CIPAC

COLLABORATIVE INTERNATIONAL PESTICIDES ANALYTICAL COUNCIL LIMITED

Commission Internationale des Méthodes d'Analyse des Pesticides (CIMAP)

Minutes of the 66th Annual meeting

The 66th meeting was held on Tuesday 14th and Wednesday 15th June 2022 using on-line communication tools.

Those attending

- Items 1 to 7 on 14th June: members, correspondents, observers and expert witnesses.
- Items 8 to 12 on 15th June: members, correspondents and observers (representatives of industry and commercial laboratories, by special invitation only)

1. Welcome and introductory remarks

The chair, Mr R. Hänel, opened the 66th CIPAC meeting, and welcomed all the participants.

2. Apologies

Apologies were received from: Mr L. Benke, Mr V. Chmil, Mrs E. Jacobsen, Mrs A. Kashouli-Kouppari, Mr V. Mikhailov and Mrs C. Breedt.

3. Adoption of the agenda

Item 4.11 will not be discussed （SYN549888 by Mr Christian Mink)

4. Reports of expert witnesses on collaborative trials

4.1 14-hydroxylated brassinosteroid by Ms Yue Wang (5311, 5312)

Ms Wang presented the results of a full scale collaborative trial for the determination of 14-hydroxylated brassinosteroid, by reversed phase HPLC and 222 nm UV-detection after phenylboronic acid derivatization. Quantitation was performed by external standardization. Two TKs and three SL formulations were sent to 18 laboratories of which 17 returned their results in time. The remarks of the participants were discussed however they were regarded as minor. The data were evaluated according to “Guidelines for CIPAC collaborative study procedures for assessment of performance of analytical methods”, and were subjected to Mandel’s k-statistics on the within-lab variance, followed by Mandel’s h-statistics on the lab means. Including the data of all participants the HorRat values ranged from 1.37-2.12. However, after elimination of two-three stragglers and outliers per sample the HorRat values were in the range of 0.66-0.99.

Ms Wang recommended this method to be accepted as a provisional CIPAC method.

The following comments were received from the meeting:

- Ms Vinke remarked that an outlier in table 1 might have been overlooked. This was not the case.

- Mr Garvey asked whether the relative high evaporation temperature of 90°C might have influenced the high number of stragglers and outliers. Ms Yue Wang responded that this was not to be expected as the derivatization was tested across the range of 70-90°C and was proven to be complete.

4.2 Difenoconazole by Mr Christian Mink (5324, 5325)

Mr Christian Mink presented the results of a full scale collaborative trial for the determination of difenoconazole by GC-FID on a DB-5 or equivalent phase with hydrogen as carrier gas, and internal standard quantitation in two TCs, one WG and two EC formulations. 23 laboratories participated and 21 have returned their results in time. Seven laboratories used H_2, 11 used He, four used N_2 and two laboratories changed the stationary phase. Including the data of all participants the HorRat values ranged from 0.5-1.4, whereas after elimination of two outliers/stragglers the HorRat values ranged from 0.5-1.0. A breakdown of the data set to evaluate whether the use of different carrier gasses was of influence to the results showed that when using H_2 or He the HorRat values were acceptable, even without removing outliers or stragglers suggesting that He could be added as an alternative carrier gas to H_2.

Mr Mink also investigated whether the proposed method could be used to investigate the isomeric ratio between the *cis* and *trans* isomers of difenoconazole in two TC samples. It was concluded that after elimination of one outlier the data were consistent with HorRat values ranging from 0.4-1.0 (n=14) indicating that the method was fit for purpose.

Mr Mink considered the method to be suitably validated and recommended this method to be accepted as a provisional CIPAC method.

