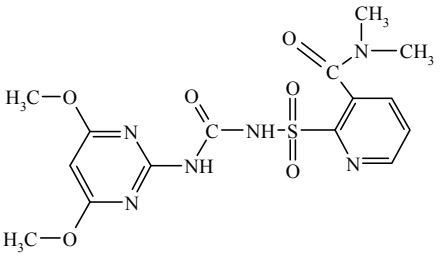
CIPAC STATUS REPORT

02/03/2015



0709 Nicosulfuron

Allocated to USA

CIPAC methods published in:

CIPAC 49th meeting, June 2005 in Utrecht

Mr P. Bloxham presented a CIPAC collaborative study for Nicosulfuron. One TC and 4 WG samples were distributed to 16 labs; results were received from all but one of the labs. The samples were analysed by reversed phase HPLC with UV detection and 3-methyl-1,1 diphenylurea as internal standard. It was proposed to accept the method as a provisional CIPAC method. One of the participating labs noted the elution of a peak after the method recommended stop time of 5min (at 7min). The small injection volume as well as the low pH of the mobile phase were questioned; it was pointed out that a pH lower than 3 was necessary for the stabilisation of the retention time of the a.i. It was suggested to explain this as a footnote in the method and to provide the information on a citrate buffer system for pH-calibration.
Decision The reversed phase HPLC method (CIPAC/4443) for the determination of nicosulfuron in TC and WG formulations was accepted as **provisional** method with the addition of footnotes that the use of internal standard is necessary due to the small injection volume and that the calibration of the pH electrode should be carried out as stated in the method.

CIPAC 50th meeting, June 2006 in Geneva.

Decision The reversed phase HPLC method (CIPAC/4443) for the determination of nicosulfuron in TC and WG formulations was accepted as **full** method with the addition of footnotes that the use of internal standard is necessary due to the small injection volume and that the calibration of the pH electrode should be carried out as stated in the method.

CIPAC 50th meeting, June 2013 in Kyiv

Mr Chen presented the results of a validation study for the **extension** of the CIPAC method 709/ TC/M/3 for determination of nicosulfuron in oil dispersion (OD) formulations. The existing CIPAC method 709/TC/M/3 is suitable and validated for the determination of nicosulfuron in TC and water dispersible granules (WG)
Validation data in accordance the CIPAC guideline for a method extension were presented. The method extension met these criteria.
Mr Chen concluded that the method is appropriate and proposed that the method extension be adopted by CIPAC.
The following comments were received from the meeting:

- Can you tell me what sample preparation was used, as this will be different to that given in the CIPAC method for the solid preparation? Mr Chen replied that the sample preparation involved dissolving the formulation in acetonitrile.
- Were there any issues with the compatibility of the oil in the OD and the mobile phase of the method? Were there any issue with or limits to the solubility of the formulation? Mr Chen replied that the acetonitrile mixed well with the mineral oil.

➢ Do you have draft of the method extension for the OD? Mr Chen replied that this will be drafted and provided shortly.

➢ There will need to be an identity test included in the draft in accordance with the CIPAC style. Did you do any statistical analysis without the internal standard? Is the internal standard necessary? Mr Chen replied that they used an internal standard as the purpose of the study was to validate and an extension of an existing CIPAC method and the original method contains the internal standard.

The meeting discussed the comments received during the open meeting.
The meeting discussed further the choice of solvent used for sample preparation. The miscibility of acetonitrile with the OD formulation was questioned, as it was mineral oil based. It was noted that some OD formulations are vegetable oil based and miscibility of acetonitrile vegetable oil is better than for mineral oils. The meeting agreed further clarification is needed. Based on the experience of one laboratory THF could be used as an alternative. The meeting considered that a bringing study comparing MeCN and THF might be useful.
The meeting also agreed that there may be a need to change the suspensibility method for nicosulfuron.
Information on the additional identity test and a description of the method need to be provided.
The meeting agreed that pending the comparison of extraction solvents and provision of the additional information the extension can be adopted **as provisional**.

Decision:
The extension of the scope (CIPAC/4903) of CIPAC method 709/TC/M/3 for the determination of the nicosulfuron content of oil-based suspension concentrate formulations (OD) was accepted as a **provisional** CIPAC method.

CIPAC 58th meeting, June 2014 in Liège

No further comments were received.

Decision: The extension of the scope (CIPAC/4903) of CIPAC method 709/TC/M/3 for the determination of the nicosulfuron content of oil-based suspension concentrate formulations (OD) was accepted as **full** CIPAC method.