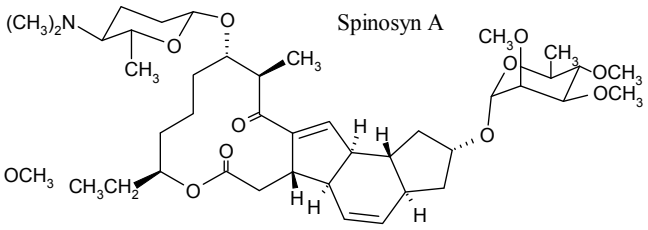
CIPAC STATUS REPORT

10/12/2013



0636 Spinosad

Allocated to UK

CIPAC methods published in:

CIPAC L, p. 121

CIPAC 48th meeting, June 2004 in Brno

Mr. Ghaoui presented the results of a of a small scale study by PAC-UK on three technical materials, and three SC formulations using reversed phase HPLC, C18-column, UV detection at 250 nm and external standardisation. Five laboratories participated in the study. One of the laboratories had submitted results that deviate from the results from the other laboratories. If one of the laboratories is excluded all RSD_R were smaller than those calculated by Horwitz equation, which was not at all the case if none of the outliers or strugglers was excluded. PAC-UK proposed to proceed to a full collaborative trial, but will include an internal standard and increase the sample size. Ms. Houdarkis asked if it was necessary to use the column specified in the method. The reply was that other columns probably could be used. A blank formulation was also provided to give the possibility to check for interferences. Mr. Hill suggested to add to the method that equivalent columns with the same selectivity could be used. Mr. Müller informed that price and toxicity should be considered when choosing the internal standard.

CIPAC 49th meeting, June 2005 in Utrecht

Mr D. Heim presented a full scale collaborative study for the determination of Spinosad A and B in technical samples, SC and GR formulations using a reversed phase HPLC method. Twelve participating laboratories were asked to analyse two TC, three SC, and three GR samples and formulation blanks. An impurity co-eluting with the internal standard made that the evaluation was done by external calibration. The evaluation of results was thus carried out only for the external standardisation method which gave acceptable results and was proposed to be accepted as a provisional CIPAC method. The identity test is based on a LC/MS method which separates the spinosyns A and B. More detailed information was requested when quoting this technique as the method for identification. One of the participants suggested the inclusion of blank samples in such studies but it was pointed out that no blank samples are normally available to official control laboratories and that this is not a general recommendation. It was noted that Horwitz criterion is taken as a guideline and that a result not meeting this criterion should not automatically be excluded.
Decision The reversed phase HPLC method (CIPAC/4456) for the determination of spinosad (spinosyn A and D, respectively) in TC, SC and GR formulations using external standardization was accepted as **provisional** method. The LC/MS identity test, with the necessary corrections in the wording, was accepted.

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CIPAC 50th meeting, June 2006 in Geneva

Mr. D. Heim presented the report of a method extension for the determination of spinosad in DT formulations. Mr Müller asked if the tablet is a hard one and if if it was difficult to grind up in a mortar and pestle. The response was that it was not a problem. Mr Hill questioned the accuracy of the data generated as he considered that the data did not indicate it. Mr Heim said that recovery tests were made in the company lab.

Decision The reversed phase HPLC method (CIPAC/4456), published in CIPAC L, p.121, for the determination of spinosad (spinosyn A and D, respectively) in TC, SC and GR formulations using external standardization was accepted as **full** method.

The extension of the scope of CIPAC method 636 (CIPAC/4511) to DT formulations was accepted as **provisional** CIPAC method.

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

Decision The extension of the scope of CIPAC method 636 (CIPAC/4511) to DT formulations was accepted as **full** CIPAC method.

CIPAC 54th meeting, June 2010 in Ljubljana

Mr David Heim presented the results of an extension of the existing CIPAC method for the determination of spinosad in EC formulation.
The concentration of the EC was outside the acceptable range. The sample preparation followed that already said for the SC, except sample weight adjusted to cover the EC concentration range. Samples should be mixed thoroughly before sampling
Comments:
Generally ECs have mineral oil which may not be compatible with the solvent system used in the existing CIPAC method. However in this instance this does not apply and it would be worth adding a footnote to the method to explain that it is compatible with the solvent systems used.
The proposed method extension was considered appropriate for the determination of spinosad in EC and proposed to be accepted as provisional.
Decision The extension of the scope of CIPAC method 636/TC/M/3 for the determination of the content of spinosad in EC formulations was accepted as a **provisional** CIPAC method, with a footnote that mineral oil is not causing clogging of the column.

CIPAC 55th meeting, June 2011 in Beijing

The EC is for public health use. At the 54th meeting, 2010 in Slovenia the method was adopted as provisional with a requirement to include a footnote that mineral oil is not causing clogging of the column. This has been addressed. No further comments were received.
Decision The extension of the scope of CIPAC method 636/TC/M/3 for the determination of the content of spinosad in EC formulations (CIPAC/4721) was accepted as a **full** CIPAC method.

CIPAC 56th meeting, June 2012 in Dublin

Mr Heim presented the results of a validation study **(4847, 4848)** for the **extension** of the scope of CIPAC method 636/GR/(M)/- for determination of spinosad in GR. The study was organised with ESPAC. The existing CIPAC method 636 is suitable and validated for the determination of spinosad in TC, SC, GR and DT; however the existing GR method does not cover a new higher concentration granule that is now available. 5 laboratories participated. Each laboratory was provided with 5 different batches of the formation and 1 blank formulation.
One laboratory commented that sample vessels larger than 100ml were needed in order to ensure efficient shaking for extraction.
Validation data in accordance the CIPAC guideline for a method extension were presented. No stragglers or outliers were identified, however the Horwitz criteria were met for only two of the 5 SC samples.
Mr Heim reminded the meeting that for the full scale collaborative trial conducted on the other GR sample in 2007 the Horwitz criteria were met for only 1 of 3 batches. RSD values found in this study are comparable with those found in the original 2007 study.
Mr Heim proposed that the method is extended.
No comments were received from the meeting.

Decision:

The extension of the scope (CIPAC/4847) of CIPAC method 636/GR/(M) for the determination of the spinosad content of a new granule formulation (GR) was accepted as a **provisional** CIPAC method.

CIPAC 57th meeting, June 2013 in Kyiv

At the 56th meeting, 2012 in Ireland the method was adopted as provisional. No further comments were received.
The method can be promoted to a full CIPAC method.

Decision:

The extension of the scope (CIPAC/4847) of CIPAC method 636/GR/(M) for the determination of the spinosad content of a new granule formulation (GR) was accepted as a **full** CIPAC method.