The following comments were received from the meeting:

- Mr Hänel remarked that the aim of CIPAC trials is to validate an analytical method and not to show that the laboratory can analyse that active substance.
- Mr Bura noticed that there are two data sets, one using H as carrier gas and one using He. If the method is strictly followed, there are only 7 valid results, however there are 11 acceptable results using He as carrier gas. If the two data sets are considered together, the method description should allow the use of both gases.
- Mrs Nováková requested why no report was sent to the participants before the meeting. Mr Mink apologized, it was due to time constraints. Furthermore, she requested why a total run time of 44 minutes was given as the compounds of interest eluted before 20 minutes and no further peaks can be seen after 25 minutes. Mr Mink replied that an impurity elutes at around 45 min, in consequence, if the runtime is shortened, interference may appear with the impurity.
- Mr Pigeon asked whether a difference in efficacy was related to both isomers. This was not known.
- Mrs Tessier asked whether both isomers were truly baseline separated as it could not be assessed based on the presented chromatograms. This was confirmed, the isomer separation is needed, because of specifications in some countries.
- Mr Garvey asked whether the deviation of Lab 7 could be assigned to a deviating method. This was not the case as only He was used as carrier gas and no further method deviations were reported.

4.3 Ethephon by Ms Yue Wang (5315, 5316)

Ms Yue Wang presented the results of a full scale collaborative trial for the determination of ethephon in two TCs, two TKs and two SL formulations. The method used consisted of HPLC based ion-chromatography using sodium carbonate and sodium hydrogen carbonate as eluent and

electrolytic conductivity detection. 14 laboratories participated and all 14 laboratories reported in time. Laboratory 14 reported the use of a method which deviated too much and therefore the results of laboratory 14 were omitted. The dataset was subjected to Grubb's straggler and outliers tests and the results of laboratory 13 in two TC samples were eliminated. The resulting HorRat values ranged from 0.38-0.77.

Ms Wang recommended this method to be accepted as a provisional CIPAC method.

No comments of the meeting were received.

4.4 Flumioxazin by Ms Li (5330, 5331)

Ms Li presented a method extension of CIPAC method 578 to the determination of flumioxazin in WG and SC formulations. Flumioxazin is determined by reversed phase high-performance liquid chromatography using UV detection at 288 nm and external standardization. The validity of the method was proven by a specificity test and a concentration range check. Both the specificity test and linearity range fulfilled the criteria and it was concluded that CIPAC method 578 was suited for the determination of flumioxazin in WG and SC formulations.

Ms Li proposed to accept the method extension to WG and SC formulations.

No comments of the meeting were received.

4.5 Isocycloseram by Mr Christian Mink (5326, 5327)

Mr Mink presented the results of a small scale collaborative study for the determination of isocycloseram in three TC and two WP formulations by reversed phase high performance liquid chromatography using UV detection at 265 nm and external standardization. The sample set was sent to four participants whereas the organizer added two independent data sets. The participating laboratories reported minor deviations which were assessed as not of influence to the results. The resulting HorRat values ranged from 0.2-0.5

Mr Mink recommended to go forward to a full scale CIPAC collaborative trial.

No comments of the meeting were received.

4.6 Matrine by Ms Yue Wang (5313, 5314)

Ms Wang presented the results of a full scale collaborative trial for the determination of matrine in two TKs and three SL formulations. The method used consisted of reversed phase HPLC with UV detection at 215 nm, and external standardization. 18 laboratories participated and 17 laboratories reported their results in time. Deviations were reported by all laboratories, however, the deviations were considered minor. When including all data, the HorRat values were between 0.52-0.93 for four out of five samples with the exception of TK-2 for which a HorRat value of 3.05 was calculated. After elimination of Mandel's h and k statistic stragglers/outliers the HorRat values for all investigated samples ranged from 0.42-0.98.

Ms Wang recommended this method to be accepted as a provisional CIPAC method.

The following comments were received from the meeting:

- Mr Garvey asked whether the removal of three outliers could be linked to certain method deviations. This was not the case.
- Ms Vinke asked whether the results of laboratory 17 could have been influenced by the use of a different HPLC column temperature (35°C instead of 30°C). This was assessed as not significant. Furthermore, Ms Vinke suggested that differences in storage temperature might have been of influence.

4.7 Metolachlor by Ms Nikita Li (5317, 5318)

Ms Li presented the results of a small scale collaborative study for the determination of metolachlor in three TCs, three EC and three EW formulations using gas chromatography on a HP-5 or equivalent capillary column with flame ionization detection, and dipentyl phthalate as internal standard. Three laboratories participated and reported results in time. HorRat values ranging from 0.23 to 0.52 were calculated using all available data sets. Grubb's outlier tests were performed and no outliers or stragglers were identified.

Ms Li recommended to go forward to a full scale CIPAC collaborative trial.

