CIPAC STATUS REPORT

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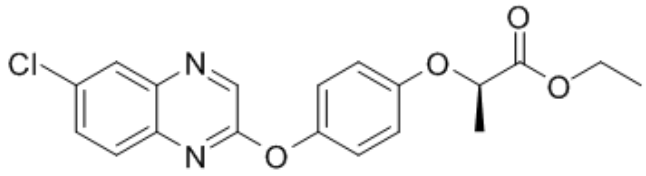

0641.202 Quizalofop-P-ethyl

Allocated to …:

CIPAC methods published in:

CIPAC 63rd meeting, June 2019 in Braunschweig

Quizalofop-P-ethyl by Mr Jinlong Huang (5197, 5198)
Mr Jinlong Huang presented the results of a small scale CIPAC collaborative trial for the determination of quizalofop-P-ethyl in two TCs; and three EC formulations.
Two methods were tested: a GC based method for quizalofop-ethyl determination and a HPLC method for quizalofop-P-ethyl determination. The identity of quizalofop-P-ethyl was confirmed by GC and HPLC retention time and infrared spectral identification.
The GC method was performed on a 15 m wide bore GC column (coated with dimethyl polysiloxane (or equivalent)), FID detection and di-n-octyl phthalate as internal standard. The retention time of quizalofop-ethyl was approx. 3.5 min. and the retention time of di-n-octyl phthalate was approx. 2.5 min. Samples for the GC method were diluted in internal standard solution (no composition mentioned) and injected without further sample preparation.
The HPLC method was performed on a normal phase, chiral HPLC column using UV detection at 237 nm. The mobile phase consisted of n-hexane/isopropanol (90/10 (v/v)) and the column temperature was 25°C. The retention time of quizalofop-P-ethyl was approx. 17.1 min. and the retention time of S-isomer was approx.18.7 min. Samples for the HPLC method were diluted in mobile phase and injected after filtration through a 0.45 µm filter if necessary.
Four laboratories (all China, three from the same company) participated in the trial. For GC analysis all participants used the same stationary phase ((5%-phenyl)-methyl polysiloxane). However, dimensions of the column varied in length (15-30 m), internal diameter (0.32-0.53 mm) and film thickness (0.25-1.5 µm). For HPLC analysis three out of four participants used the same CHIRALPAK AD-H column; however, one participant used the CHIRALPAK OJ-H column.
Statistical evaluation of the data was performed following "Guidelines for CIPAC Collaborative Study Procedures for Assessment of Performance of Analytical Methods", and included Cochrans' and Grubbs tests for outliers and stragglers. No outliers or stragglers were found, however only the results of the HPLC method for quizalofop-P-ethyl determination were reported. Horrat values were 0.16, 0.16, 0.42, 0.55, and 0.69 for TC-1, TC-2, EC-1, EC-2, and EC-3 respectively.

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

The following comments were received from the meeting:

- Mr Pigeon remarked that the use of a wide bore GC column was not common anymore and might lead to a small number of participants. Mr Jinlong Huang replied that the best results were obtained with a wide bore GC column.
- Mr Patrian remarked that also two capillary columns were used with good results so the method could be changed into prescribing a capillary column. Mr Jinlong Huang replied that no other columns can be used.
- Mr Patrian remarked that hexane should be replaced by heptane for toxicological reasons.

- Ms Santilio remarked that the GC temperature settings in two laboratories are identical for injector, GC-column, and detector and that this was rather unusual. Mr Jinlong Huang confirmed the remark but could not explain why the settings were as mentioned.
- Mr Kratzer related to the application of two methods where the HPLC method would suffice. Mr Jinlong Huang replied that this was done to gain more information.

Closed Meeting:
A small scale trial was presented and the method was proposed for a **large scale collaborative trial**.
Mrs Tessier remarked that not all questions were properly answered. Mr Hänel replied that a large-scale trial could be performed, however the questions need to be answered and Mr Bura and he would contact the organizers for clarification. Clarification is also needed on why a wide bore GC column is mandatory, as to labs used a capillary column and the results were acceptable; is it possible to replace hexane by heptane for safety reasons? Before running the full scale trial, it should be clarified if the chiral method also quantifies the substance, because in this case there is no need for the GC method.

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

The chiral phase HPLC method (CIPAC/5255) for the determination of quizalofop-P-ethyl in TC and EC formulations was accepted as a **provisional** CIPAC method, with additional modifications in the description of the method considering the selectivity of the column (using the right eluent) and inclusion of a second selective identity test.

CIPAC 65th meeting, June 2020 virtual

A second identity test was required after last year meeting as the IR spectrum doesn't distinguish between the R and S isomer. The company proposes the identity test by both HPLC with chiral column and IR. HPLC can be used to separate the R and S isomers, the retention time of the R isomer is about 15.1 min and the retention time of the S isomer is 16.2 min. The retention time of the test substance is the same as that of the R isomer, which confirms that it is quizalofop-Pethyl. The chemical structure of quizalofop-ethyl can be confirmed by measuring IR, the latter method. It was proposed that this method is accept as full method after editorial modification as follows:
Use the HPLC method below. After confirming that the R and S isomers can be completely separated, the relative retention time of quizalofop-P-ethyl in the sample solution should not deviate by more than 1.5% from that of calibration solution.
The chiral phase HPLC method can be promoted to **full CIPAC method**, with additional modifications in the description of the method concerning the identity test.