lower concentration than the other samples. Mr Haustein replied the wavelength had been chosen to avoid saturation of the detector, and that even at the low concentration of the AL formulation the results gave good repeatability.
- The retention time of the active ingredient is quite early; within 1-3 min. Mr Haustein replied

that this was intentional to give a fast method.

- It was further commented that not all labs will use the exact same column and may have problems with this early elution. Mr Haustein replied that some laboratories in the study had used a double length column and this gave a longer retention time. The results were still acceptable with the longer column.
- It would have been be useful if participants could have received the report before the meeting so they would have the chance to look at the data in detail. Mr Haustein apologised and replied that the report will be sent out soon.
- It was noted that you used an EC formulation in the collaborative study. There used to be a trifloxystrobin DC formulation. Did you reformulate? Mr Haustein replied that he didn't know but remarked that EC was now a common formulation type.
- Is it necessary to change the flow rate of the HPLC pump for the column-flush phase of the method? Mr Haustein replied that this was done to make the method quicker and that it is not mandatory to have the flow rate change.

The closed meeting discussed the comments received during the open meeting.
The meeting considered that the proposed sonication time of 15 minutes was too long and it was agreed that the description for the sample preparation should be amended to clarify that this time is only needed if there is an issue with dissolution of the samples.
The meeting also discussed whether the proposed injection volume of 3 µl was acceptable. It was noted that no remarks from the laboratories were presented to the open meeting so it was not clear if other laboratories had had issues with the smaller injection volume. From the response from the company it appears that some laboratories did use 5 µl. The meeting agreed that as the method states to use 3 µl this should remain but a footnote could be included to indicate that injection volumes up to 5 µl are also appropriate.
The meeting agreed that a clarification in the method about the short retention time would also be useful.

Decision The reversed phase HPLC method (CIPAC/4954) for the determination of trifloxystrobin in TC, EC, FS, SC, WG and AL formulations was accepted as a **provisional** CIPAC method with the proposal of amending the description of the method concerning sonication time and clarification of a possible inclusion of a note concerning injection volume.

CIPAC 59th meeting, June 2015 in Athens

At the 58th meeting, 2014 in Liège the method was accepted as provisional. No further comments were received.

Decision: The method can be promoted to a **full CIPAC method**.

CIPAC 65th meeting, June 2021 virtual

Determination of the relevant impurity CGA 344605 in trifloxystrobin formulations by Mr Friedhelm Schulz (5289, 5290): Mr Friedhelm Schulz presented the results of a small scale collaborative trial for the relevant impurity CGA 344605 in trifloxystrobin formulations with five participants from Germany. The method was based on HPLC (C18) using UV detection (210 nm) and external standard calibration.
A technical material, a water dispersible granule, a suspension concentrate and an emulsifiable concentrate were investigated. Furthermore, spiking experiments were performed with blank formulation.
Statistical evaluation was performed on five quality parameters: specificity and interferences, linearity, precision, accuracy (on two levels), and a limit of quantification (LOQ) was established. All criteria were easily met with RSDr based HorRat values below 0.5 for each laboratory and each formulation type. Detection limits ranged from 0.0026% (w/w) for the EC formulation to 0.040% (w/w) for the technical material.
Mr Schulz recommended the acceptance of the method.

There weren't comments received from the meeting.

Closed Meeting:
A small scale collaborative trial for the relevant impurity CGA 344605 in trifloxystrobin formulations was presented The reversed phase HPLC method for the determination of the relevant impurity CGA 344605 in trifloxistrobin TC, SC, EC and WG formulations was **noticed and adopted.**