The following comments were received from the meeting:

- Mr Bura remarked that the internal standard showed fronting and recommended to investigate whether another internal standard might lead to a more symmetrical peak. Mr Garvey added that the analyte also showed significant fronting.
- Mr Pigeon asked why the column temperature of 182°C was so precise. It was chosen because it was the best option resulting the most reliable results and better separation.
- Ms Vinke recommended that the total run time should be added to the description of the method.

4.8 Methoprene by Ms Junhua Song (5305, 5306)

Ms Song presented a method extension of CIPAC method 414 to the determination of methoprene in GR, GR-SB and CS formulations. CIPAC method 414 describes a method based on capillary gas chromatography using flame ionisation detection and internal standardization for TC material. Minor changes were needed to make CIPAC method 414 suited for the GR, GR-SB and CS formulations. For the extension of the method a specificity test and a quantification/repeatability test were required. The specificity test was performed by comparing the GC mass spectra of methoprene with authentic standards. The quantification/repeatability tests were performed by two laboratories on two days and resulted in RSD% of 1.0-1.6%. No HorRat values were calculated.

Ms Junhua Song proposed to accept the method extension of CIPAC 414 to GR, GR-SB and CS formulations.

The following comments were received from the meeting:

- Mr Pigeon remarked that the specificity test was an identity test and not a specificity test.

4.9 Pirimiphos-methyl by Mr Molingzhi (5301, 5302)

Mr Molingzhi presented a method extension of CIPAC method 239 to the determination of pirimiphos-methyl in LN formulations. CIPAC 239 is based on gas chromatography, a DB-1 or equivalent stationary phase and flame ionization detection, internal standard quantitation and was developed for TC. In the method adjusted the sample amount, the GC temperature program, and the time in the ultrasonic bath were adjusted resulting in the necessity to perform a repeatability test. Two laboratories participated by analyzing five LN formulations on two different days. HorRat values ranging from 0.30-0.64 were obtained without removing data from the data sets.

Mr Molingzhi proposed to accept the method extension of CIPAC 239 to LN formulations.

No comments of the meeting were received.

4.10 S-methoprene by Ms Junhua Song (5307, 5308, 5309, 5310)

Ms Song presented the results of a small scale collaborative study for the determination of free (non-encapsulated) methoprene in three CS formulation by GC-FID on a capillary chemically bonded and cross-linked dimethylpolysiloxane stationary phase with internal standardization. Four

laboratories were invited and all reported results in time. The extraction method consisted of an extraction step with *n*-hexane and a rotary evaporator as rolling apparatus. Each sample was analyzed in quadruplicate on two different days and no major method deviations were registered apart from laboratory four which was excluded from the dataset. After statistical evaluation of the data following the "Guidelines for CIPAC Collaborative Study Procedures for Assessment of Performance of Analytical Methods", the HorRat values ranged from 2.8-6.4. This was attributed to the dynamic equilibrium between the inside and outside of the capsule, and the uncertainties during the extraction process. However, considering the fact that the purpose of the method is to monitor the content of free S-methoprene in CS formulations, as indicated in the proposed WHO specification "not greater than 5% of the total AI content", it is believed that the method can fulfil its function.

Ms Song proposed to go forward to a full scale CIPAC collaborative trial.

The following comments were received from the meeting:

- Ms McNally asked whether there were any proposals for change from the participants or they just sent the results. The answer was that the participants didn't send proposals, they used the method as it was requested. The only comment was the amount of water needed for dispersion.
- Mr Pigeon remarked that the use of the rotary evaporator might be the cause of the deviations. He also asked whether other types of equipment were tested as replacement of the rotary evaporator. This was not the case. A complication when using a rotary evaporator is that solvent can evaporate. However, as the internal standard is added before using the rotary evaporator no influence on the quantitation procedure is expected. Ms Song answered that the rotary evaporator is a more common equipment then a roller and that there wasn't much evaporation during the extraction.
- Mr Pigeon asked whether the proposed method could also be applied for determination of the release rate. This was not tested.

