CIPAC STATUS REPORT

30/01/2008

$$H_{3}C - (CH_{2}) = H_{11} - N - C - NH_{2} + H_{3}C - C - O^{-1}$$

0101 Dodine

Allocated to AOAC

CIPAC methods published in :

CIPAC 1B, p. 1802 (titr.)

CIPAC 10th meeting, June 1966 in France

Mr. Schechter said that Mr. Caswell had appointed Mr. Pasarela of The American Cyanamid Co. as a referee. Dr. Henriet said he would like to collaborate in this work, while Mr. Schechter said that it could usefully be a joint study between CPAC and AOAC.

CIPAC 11th meeting, June 1967 in London

Mr. Schechter stated that Mr. Pasarela would be organizing coll. work on behalf of AOAC. Dr. Henriet said he would like to take part. Prof. Zeumer observed that dodine caused offflavours in strawberries. Dr. Ashworth said that there was a British Committee on taint in a variety of crops, and he thought members might like to see their report. Members agreed.

CIPAC 12th meeting, June 1968 in Braunschweig

Dr. Henriet said that he would like to take part in the collaborative work organized by AOAC.

CIPAC 14th meeting, June 1970 in Gembloux

Decision The nonaqueous titration method is adopted as AOACCIPAC method.

CIPAC 49th meeting, June 2005 in Utrecht

Mr J.L. Lamproye presented a small scale study for the modification of the CIPAC 101 method for the determination of dodine, on behalf of Chimac Agriphar as the existing titration method had an overestimation of the a.s. The a.i. is determined by ion-pair chromatography on an ODS2 column, using 0.005M heptane sulphonic acid at pH 3.5 (H₃PO₄) and UV detection. Three labs participated in the study and were asked to analyse one technical sample, one WP and one FL sample. Blank formulations were also provided. The method gave acceptable reproducibility and repeatability values for the technical material and the formulations analysed. No interfering peaks were detected on the chromatograms. It was proposed to proceed to a full scale study.

CIPAC 51th meeting, June 2007 in Umhlanga Rocks, South Africa

Mr Pigeon presented a full scale study on behalf of Agriphar, where dodine is determined with ion pair HPLC with UV detection at 200 nm using heptanesulfonate as counter ion on an Inertsil 5 ODS 2 column.

One TC, one SC, a plain formulation, WP 65, SL,

12 labs participated. All participants had RSD_R above Horwitz for TC, WP and with elimination some above, some below.

The discussion was about incompleteness and shortcomings in the method, like pH adjustment in

the mobile phase already containing acetonitrile, lack of obvious reasons to eliminate participants. <u>Decision</u> The reversed phase ion-pair HPLC method (CIPAC/4564) for the determination of dodine in TC and SL formulations has to remain a draft method until further considerations and/or modifications.