CIPAC STATUS REPORT

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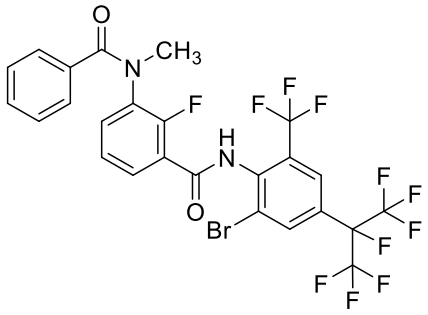

0994 Broflanilide

Allocated to JAPAC

CIPAC methods published in:

CIPAC P

CIPAC 62nd meeting, June 2018 in Panama City

Mr Takeo Okochi presented the results of a small scale collaborative trial on the determination of the active ingredient broflanilide in two technical materials and three wettable powders. Broflanilide was determined by reversed phase HPLC using UV detection at 254 nm and external standardization. Elution was performed with acetonitrile-water (65-35 (v/v)) on an XSelect CSH C18, 250 x 4.6 mm (i.d.), 5 µm reversed phase column with a flow rate of 1.0 ml/min. The retention time of broflanilide was approx. 11.5 min.
Three Japanese laboratories participated in the trial and reported results. Laboratory 3 reported technical problems with the analysis on day 2. However as no scientific reasons could be identified the data of day 2 were accepted and statistically evaluated.
After statistical evaluation one Cochran's outlier was identified for technical material 2 (lab 3). However as the reproducibility relative standard deviation (RSDR) of all five samples was well within the calculated Horwitz values (even including the Cochran's outlier) all results were accepted.
The organizers recommended that the broflanilide method should progress to a full scale collaborative study.

The following comments were received from the meeting:

- Mr Ramesh asked whether dissolution was complete or whether precipitation occurred. Mr Okochi answered that the duplicate results showed excellent agreement this was considered to be not relevant.
- A second question related to the large difference in flow rate between the labs. Mr Okochi answered that no influence of the differences in flow rate on the results was expected and this was done to adjust the RT to approx. 5 min.

Closed Meeting:
The small scale trial presented was proposed for a full scale collaborative study with the conditions of the proposed method (as the participating labs used different conditions).

CIPAC 63rd meeting, June 2019 in Braunschweig

Broflanilide by Mr Takeo Okochi (5213, 5214)
Mr Takeo Okochi presented the results of a large scale CIPAC collaborative trial for the determination of broflanilide in two TCs; and three WP formulations. The method consisted of a dilution (in case of

the TC) or extraction (in case of the WP) with HPLC mobile phase (acetonitrile/water, 65/35 (v/v)) followed by sonication and broflanilide determination by reversed phase HPLC using UV detection at 254 nm and external standardization. The retention time for broflanilide is approx. 11.5 min.
23 Laboratories (Europe, the Americas, and Asia) participated in the trial and reported results. Three laboratories used an identical column as described in the method. The other participants used reversed phase C18 HPLC columns with the same dimensions and particle sizes but from different suppliers. One laboratory (lab 21) used a reversed phase C18 UPLC column. The participants reported several remarks mainly associated with changes in the flow-rate or injection volume. The calculated results were subjected to the Cochrans' and Grubbs test. In TC-1 four Cochrans' outliers (lab 5, 10, 14, and 21) and two Grubbs outliers (20 and 21) were identified. In TC-2 three Cochrans' outliers (lab 10, 14, and 21), one Grubbs straggler (lab 21) and two Grubbs outliers (4 and 20) were identified. In WP-1 three Cochrans' outliers (lab 10, 14, and 20) and one Grubbs outliers (20) were identified. In WP-2 one Cochrans' outliers (lab 10) and one Grubbs outliers (20) were identified. And finally, in WP-3 one Cochrans' outlier (20) was identified. However, all data were retained as no obvious reasons existed for removing the outliers and stragglers. Including all stragglers and outliers the Horrat values were 0.85, 0.94, 0.68, 0.74, and 0.78 for TC-1, TC-2, WP-1, WP-2, and WP-3 respectively.

The organizers proposed that the method would be accepted as a provisional CIPAC method.

The following comments were received from the meeting:

- Mr Benke commented that this collaborative trial was an excellent example of a well-organized large scale CIPAC collaborative trial.

Closed Meeting:
A large scale trial was presented and the method can be promoted to **a provisional CIPAC method**.

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

The reversed phase HPLC method (CIPAC/5213) for the determination of broflanilide in TC and WP formulations was accepted as a **full** CIPAC method.

CIPAC 68th meeting, June 2024 Wageningen

Broflanilide by Ms Laetitia Leroy (5388, 5389)

Ms Laetitia Leroy presented (on behalf of Carlos Moncada) a method extension of the CIPAC method for broflanilide TC to a UL formulation (994/TC/M/3) in which two laboratories participated. The broflanilide content was determined by reversed phase HPLC (Waters XSelect CSH C18 (or eq.), 250 × 4.6 mm, 5.0 µm) at 40°C with UV detection at 254 nm and external standardization. The eluent was acetonitrile : water, 65:35 (v/v) at a flow rate of 1.0 ml/min. During the performance of the method it appeared that dilution of the sample with eluent resulted in a bi-phase system which resulted in inadequate quantification. Changing the dilution solvent from eluent to neat acetonitrile solved this problem and quantitation was possible. Changing the dilution solvent was assessed to be a minor change as the retention time of broflanilide did not deviate by more than 2% from that of the calibration solution. The results of the two laboratories were combined and resulted in a HorRat value of 0.5.

Ms Laetitia Leroy considered the result fully acceptable and proposed acceptance of the extension.
Questions and remarks from the meeting.

No comments were given or questions were asked by the meeting.

Closed Meeting:

No comments were given or questions were asked by the meeting. The method was accepted as **provisional** method.