Ms Song presented also the results of a small scale collaborative study for the determination of the ratio of *R*- and *S*-methoprene in three TC samples by normal phase using a Chiralpak AD-H silica HPLC column with UV-detection at 254 nm. Four laboratories were invited and 3 reported results in time. The samples were dissolved in *n*-hexane and analyzed in quadruplicate on two different days. There weren't major method deviations registered apart from laboratory four which was excluded from the dataset. After statistical evaluation of the data following the "Guidelines for CIPAC Collaborative Study Procedures for Assessment of Performance of Analytical Methods", the HorRat values ranged from 0.15-0.16.

Ms Junhua Song recommended to go forward to a full scale CIPAC collaborative trial.

The following comments were received from the meeting:

- Mr Pigeon asked whether the method could be applied to formulated products. This was not tested and as the column is rather expensive this will not be proposed.
- Mr Pigeon asked if there is the intention also to develop a method for release rate. Ms Song will check with the company.

4.11 SYN549888 by Mr Christian Mink (5328, 5329) was not discussed

4.12 Trifluralin by Ms Junhua Song (5303, 5304)

Ms Song presented the results of a full scale collaborative trial for the determination of trifluralin in two TCs and three EC formulations by reverse phase high performance liquid chromatography using UV detection at 280 nm and external standardization. 22 Laboratories participated and 20 of them returned results in time, whereas some of them reported minor deviations. After statistical

evaluation including all data following the "Guidelines for CIPAC Collaborative Study Procedures for Assessment of Performance of Analytical Methods", the HorRat values ranged from 0.56 to 0.99. After removal of up to four outliers the HorRat values ranged from 0.44 to 0.51.

Ms Song recommended this method to be accepted as a provisional CIPAC method.

The following comments were received from the meeting:

- Mr Garvey asked (and Mr Bura seconded) why so many outliers had to be removed especially as the dataset without removing of the outliers was already acceptable. Mr Hänel remarked that it has already been discussed and agreed that if the validation criteria are met, outliers/stragglers are not removed.

4.13 Deltamethrin + chlorfenapyr by Ms Marie Baes (5297, 5298)

Ms Baes presented the results of a full scale collaborative trial for the determination of deltamethrin in two TCs, chlorfenapyr in two TCs and deltamethrin + chlorfenapyr in two LN formulations by HPLC UV on a CN chromatographic column, UV detection at 230 nm and external quantification. TC analysis were performed in duplicate whereas LN analysis was performed in triplicate. 17 Laboratories participated and 12 returned results in time and the reported method deviations were assessed to be not relevant for the results. The resulting HorRat values ranged from 0.43 to 2.34. After removal of the Cochran outliers the HorRat values ranged from 0.42 to 1.12. The somewhat larger variation was due to the inhomogeneity of the LN samples and not related to the analytical method.

Ms Marie Baes recommended this method to be accepted as a provisional CIPAC method.

The following comments were received from the meeting:

- Ms Vinke asked whether the method was tested for complete extraction. This was the case but it was remarked that the extraction of coated nets was much easier
- Ms Vinke asked whether the outlier laboratories had changes in the method. The answer was that no changes in the method were seen, however the calibration was very different in the two days.

4.14 Bifenthrin, pyriproxyfen and PBO by Ms Marie Baes (5299, 5300)

Ms Marie Baes presented the results of a small scale collaborative study with five participants for the determination of bifenthrin, pyriproxyfen and piperonyl butoxide (PBO) in individual TC materials and LN samples after sonification in heptane for 60 min at 80° by GC-FID on a DB-210 capillary column and internal standardization. The LN materials consisted of incorporated fibres, hence the long extraction time at an elevated temperature. The extraction time was tested and 60 min was the optimum extraction time. The resulting HorRat values were acceptable when the group of five samples was split in subgroups of three and two samples. This is a clear indication of inhomogeneous samples which is not uncommon with LN formulations.

Ms Marie Baes recommended to go forward to a full scale CIPAC collaborative trial.

The following comments were received from the meeting:

- Mr Shahabuddin asked why the extension of individual methods are discouraged. Ms Baes answered that to test each molecule with different extraction process is more expensive. Mr Pigeon mentioned that there are CPAC methods for the 3 individual actives, but not for the LNs. One could have method extensions, but this would mean 3 different methods for the same product, doing 3 extensions. The best way for such net, containing 3 active ingredients, is to develop a new method with a single extraction and injection. This is more convenient for QC laboratories.

