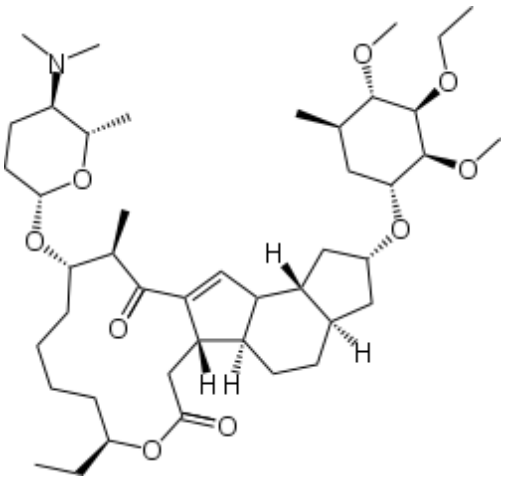
CIPAC STATUS REPORT

19/09/2021



0802 Spinetoram

Allocated to …:

CIPAC methods published in:

CIPAC 63rd meeting, June 2019 in Braunschweig

Spinetoram by Mr Kevin King (5189, 5190)
Mr Kevin King presented the results of a small scale CIPAC collaborative trial for the determination of spinetoram in two TCs; and two SC formulations. Both the TC and the SC are dispersed in water followed by extraction with methanol and filtration through a 0.45 µm filter (if necessary). Quantification was performed with reversed phase C8 HPLC using UV detection (250 nm) and external standardization resulting in an approx. retention time for spinetoram-J of 10-12 min. and 12-15 min. for spinetoram-L.
Eight laboratories (from Europe and the USA) participated and seven laboratories sent results back in time. However as one laboratory analyse the samples twice with different samples and different HPLC columns still eight results were obtained. Five out of eight sets of results were obtained with an identical HPLC column; two sets results were obtained with a comparable C8 HPLC column and one set of results was obtained with a C18 HPLC column. Both spinetoram forms were quantified separately.
Statistical evaluation of the data was performed following "Guidelines for CIPAC Collaborative Study Procedures for Assessment of Performance of Analytical Methods", and included Grubbs test for outliers and stragglers. Stragglers were identified for TC-1 (lab 3) and SC-1 (lab 3); outliers were identified for TC-2 (lab 3) and SC-2 (lab 3). As laboratory three was the only laboratory that consequently reported low results and used a reversed phase C18 HPLC column, the recommendation was made to only use a C8 column in the analysis of spinetoram TC and SC formulations. Including the outliers and stragglers the Horrat values were 1.0, 1.1, 0.6, and 1.3 for TC-1, TC-2, SC-1, and SC-2 respectively. After omitting of the outliers and stragglers the Horrat values were 1.0, 0.4, 0.6, and 0.3 for TC-1, TC-2, SC-1, and SC-2 respectively.
All deviations of the proposed method (except the use of a C18 HPLC column) were deemed to have no influence on the result of the analysis.

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

The following comments were received from the meeting:

- Mrs Nováková asked why both forms were independently quantified. Mr King answered that both forms have different specific coefficients and therefore have to be quantified separately.

- Mr Pigeon suggested to extend the scope to WG and DT formulations

Closed Meeting:
A small-scale trial was presented and the method was proposed for a **large scale collaborative trial** with the recommendation to include WG and DT formulations, but this is up to the company to decide.

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

The reversed phase HPLC method (CIPAC/5249) for the determination of spinetoram in TC, SC, WG, and DT formulations was accepted as a **provisional** CIPAC method pending on the amendments of the description of the method, inclusion of the identity tests, method for the determination of suspensibility for SC formulation, representative chromatograms.

CIPAC 65th meeting, June 2021 virtual
At the previous meeting, the method was accepted as provisional with some amendments, that were provided by the organizer. The method can be promoted to **full CIPAC method**.