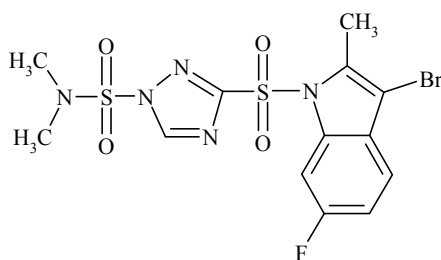


CIPAC STATUS REPORT

02/03/2015



0789 Amisulbrom

Allocated to J

CIPAC methods published in:

Not published

CIPAC 56th meeting, June 2012 in Dublin

Mr Yokouchi presented the results of a **small scale** collaborative study (**4839, 4840**) on the determination of amisulbrom in technical product (TC), water dispersible granules (WG) and suspension concentrate (SC) formulations using HPLC-UV, detection at 254nm and external standard calibration. The trial was organised by JAPAC. Two samples of TC, one sample of WG and two samples of SC were provided. Three laboratories participated.

One laboratory commented that further ultrasonication was needed for WG and that both the analytical standard and the TC were affected by static during weighing.

The statistical evaluation was carried out according to the CIPAC guidelines. No stragglers or outliers were identified during the statistical analysis of the data.

The “pure” between lab standard variation was not calculated for two formulations.

JAPAC consider that the proposed method is appropriate for the determination of amisulbrom in TC, WG and SC and that a full scale trial can be conducted.

No comments were received from the meeting.

A full scale trial is recommended.

CIPAC 57th meeting, June 2013 in Kyiv

Mr Takahashi presented the results of a **full scale** collaborative study on the determination of amisulbrom in technical product (TC), water dispersible granule (WG) and suspension concentrate (SC) formulations using HPLC-UV, detection at 254 nm and external standard calibration. The trial was organised by JAPAC. Two samples of TC, one sample of WG and two samples of SC were provided. 22 laboratories offered to participate and results and data were received from 20.

Mr Takahashi remarked that a YMC Pack column is the same as an ODS column but is supplied by a Japanese company. Various types of different ODS column were used by the laboratories, and some slightly changed the mobile phase composition. Some laboratories also used shorter columns – but even when these were used no interference from other peaks was noted for the TC and WG and the retention time for amisulbrom fell within the acceptable range. For the SC there was a larger interfering peak observed but it was well separated from the amisulbrom peak. It could be concluded therefore that there were no problems with using a shorter column.

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One laboratory remarked that TC-1 and TC-2 were analysed on Day 3 because of low repeatability on Day 2.

Three laboratories reduced the amount of sample weighed out but maintained the same concentration levels as describe in the method.

The statistical evaluation was carried out according to the CIPAC guidelines.

For TC 1 Labs 6, 7, 8, 10 and 17 were Cochran's outliers

For TC 2 Labs 7, 8 and 10 were Cochran's outliers and Lab 12 was identified as a Grubb's outlier

For WG 1 Lab 12 was identified as a Cochran's outlier

For SC 1 Lab 13 was identified as a Cochran's straggler

For SC 2 Lab 10 was identified as a Cochran's straggler

No data were excluded from the initial evaluation. When all the data were included the Horwitz criteria were met in all cases.

Mr Takahashi concluded that JAPAC propose the method is appropriate for the determination of amisulbrom in TC, WG and SC and that it is adopted as a provisional CIPAC method.

The following comments were received from the meeting:

- How important it is to use a YMC column? Could you describe what is meant by YMC column or equivalent? It would be desirable to have some sort of check for the user to do so that they can determine if their column is equivalent. Mr Takahashi replied that the YMC column is manufactured by a Japanese company. But the column is of a type (C18) that is used worldwide so he believes there are equivalent columns available.
- In the presentation you mentioned possible interferences as shown in some example chromatograms and also provided information on the sum of total interferences as a % of the active ingredient peak. Perhaps this type of chromatogram and information could be used to indicate what labs should look for to see if their column is equivalent
- 9 out of 20 labs have used the recommended column and 11 used variations. One of the labs that participated in the trial commented that they used a Zorbax C18 150 mm column with no problems at all. It should be possible to determine an equivalent column from all those used by the labs.

The meeting discussed the comments received during the open meeting. Written remarks from Mr Martijn were also received.

10 different types of column were used – the meeting considered how far a laboratory can deviate from the method that is proposed. It was agreed that to such problems in the future the procedure should be written in a way that makes it clear when deviations are necessary/acceptable.

The meeting noted that even when there were 10 types of column used, the results were acceptable. This demonstrates the ruggedness of the method.

The meeting agreed the method could be **adopted as provisional**

Decision: The reversed phase HPLC method (CIPAC/4883) for the determination of amisulbrom in TC, WG and SC formulations was accepted as a **provisional** CIPAC method.

CIPAC 58th meeting, June 2014 in Liège

No further comments were received.

Decision: The reversed phase HPLC method (CIPAC/4883) for the determination of amisulbrom in TC, WG and SC formulations was accepted as **full** CIPAC method.