4.15 Attrition resistance of tablets by Mr Oliver Gutsche (5321, 5322)

Mr Gutsche presented the results of a small scale collaborative study with five participants for the determination of attrition resistance in EG, SG, WG, DT, ST, WT and GR (<1 cm) formulations with the aim to replace CIPAC MT 178.1 and 178.2 by a new, combined method MT 178.3. Adding glass beads to DT, ST, WT and GR (<1 cm) formulations improved the method as friction and resulting surface attrition were higher.

Mr Gutsche recommended that this method will supersede CIPAC MT 178.1 and 178.2.

The following comments were received from the meeting:

- Mr Wenzel asked whether pharmaceutical methods were investigated. This was done in the preparation of the new method, however those methods are not suitable for these types of products.

4.16 Discharge rate of aerosol dispensers by Ms Claudia Vinke (5153, 5320)

Ms Vinke presented the results of a full scale collaborative trial for the determination of the discharge rate of two types of aerosol dispensers with 10 participants. One dispenser was contained compressed air and an inner bag whereas the other dispenser was an aerosol can with a propane/butane/isobutane mixture as propellant. The discharge rate of the aerosol dispenser was determined by measuring the quantity of the material expelled through the valve for 10 s until empty. The resulting discharge rate diminished from 1.98-1.25% until approximately 11% material was remaining and was consistent with all participants when applying the method to the compressed air dispenser. When applying the method to the aerosol can two distinct sets of data were obtained based on whether the laboratory used the option of adding an additional spray tube to the nozzle. The discharge rates in those were lower due to the back pressure caused by the narrow tubing. However, the results were in line with each other until approximately 6% of the material was left in the can. For the laboratories not using the additional tubing the results were in in line with each other until approximately 20% material remained.

Ms Vinke recommended this method to be accepted as a provisional CIPAC method.

The following comments were received from the meeting:

- Mr Hänel asked whether the use of the tubing was required on the label. It was free of choice and therefore also not requested from the participating laboratories.

4.17 Discharge rate of trigger sprayer by Ms Claudia Vinke (5152, 5319)

Ms Vinke presented the results of a full scale collaborative trial for the determination of the discharge rate of two different types of trigger dispensers with 10 participants. One trigger dispenser was equipped with a press button whereas the other trigger dispenser was equipped with a hand trigger. The test was performed by pushing (or triggering) ten times and weighing the can before and after the described motions, and to repeat this three times with each trigger dispenser. For the push button dispenser eight laboratories determined discharge rates between 0.20 to 0.22 g/stroke and two laboratories determined discharge rates of approximately 0.16 g/stroke. For the hand trigger dispenser ten laboratories determined discharge rates between 1.09 to 1.25 g/stroke (after a repeated trial by laboratory five).

Ms Vinke recommended this method to be accepted as a provisional CIPAC method.

The following comments were received from the meeting:

- Ms Tessier remarked that both products were very different from each other and that the press button trigger dispenser was very similar to an aerosol dispenser.

5. Reports of expert witnesses on other matters

5.1 MT 160 Spontaneity of dispersion of suspension concentrates by Mr Christoph Czerwenka (5323)

Mr Czerwenka presented a procedure which aligned MT 160 with MT 184.1, resulting in MT 160.1 which should supersede MT 160. The method involves preparing a suspension of the formulation in standard water. After standing under defined conditions the top 9/10ths are drawn off and the remaining 1/10th is assayed. The spontaneity of dispersion is calculated as the percentage of sample remaining suspended. The remaining material can be assayed chemically or gravimetrically. The solvent extraction step in MT 160 was removed and the addition of a U-shaped metal tube was added to the method in order to simplify the workload.

Mr Czerwenka recommended this method to be accepted as a provisional CIPAC method.

No comments of the meeting were received.

6. Revision/update of CIPAC guidelines

6.1 Validation of analytical methods (draft guidance) by Ms Angela Santilio (5259)

Ms Santilio presented a draft proposal of the revision of the CIPAC document on how to validate the analytical methods for the determination of active ingredient in plant protection products by GC and HPLC. The proposal is addressed to the control laboratories. The draft proposal is based on the comments of CIPAC members, SANCO 3030/99/rev. 5, appendix F of the AOAC official methods of analysis, 2012, and on the CIPAC Guidelines on method validation to be performed in support of analytical methods for agrochemical formulations. Requested parameters are specificity, linearity, accuracy, and precision. The parameter recovery is difficult to determine as laboratories normally do not access to blank formulations. However, it was suggested that recovery could be assessed by comparing the experimental concentration with the nominal concentration of the active substance.

