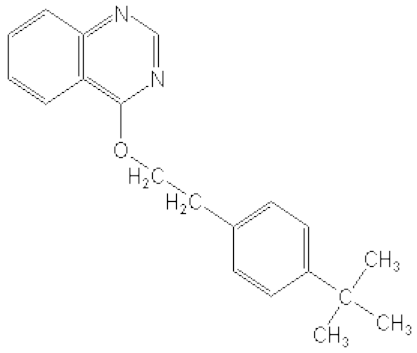
CIPAC STATUS REPORT

18/09/2017



0693 Fenazaquin

Allocated to ESPAC

CIPAC methods published in:

Not published

CIPAC 58th meeting, June 2014 in Liège

Fenazaquin by Mr Rene Cochran (4975, 4976)

No information was received or presented at the meeting.

CIPAC 59th meeting, June 2015 in Athens

Mr Cochran presented (5006, 5007) the results of a **small scale** trial on the determination of fenazaquin TC and SC formulations using HPLC-UV with a reversed phase C8 column, UV-detection at 260 nm, and external standard calibration. Mr Cochran also presented the results of a **small scale** trial on the determination of a fenazaquin EC formulation using HPLC-UV with a normal phase silica column, UV-detection at 260 nm, and external standard calibration.
One TC sample, one SC sample, one EC sample, one SC formulation blank, one EC formulation blank, and one fenazaquin reference standard were investigated by two laboratories.
One laboratory mentioned that the retention time of the major component was sensitive to the quality of iso-octane used in the mobile phase. Both laboratories mentioned sampling difficulties with the SC formulation due to static electricity build-up during sampling.
The statistical evaluation was carried out according to ISO 5725 and showed no Grubb's or Cochran's outliers or stragglers.
When all the data were included the Horwitz criteria for RSD_R were met. Mr Cochran proposed to skip the small scale collaborative trial and to go directly to a full CIPAC trial.
The following comments were received from the meeting:

- Why were two methods proposed as the EC formulation can also be analysed with reversed phase chromatography? Mr Cochran replied that this was caused by the nature of the solvent in the EC formulation, not compatible with the acetonitrile/water system.
- It was asked -it the 2 ml/min flow rate could be diminished. The answer was that this is possible if in the same time the eluent ratio is changed. However this would imply different chromatographic profile with implications in the registration processes. Both methods were used for registration purposes and the company preferred to keep the original analytical methods.
- The lack of retention time stability was of concern for the meeting. Small differences in water content might be the cause of large retention time deviations.

It was suggested to perform a small scale trial to identify any problems, to solve the retention time problem. It was suggested to contact ESPAC if the decision is to conduct a small scale trial.

Closed meeting:

The meeting discussed the comments received during the open meeting.
The need for the normal phase HPLC method was regarded questionable, as it proved to result in non-reproducible retention times. Both the technical and the EC and SC products should preferably be analysed with one method, preferably using the reversed phase HPLC column. Therefore a **small scale trial** was strongly recommended. This advice will be forwarded through ESPAC (Mr Garvey).

CIPAC 60th meeting, June 2016 in Tokyo

Fenazaquin by Mr Rene Cochran (5036, 5037)
Mr Cochran presented the results of a full scale trial on the determination of fenazaquin in TC and SC formulations using HPLC-UV with a reversed phase column, UV-detection at 260 nm, and external standard calibration. The measurements were carried out in duplicate weighings and two injections on two different days. Two TC samples and two SC samples were investigated by the 14 participating laboratories; the results were evaluated according to ISO 5725 guidelines.
According to Cochran's and Dixon's tests, among the received results various stragglers and outliers were observed.
If no data were excluded (elimination of stragglers and outliers by Dixon's and Cochran's tests) and all the results provided by each one of the fourteen participants was used for the statistical evaluation, the Horwitz criterion was satisfied in all four cases: Fenazaquin TC1, Fenazaquin TC2, Fenazaquin 200 SC1 and Magister 200 SC2.
Mr. Cochran considered that the proposed methods can be considered to be suitable for the fenazaquin determination in technical and suspension concentrate and proposed the method to be accepted as a provisional CIPAC method.
The following comments were received from the meeting:

- There were some comments on sending the collaborative trial results, the participants did not receive the report before the CIPAC meeting (Luis, and others)
- As a general remark one participant draw the attention of the laboratories on the importance of the right analytical view regarding the interpretation and spread of the results. In case of TC-2 one laboratory obtained 1003.7g/kg on day1 and for the same sample 964.4 g/kg on day2. In such cases it is highly recommended to check the measurement for possible errors.

Closed meeting:
The method was accepted as **provisional CIPAC method**.

CIPAC 61th meeting, June 2017 in Rome

The method was provisional. It was promoted to full CIPAC method.
The reversed phase HPLC method using external standardization (CIPAC/5036) for the determination of fenazaquin in TC and SC formulations was accepted as a **full** CIPAC method