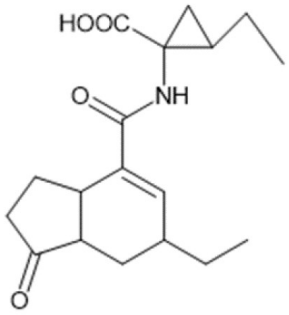
CIPAC STATUS REPORT

10.08.2024



XXXX Coronatine

Allocated to CHIPAC

CIPAC methods published in: CIPAC Handbook -

CIPAC

CIPAC 67th meeting, June 2023 Braunschweig

Coronatine by Ms Xu Rong (5339, 5340)

Ms Rong presented the results of a small scale collaborative study for the determination of the coronatine content in two TC materials and three SL formulations by C18 HPLC with UV detection at 220 nm and quantification by external standardization. The extraction was performed with methanol followed by a final dilution step with HPLC eluent (acetonitrile: 0.1% phosphoric acid in water 30:70 (v/v)). Four laboratories participated, all laboratories reported in time and no method deviations were reported. The statistical evaluation was performed according to DIN ISO 5725 and the 'Guidelines for CIPAC Collaborative Study Procedure for Assessment of the Performance of Analytical Methods'. Testing for outliers/stragglers was not performed. The HorRat values ranged from 0.04-0.3 indicating compliance to the test criteria.

Ms Rong considered the method to be suitable validated and recommended continuation with full scale collaborative trial.

The following comments were received from the meeting:

- Ms Tessier suggested to change the sample amount from 19 to 20 mg.
- Ms Nováková asked why different injection volumes were applied. Ms Rong responded by explaining that the concentration of coronatine in SL formulations is very low, there is a need to inject more.
- Mr Pigeon asked whether all isomers were included. Ms Rong replied by explaining that the substance was obtained after fermentation and that as a result only one isomer is produced and was investigated.

Closed meeting: Mr De Rijk remarked that no explanation was presented related to the HorRat values below 0.3.
Mr Hänel replied that this is only requested when performing a full scale trial.
No further comments were received, the method was recommended to a **full scale** CIPAC trial.

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CIPAC 68th meeting, June 2024 Wageningen

Coronatine by Ms Yue Wang (5382, 5383)

Ms Yue Wang presented the results of a full scale collaborative trial of coronatine in two TCs and three SL formulations by 18 participants which returned 18 sets of results. The analysis was performed by high performance liquid chromatography on a reversed phase column (C18, no brand indicated, 250 × 4.6 mm, 5.0 µm) at 35°C with UV detection at 220 nm and external standardization. The eluent was acetonitrile : aqueous phosphoric acid, 30:70 (v/v) at a flow rate of 1.0 ml/min. Seventeen of the participating laboratories used a variety of HPLC columns with comparable reversed phases and one laboratory used a comparable reversed phase however with different dimensions. None of the deviations were assessed to be critical. 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. With the SL1 formulation two Grubbs' stragglers were identified, no further Grubbs' stragglers or outliers were encountered. HorRat values, calculated without elimination of the stragglers of 0.3-0.4 were identified for all samples.
Ms Yue Wang finalised her presentation by stating that CHIPAC considered the method for the analysis of coronatine in TC and SL formulation to be suitable validated and recommended to become provisional.

Questions and remarks from the meeting.

- The data of SL-1 and SL-2 seemed to be identical. Please check.
    - Probably a mistake was made. Data will be double checked.
- The target concentration for the SL formulations is missing. Please adjust the report.
    - In this case the sample preparation is different, as there was no dilution of the sample. The description of the method will be updated.
- Reversed phase chromatography was applied whereas coronatine theoretically has multiple isomers. Normal phase chromatography would be method of choice for distinguishing between the isomers.
    - Only one isomer is present in the coronatine material so normal phase chromatography was not needed. This will be clarified in the text of the method.

Closed meeting:

Some remarks were given by the meeting.

- The datasets of SL1 and SL2 are identical and a further explanation/clarification by the presenter/company has to be given.

Conclusion: pending the clarification of the datasets of SL1 and SL2 the method can be accepted as **provisional** method.