Ms Santilio then suggested that the proposed document was open for discussion.

The following comments were received from the meeting:

- Mr Haustein remarked that it was not clear asked whether in the case of linearity reference is made to r or r^2. Should be r.
- Mr Czerwenka commented that recovery calculations based on the nominal concentration of the active substance cannot be trusted as it is based on company information. Furthermore, he asked whether a linearity value of $r \geq 0.98$ is acceptable and he suggested that it should be at least ≥ 0.99.
- Mr Wolfram remarked that this document was aimed at official control laboratories and therefore was of no interest to CIPAC. Mr Hänel remarked that this will be discussed in the closed meeting.
- Mr Pigeon remarked that the proposed recovery procedure was not acceptable. Ms Santilio then suggested to skip the parameter recovery from the proposal.
- Mr Bura remarked that the aim of the revision was for CIPAC only and not aimed at official control laboratories.
- Mr Garvey remarked that the proposed document could be helpful in the discussions official control laboratories might have with the local boards of accreditation.

7. Replacement of obsolete methods, comments to existing methods, errata

Mr Bura reported that no remarks have been received.

8. Minutes of the 65th meeting (5294/P)

The minutes were circulated to the participants by e-mail. There weren't comments received, as a

conclusion the minutes were accepted as a true record of the last year meeting.

9. Secretary's report (5295/P)

Mr Bura presented the Secretary's report. The report was previously circulated to members by e-mail. No comments were received. It was accepted.

10. Discussion of individual compounds

1006 28-homobrassinolide

At the previous meeting, the method was accepted as provisional. No further comments were received. The clarifications requested last year were submitted and considered acceptable. The method can be promoted to a **full CIPAC method**.

221 chlorpyrifos

At the previous meeting, the method was accepted as provisional. No further comments were received. The method can be promoted to a **full CIPAC method**.

133 ametryn

At the previous meeting, the method was accepted as provisional. No further comments were received. The method can be promoted to a **full CIPAC method**.

494 tebuconazole

At the previous meeting, the method was accepted as provisional. No further comments were received. The method can be promoted to a **full CIPAC method**.

Active substances discussed this year:

4.1 14-Hydroxylated brassinosteroid

Mr Garvey questioned whether the extensive use of removing outliers was necessary. Mr Hänel replied that this is allowed and agreed upon when the HorRat values are not met.

The method for the determination of 14-hydroxylated brassinosteroid in TK and SL formulations can be promoted to a **provisional CIPAC method** with additional justification for the Horrat >1 and for the eliminations.

4.2 Difenoconazole

Mrs Tessier requested whether baseline separation was achieved. Mr Patrian, seconded by Mr Pigeon, added that this was nearly achieved so the separation was sufficient.

Mr Bura requested what kind of dataset was accepted as several carrier gases have been applied. The data from 15 participants (H_2 and He based) were accepted.

Mr Garvey requested what the actual method was as many variations were present, therefore, a stricter description was required. Ms Vinke, seconded by Mr Pigeon, replied that both H_2 and He were carrier gases leading to acceptable results, only N_2-based analytical results were not acceptable.

The method can be promoted to a **provisional CIPAC method**. considering the data sets using hydrogen or helium as eluent gas with the need for a stricter description of the method.

4.3 Ethephon

No further comments were received, the method can be promoted to a **provisional CIPAC method**

without the need to eliminate outliers in the case of TC samples

4.4 Flumioxazin

No further comments were received, the **method extension** can be accepted.

4.5 Isocycloseram

No further comments were received, the method can be promoted to a **full scale CIPAC trial**.

4.6 Matrine

No further comments were received, the method can be promoted to a **provisional CIPAC method**.

4.7 Metholachlor

Clarification was requested if 180ºC would be acceptable instead of 182ºC. Mr Pigeon questioned whether the column was fit for purpose. It should be clarified whether using a column of different polarity would lead to peak without fronting. No further comments were received, the method can be promoted to a **full scale CIPAC trial**.

