CIPAC STATUS REPORT

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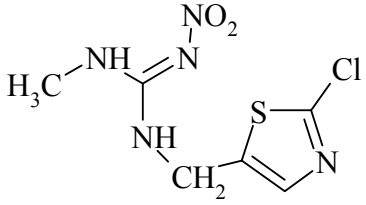

0738 Clothianidin

Allocated to J

CIPAC methods published in:

CIPAC

CIPAC 52nd meeting, June 2008 in Braunschweig

Mr Yasushi Asada presented the results of a small-scale collaborative study by JAPAC on the determination of clothianidin in two TC and three WG formulations, using RP HPLC with UV detection at 269 nm and external standardisation. Seven data sets were obtained from seven participants. Summary and detailed statistical evaluations were shown. The statistical evaluations were carried out according to ISO 5725. The discussion on stragglers and outliers was as follows: WG-1 - The variance of Lab. 6 was identified as an outlier. The data were retained because there were no reasons to remove them. WG-3 - The variance of Lab. 3 was identified as an outlier. The data were retained because there were no reasons to remove them. For all samples, the values of RSD_R (reproducibility relative standard deviation) were smaller than those calculated by Horwitz's equation.
The proposed method is considered appropriate for the determination of clothianidin in technical product and water dispersible granule. JAPAC proposed to proceed to a large-scale collaborative trial.
Decision The reversed phase HPLC method (CIPAC/4604) for the determination of clothianidin in TC and WG formulations was recommended for a full scale trial.

CIPAC 53rd meeting, June 2009 in Sonsonate/El Salvador

1. Mr Yasushi Asada presented the results of a full-scale collaborative study on the determination of clothianidin in technical product (TC), water dispersible granule (WG), suspension concentrate (SC), granule (GR), and water soluble granule (SG), using RP HPLC analysis with UV detection at 269 nm and external standardization. The results of all 19 participating laboratories have been taken into account for the statistical evaluation. For all samples, the values of RSD_R (reproducibility relative standard deviation) were smaller than those calculated by Horwitz's equation. The proposed method is considered appropriate for the determination of clothianidin in the TC, WG, SC, GR, and SG. JAPAC proposes that the method be accepted as a provisional CIPAC method. In the method, it is specified "Zorbax Eclipse XDB-C18 (5 µm), or equivalent", clarification was requested on the columns considered equivalent. Mr Asada's answer was that according to the column manufacturer this is a common C18 column. It was also suggested and accepted that the injection volume should be changed to 5 µl and to modify the sample preparation for the SC and SG formulations by adding water before acetonitrile is added.
2. Mr Martin Feyerabend presented the results of the extension of the scope of a method collaboratively tested to the determination of clothianidin in FS and WS formulations. The only change proposed was in the sample preparation, addition of 5 ml water prior to addition of acetonitrile. The inject volume was also reduced.

Decision

1. The reversed phase HPLC method (CIPAC/4658) for the determination of clothianidin in TC, WG, SC, GR and SG formulations was accepted as a **provisional** CIPAC method, subject to changing the injection volume to 5 µl, to modify the sample preparation in the case of SC and SG formulations by adding water and introducing a footnote to the column drawing attention to the shape of the peak.
2. A method extension to a method just presented which got the provisional status at the meeting cannot be accepted. It needs to be discussed at the next meeting.

CIPAC 55th meeting, June 2011 in Beijing

The method extension was presented at the 53rd meeting, 2009 in El Salvador. It could not be accepted as a method extension for technical reasons as the method itself was also presented at the same meeting. At the 54th meeting, 2010 in Slovenia, the method was accepted as provisional method.
Mr Martjin raised the issue in writing before the meeting that for suspensibility (where the a.i. content should be determined) the sample preparation is different to what was proposed for the method and method extensions. The meeting considered that this was not a major issue.
Decision: The extension of the scope of the HPLC method (CIPAC/4658) for the determination of clothianidin in FS and WS formulations (CIPAC/4692) was accepted as a **full** CIPAC method.

CIPAC 57th meeting, June 2013 in Kyiv

Mr Bascou informed the meeting that they had note for the clothianidin (CIPAC 738) methods, in the method for suspensibility there is a paragraph describes the usual suspensibility test for SCs when in fact clothianidin is really an FS. He proposed that a correction is needed to reflect this and offered to provide a proposal to the secretary in writing

Mr Bura remarked that comments and corrections with CIPAC methods are always welcomed and that errata can be made on the web site. He asked the meeting if there were any other comments on existing methods.

CIPAC 60th meeting, June 2016 in Tokyo

Clothianidin by Mr Michael Haustein (5051, 5052)
Mr Haustein presented the results of a study carried out for extension of CIPAC method 738 to WP formulations by two independent laboratories on five batches of a WP formulation.
Measurement of each individual batch was carried out at two different days in two different laboratories, double determination and double injection each. For the analysis a Zorbax Eclipse XDB-C18, 150 mm x 4.6 mm, 5 µm particle size column was used, a mobile phase of with acetonitrile/ water/phosphoric acid, 150/850/1 (% v/v/v) as eluent, with a flow rate of 1 ml/min. The injection volume was 5 µl and the detector wavelength 269 nm.
Based on the relative standard deviation results (RSDr) obtained for the five individual WP batches, the CIPAC method was proposed suitable for the extension to WP formulation types. The repeatability results (ranging from 0.5 % to 0.7 % relative) were below the modified Horwitz criterion and were comparable to those of the original full scale trial for a WG formulation with equal a.i. content.

➢ There were no questions or comments to the presentation

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 extension of the scope (CIPAC/5051) of CIPAC method 738/WG/M/ for the determination of the clothianidin content of WP formulations was accepted as a **full** CIPAC method