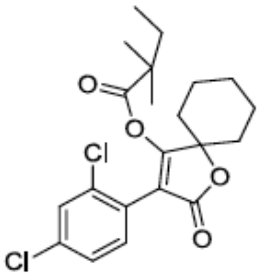
CIPAC STATUS REPORT

16/09/2020



0737 Spirodiclofen

Allocated to

CIPAC methods published in:

CIPAC P

CIPAC 62nd meeting, June 2018 in Panama City

Spirodiclofen by Ms Lyu Cong (5148, 5149)
Ms Lyu Cong presented the results of a small scale collaborative study for spirodiclofen in two technical samples (TC-1 and 2), three suspension concentrates (SC-1, 2, and 3), and four participants.
The method consisted of methanol extraction and reversed phase high performance liquid chromatography (C18, 250 mm x 4.6 mm; flow rate 1.0 ml/min), using UV detection at 260 nm and external standardization. The reported individual analytical conditions were assessed as "basically the same".
Statistical evaluation of the data was accomplished following the "Guidelines for CIPAC Collaborative Study Procedures for Assessment of Performance of Analytical Methods", according to DIN ISO 5725. In SC-1 one Grubb's outlier was detected, but in the remaining formulations no outliers were established. Without elimination of the outlier all results complied fully to the Horwitz criteria.
The organizers recommended that the spirodiclofen method should progress to a full scale collaborative study.

No comments were received from the meeting.

Closed Meeting:
A small scale trial was presented and the method was proposed for a **full scale collaborative study**.

CIPAC 63rd meeting, June 2019 in Braunschweig

Spirodiclofen by Mr Jason Zhang (5195, 5196)
Mr Jason Zhang presented the results of a large scale CIPAC collaborative trial for the determination of spirodiclofen in two TCs; and three SC formulations. Samples were sonicated with methanol and the spirodiclofen content was determined by reversed phase C18 HPLC using UV detection (260nm) and external standardization.
18 Laboratories (Europe, USA, and Asia) participated in the trial. Nearly all laboratories used a reversed phase C18 column with dimensions 250x4.6 mm, and 5µm particle size (as described in the method). However, lab 4 used a UPLC column with dimensions 100x2.1 mm, and 1.7 µm particle size, and lab 16 used a deviating mobile phase.
Statistical evaluation of the data was performed and included Grubbs test for outliers and stragglers. Stragglers were not identified but three outliers were encountered for TC-A (lab 4), SC-

C (lab 16), and SC-D (lab 16). Including all outliers the Horrat values were 0.47, 0.50, 0.84, 0.82, and 0.90 for TC-A, TC-B, SC-C, SC-D, and SC-E respectively. Without the outliers the results were even better.

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

The following comments were received from the meeting:

- Mr Perez Albela Vera remarked that the use of UPLC in case of lab. 4 explains the difference in results.

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/5195) for the determination of spirodiclofen in TC and SC formulations was accepted as a **full** CIPAC method.