4.8 Methoprene

No further comments were received, the **method extension** can be promoted to a **provisional CIPAC method**.

4.9 Pirimiphos-methyl

No further comments were received, the **method extension** can be promoted to a **provisional CIPAC method**.

4.10 *S*-methoprene

It was confusing for the audience that two different reports were presented on this subject.

The first being a small scale study into the presence of free methoprene in CS formulations and a second presentation about the determination of *S*-methoprene in TC material.

Related to the small scale study for the determination of the presence of free methoprene in CS formulations Mr Pigeon asked if it was fully known whether complete extraction had been achieved. This should be clarified. After clarification the method can go to a **full scale CIPAC trial**.

On the small scale collaborative study for the determination of the ratio of *R*- and *S*-methoprene in three TC samples Ms Vinke, Mr Pigeon, and Mr Patrian asked to clarify the aim of the method. Clarification was also asked why Turbovap equipment was used as horizontal rollers are described in the original CIPAC method. This should be clarified and Mr Pigeon will contact Ms Song.

4.11 SYN549888 was not discussed.

4.12 Trifluralin

The method can be promoted to a **provisional CIPAC method** considering all data sets, without the elimination of outliers.

4.13 Deltamethrin + chlorfenapyr

Mr Garvey remarked that the column was not specified in the original method and they tried 3 columns until the proper one was found and asked to define the column needed. Mr Pigeon remarked that a specific column was mentioned in the presentation but it was missing in the published method. Mr Garvey suggested that, instead of requiring a very specific HPLC column, it might be more useful to specify the peak resolution parameter as a base for the HPLC column selection.

Mr Hänel requested whether the abbreviation for long lasting nests should be LN or ITN. Mr Pigeon replied that CropLife uses LN but that WHO has changed it into ITN. Mr Hänel then remarked that CIPAC follows CropLife and therefore the abbreviation should be LN. A footnote should be added to the method explaining the relation between LN and ITN.

The method can be promoted to a **provisional CIPAC method** after fulfilling the requested changes in the method.

4.14 Bifenrhrin, pyriproxyfen and PBO

The method can be promoted to a to a **full scale CIPAC trial** when a clarification about sample inhomogeneity is added.

4.15 Attrition resistance of tablets

This was a DAPF trial and the method wasn't changed. Mr Loreto wondered whether there was any difference between attrition using glass beads or not. Ms Vinke will clarifiy. Ms Tessier suggested to alter the use of tablet length into tablet diameter.

The method can be promoted to a **provisional CIPAC method** after the clarifications. The method will replace the existing ones.

4.16 Discharge rate of aerosol dispenser

Mr Hänel requested whether the use of tubing has to be specified as it is not requested on the label. And more in general Ms Tessier asked how CIPAC should deal with clear differences in packaging types as they result in different application rates. Mr Pigeon remarked that both tested packaging types were functional on which Ms Tessier replied that pesticides are within CIPAC territory but the packaging types are not. Mr Hänel proposed to promoted the method to a provisional CIPAC method but that a clarification from DAPF would be necessary. This was accepted by the meeting.

The method can be promoted to a **provisional CIPAC method** when an acceptable clarification from DAPF was received.

4.17 Discharge rate of trigger sprayer

Mr Wiese remarked that there are a lot of formulations which are a kind of devices. The method can be promoted to a **provisional CIPAC method**.

5.1 MT 160 Spontaneity of dispersion of suspension concentrates

MT 160.1 can be accepted as a **provisional CIPAC method** and it supersedes MT 160.

6.1 Validation of analytical methods (draft guidance)

This item will be discussed at tomorrow's closed meeting.

11. Matters related to FAO and WHO specifications

Mrs Yang (FAO) expresses their genuine appreciation of all the work CIPAC performs, for the productive and fruitful meeting where 95% of the methods discussed are relevant for the FAO specifications. She hopes to continue this excellent collaboration as the methods for impurities are also a weakness for JMPS.

12. Any other business

No remarks, comments or questions were received.

13. Closure

Mr Hänel thanked the organising team and the participants for their contribution to the success of the meeting and closed the meeting.

Ralf Hänel	László Bura	Theo de Rijk
Chairman	Secretary	Assist. secretary