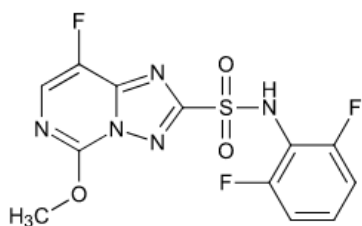


CIPAC STATUS REPORT

19/09/2021



0616 Florasulam

Allocated to....:

CIPAC methods published in:

CIPAC 63rd meeting, June 2019 in Braunschweig

Florasulam by Mr Eric Zhao (5205, 5206)

Mr Eric Zhao presented the results of a small scale CIPAC collaborative trial for the determination of florasulam in one TC; and in one SC formulation. The samples were extracted with acetonitrile followed by sonification. Quantification of the florasulam content was determined by reversed phase C18 HPLC, using a water+phosphoric acid/acetonitrile gradient, UV-detection (260 nm) and external standard calibration. The retention time of florasulam was approx. 6.4 min. The recoveries obtained from spiked samples to blank formulation ranged from 99.1-99.8% with typical RSDs below 0.6% (n=3 to 6).

Four laboratories participated in the trial out of which three used an identical reversed phase C18 HPLC column and one laboratory used a reversed phase C18 column from a different supplier, however all dimensions were identical.

The calculated Horrat values were 0.17 and 0.19 for the TC and SC respectively. As explanation of the low Horrat value was proposed that only a small number of laboratories participated in the small scale collaborative trial.

The organizers proposed to proceed with a large scale CIPAC collaborative trial.

The following comments were received from the meeting:

- Mrs Nováková asked whether the addition of a small amount of water, prior to the addition of acetonitrile was tried to enhance solubilisation of florasulam. Mr Zhao replied that this was not tried as there were no problems in the analysis of the samples.
- Mrs Vinke asked whether the specificity was tested against a blank formulation or against a solvent blank. Mr Zhao replied that the specificity was tested against a blank formulation, and this will be adjusted in the report.

Closed Meeting:

A small scale trial was presented and the method was proposed for a large scale collaborative trial.

Determination of 2,6-difluoroaniline in agricultural formulations containing florasulam by Mr Todd Kajdan (5211, 5212)

Mr Kajdan presented the results of a development study for the determination of 2,6-difluoroaniline (DFA) in florasulam formulations which in 2015 became a relevant impurity in the EU. Analysis by HPLC-UV or LC-MS proved to be insufficient sensitive or suffered from severe interference from formulation components. GC-MS analysis with SIM detection (m/z 129, 109, 101, and 82) was successful although difficulties were encountered with peak tailing, DFA

CIPAC STATUS REPORT

19/09/2021

degradation and memory effects. All these effects could be overcome by extracting the sample with water/1N NaOH/ethyl acetate and reducing the GC-inlet temperature to 150°C. The resulting method was tested with one EC, five SCs, five SEs, one OD, and 10 WGs. The overall average recovery of DFA was 104% with an RSD_r of 3.0% across a florasulam concentration range of 0.10-25.0% and in the presence of 10 other active ingredients.

The organizers of the study proposed to continue with a small scale collaborative trial.

The following comments were received from the meeting:

- Mr Kratzer asked whether hydrolysis was observed while treating the samples with water and 1 N NaOH. Mr Kajdan answered that this was investigated but not observed.
- Mr Wiese asked whether the extraction efficiency was investigated. Mr Kajdan answered that this was not investigated as the recovery experiments returned good results.

Closed Meeting:

CIPAC supports the intention to start a trial for the determination of the relevant impurity via the CIPAC platform. Mr Bura will agree with the organizers on an info sheet so that a small-scale collaborative trial can be performed.

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

The reversed phase HPLC method (CIPAC/5257) for the determination of florasulam in TC and SC formulations was accepted as a **provisional** CIPAC method, with some additional editorial amendments in method description, inclusion of method for the determination of suspensibility for SC formulation

CIPAC 65th meeting, June 2021 virtual

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**.