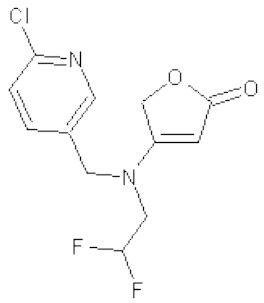
CIPAC STATUS REPORT

22/09/2018



0987 Flupyradifurone

Allocated to DAPA

CIPAC methods published in:

CIPAC

CIPAC 60th meeting, June 2016 in Tokyo

Flupyradifurone by Mr Michael Haustein (5049, 5050)
Mr Haustein presented the results of a small scale study carried out by DAPA with 6 laboratories on one TC and six different formulation samples (AL; EC; EW; FS; SL; WG) to demonstrate that the method is suitable for the determination of flupyradifurone in technical and in main formulation types. The homogenized sample containing flupyradifurone was dissolved in solvent mixture acetonitrile / purified water followed by active ingredient determination using gradient reversed phase high performance liquid chromatography, UV detection at 280 nm with an external standard calibration using a Phenomenex Kinetex C18, 50 mm x 4.6 mm, 2.6 µm particle size column.
The linearity of the method was checked in two concentration ranges 0.005-0.1mg/ml and 0.1-0.75 mg/ml. Both calibrations were linear. For all samples, the values of RSD_R were smaller than those calculated by Horwitz's equation. The proposed method was considered to be appropriate for the determination of flupyradifurone in technical and main formulation types.
The following comments were received from the meeting:

- Was it necessary to sonicate the samples for 15 minutes, this can warm up the samples? The answer was that in some cases shorter times may be sufficient.

Closed Meeting:
Proposed for full scale collaborative trial, with the note of clarifying before the start of the trial whether 15 min of sonication is needed, or can be reduced.

CIPAC 61th meeting, June 2017 in Rome

Mr Michael Haustein presented the results of a full-scale study carried out by 22 laboratories on a TC and six different formulation samples (AL; EC; EW; FS; SL; WG) to demonstrate that the method is suitable for the determination of flupyradifurone in technical and in main formulation types. The homogenized samples containing flupyradifurone were dissolved in a solvent mixture of acetonitrile/purified water, followed by active ingredient determination using gradient reversed phase high performance liquid chromatography, UV detection at 280 nm with an external standard calibration, using a Phenomenex Kinetex C18, 50 mm x 4.6 mm, 2.6 µm particle size column. 22 laboratories sent back the results, 13 participants used the column material described in the CIPAC trial, 8 laboratories used a different column type and data from one laboratory were not considered due to significant changes applied to the method.

In summary, it can be stated that the method deviations, noted by the 13 participants, who used the Kinetex C18 column, did not affect the analytical results significantly and therefore all data sets were included within the statistical assessment.
The EW formulation showed crystallization due to insufficient cold stability of the formulation. The crystallization was reported by several participants. As a result of the observed crystallization only eight results were used in the evaluation of the EW formulation.
In the first round the statistical evaluation was carried out on the data of 13 laboratories which used the column described in the CIPAC method.
In addition, the data of all the 21 laboratories participating in the CIPAC collaboration trial have been evaluated.
The data presented in the statistical summary showed that the method was suitable to gain acceptable and reproducible results for all samples tested and was therefore regarded to be robust.

The following comments were received from the meeting:

- It was asked whether sonication for 50 min was necessary and in which cases would be also shorter sonication time enough. The answer was that in case of FS and SE formulations it was necessary to sonicate for a longer time.
- One participant commented that the selection of 13 participants from the 21 (who used exactly the prescribed column during the trial) is not encouraged by CIPAC. Were there any specific reasons that the organisers excluded from the statistical calculation the other laboratories which did not use the Kinetex column?
- Mr Bura told that the use of the Kinetex column was a recommendation and in the final method it will be mentioned that similar columns can also be used.
- One participant asked if there is any explanation for the different behaviour of the WG formulation. The answer was that probably the insufficient extraction could cause the problem; however, no investigation was performed to detect its cause.

Closed Meeting:
A full scale collaborative trial was presented, the method was accepted as provisional.
(One remark was received: A note should be inserted in the method regarding the sample preparation of WG formulations)
The reversed phase HPLC method (CIPAC/5094) for the determination of flupyradifurone in TC, AL, EC, EW, FS, SL and WG formulations was accepted as a **provisional** CIPAC method with the need to insert a comment concerning the sample preparation for the WG formulation.

CIPAC 62nd meeting, June 2018 in Panama City

At the previous meeting, the method was accepted as a provisional CIPAC method. No further comments were received.
The method can be promoted to a full CIPAC method.
Decision: The reversed phase HPLC method (CIPAC/5094) for the determination of flupyradifurone in TC, AL, EC, EW, FS, SL and WG formulations was accepted as a **full** CIPAC method with the need to insert a comment concerning the sample preparation for the WG formulation.