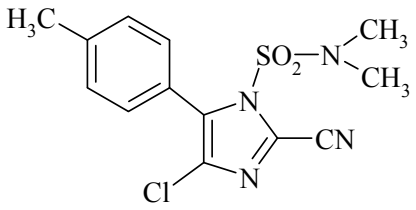
CIPAC STATUS REPORT

28/12/2013



0653 Cyazofamid

Allocated to ESPAC

CIPAC methods published in:

CIPAC

CIPAC 55th meeting, June 2011 in Beijing

Mr Jim Garvey presented the results of a **small scale** collaborative study, on the determination of cyazofamid in technical product (TC) and suspension concentrate (SC) using HPLC-UV detection at 280 nm and external standard calibration. The trial was organised by ESPAC in conjunction with ISK Belgium. 5 laboratories participated in the study. 2 samples of TC and 3 samples of SC were provided.
3 participating laboratories commented that the run time of 60 minutes was too long. Laboratory 3 commented that in all chromatograms there was an interference at the beginning of cyazofamid peak.
The statistical evaluation was carried out according to the CIPAC guidelines.
For TC 1, TC 2 and SC 2 Lab 4 was a Cochran's outlier. For SC1 and SC3 Lab 1 was a Cochran's outlier. No data were excluded from the initial evaluation.
TC 1, TC 2, SC 1 and SC 2 meet the Horowitz criteria when all the data are included. SC 3 did not meet the Horowitz criteria when all the data is included. When the results from Lab 1 (Cochran's outlier) were omitted and the statistical evaluation was repeated the Horwitz criteria were met in all cases.
ESPAC consider that the proposed method is appropriate for the determination of cyazofamid in TC and SC and that a full scale trial can be conducted.

The following comments were received from the meeting:

- Why is the acetic acid necessary in the mobile phase? Mr Garvey replied he believed it was there to adjust the pH.
- Methanol and acetonitrile are both used in the mobile phase. It would be preferable to use a mobile phase with only one organic solvent.
- Lab 3 reported interferences at the beginning of the peak. Was this noted by any other labs? What column did Lab 3 use and was it equivalent? Mr Garvey replied that no other labs reported this interference and that the interference was very small.
- Reducing the column length could shorten the run rime
- It would be preferred to have a fixed operating temperature for the HPLC analysis as ambient temperature can vary from country to country.

Decision: The meeting considered that provided the comments received were taken on board CIPAC could recommend moving to a full scale trial.
A full scale trial is recommended.

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CIPAC 56th meeting, June 2012 in Dublin

Mr Joris presented the results of a **full scale** collaborative study **(4833, 4834)** on the determination of cyazofamid in technical product (TC) and suspension concentrate (SC) formulations using HPLC-UV, detection at 280 nm and external standard calibration. Two samples of TC and three samples of SC were provided. 15 laboratories participated however only results for 14 labs were presented for the TC due to problems with sample shipment to 1 lab.
The temperature of the HPLC analysis had been adapted to 40°C following the small scale trial in order to reduce the run time from 60 minutes to 45 minutes; however 3 labs commented that the run time was still too long.
3 labs commented that a longer sonication time was needed to dissolve some samples fully.
No data were excluded from the initial evaluation. Will all the data included all samples meet the Horwitz criteria. Some outliers were identified for the SC samples however the Horwitz criteria were met with all data included.
Mr Joris concluded that the proposed method is appropriate for the determination of cyazofamid in TC and SC and proposed that the method be adopted by CIPAC as a provisional method.

The following comments were received from the meeting:

- The run time of the HPLC analysis is long. It is not so common to have such long runs these days. Looking at the chromatograms it seems that there is nothing eluting after 30mins so perhaps it not necessary to have a run for 45 min? Mr Joris replied that this was true but the company believes that the long runtime was needed as some impurities in the TC can be eluted at about 40 min.
- It may still be possible to change the HPLC conditions to shorten the run time.
- It was noted that the identity of the labs was given in the report. Please kindly note that this is not standard practice.

The meeting discussed the comments received.
The meeting considered the issue of the long run time. The analysis used an isocratic run so it was suggested that a flush gradient could be added towards the end of the run to speed up the run time. This may be more time consuming as you would have to re-equilibrate the system after gradient but the company should consider this option.
The meeting agreed that CIPAC could not cut the length of the run time if, as was suggested, some impurities could elute later in the run. It was also agreed that the length of the run is not a good enough reason to reject the acceptability of a method. The meeting agreed that the company should be asked to add a footnote suggesting it may be a possible to include a flush gradient in the run.

Decision:

The reversed phase HPLC method (CIPAC/4833) for the determination of cyazofamid in TC and SC formulations was accepted as a **provisional** CIPAC method with the amendments in the description of the method concerning the gradient flush after the elution of the a.i.

CIPAC 57th meeting, June 2013 in Kyiv

At the 56th meeting, 2012 in Ireland the method was adopted as provisional. No further comments were received
The method can be promoted to a **full CIPAC method**.

Decision:
The reversed phase HPLC method (CIPAC/4826) for the determination of chlorfenapyr in TC and SC formulations was accepted as a **full** CIPAC method with the need to define the column temperature.