# CIPAC

## COLLABORATIVE INTERNATIONAL PESTICIDES ANALYTICAL COUNCIL LIMITED

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

## Minutes of the 67th Annual meeting

## The 67th meeting was held on Wednesday 21st and Thursday 22nd June in Braunschweig, Germany

### Those attending

- Items 1 to 7: members, correspondents, observers and expert witnesses.
- Items 8 to 12: 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 67th CIPAC meeting, and welcomed all the participants.

### 2. Apologies

Apologies were received from: Mr W. Bergermayer, Mr V. Chmil, Mr J. Garvey, Mr. B. Hocken, Ms V. Kmecl, Mr L. Manso, Ms Ch. Østerballe Pedersen, Ms A. Santilio, Mr Shahabuddin and Mr M. Müller.

### 3. Adoption of the agenda

The order of the agenda was changed. The minutes follow the adapted order of the agenda. Agenda 4.12 (Clethodim by Ms Junhua Song) was a last minute addition.

### 4. Reports of expert witnesses on collaborative trials

### 4.12 Clethodim by Ms Junhua Song (5362, 5363)

Ms Song presented the results of a small scale collaborative trial of clethodim TC, TK and EC in which six laboratories from China participated. Four samples were distributed: one TC material, one TK material and two EC formulations. The analysis was performed by HPLC using an $SiO_2$ column and DAD detection (254 nm). Quantitation was performed by external standard. Four laboratories used the prescribed column, two laboratories used a comparable column from a different brand and all laboratories returned results in due time. 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'. No stragglers or outliers were identified. The HorRat values of the TC, TK and EC samples were 0.40, 0.29, 0.39 and 0.47 respectively and therefore fulfilling the test criteria.

Ms Song recommended to go forward with a full scale trial.

The following comments were received from the meeting:

- Mr Bura asked if the active substance is only the *E* isomer? It was clarified that the name clethodim was originally approved for the substance with (*E*)-stereochemistry at the C=N

double bond, but in 2008 the manufacturer determined that the (Z)-isomer is also present in the manufactured product and requested that the definition be changed.
- Mr Ramesh asked whether the clethodim content was recalculated, as a lithium salt was used in preparation of the standard. Ms Song confirmed that this was taken into account.
- Mr Pigeon requested why dichloromethane was used as in some countries the use of halogenated solvents has to be drastically reduced. Ms Song will contact the laboratory which developed the method to investigate whether dichloromethane can be replaced.

### 4.6 Methoprene by Ms Junhua Song (5357, 5358)

Ms Song presented the results of a full scale collaborative study for the determination of free (non-encapsulated) methoprene in three CS formulations by GC-FID. Ten laboratories participated and eight laboratories from Europe and China returned results. The method consisted of extraction with *n*-hexane according to a strictly defined procedure and quantitation was performed by GC-FID on a cross linked dimethylpolysiloxane capillary column and internal standard correction. Four independent determinations per sample were performed. The participants reported several deviations of the method, however none of them were assessed to be critical and therefore all results were accepted. 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'. The results were very variable which was reflected by the HorRat values of 8.5, 9.9 and 9.2 respectively. However, Ms Song believed that the method can fulfil its function to monitor the content of the free active ingredient in CS formulations as the encountered concentrations of methoprene was way below 5% of the total active ingredient content as specified by WHO.

The following comments were received from the meeting:

- Mr Benke remarked that the uncertainty was coming from the sample preparation and that statistical evaluation should not have been performed when there were such huge differences in the results. Ms Song responded by stating it was the best they could do.
- Mr Mink added that the extraction method was clearly not robust and therefore the method was not fit for purpose. Already very slight variations of the extraction procedure had a very large impact on the results.
- Mr Hänel remarked that the evaporation depends on the speed of the rotary evaporator and if you can choose any speed it is not possible to compare the results.

### 4.7 *S*-Methoprene by Ms Junhua Song (5359, 5360)

Ms Song presented the results of a full scale collaborative study for the determination of S-methoprene in three TC materials by normal phase HPLC. Twelve laboratories participated and nine laboratories from Europe and China reported results. The aim of the analysis was to distinguish *R*-methoprene from *S*-methoprene and to determine the *S*-methoprene content of TC material. The sample was dissolved in *n*-hexane and the ratio of *S*-methoprene is determined by normal phase HPLC on a Chiralpak AD-H silica column with detection at 254 nm. One laboratory reported that resolution of the peaks of interest could not be achieved and that retention times drifted during the run. However, the data of this laboratory were not rejected. 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'. The results were in line with the requested HorRat ratios with 0.47, 0.55 and 0.51 respectively.

Ms Song proposed the method to be accepted as a provisional method.

The following comments were received from the meeting:

- Mr Patrian asked what the purpose of the analysis was as one laboratory clearly indicated that no separation could achieved but the result was accepted for final evaluation. Is it to analyse *S*-methoprene as an identity, or to quantify *S*-methoprene? Ms Song answered that this was developed for the purpose of the FAO specification.
- Mr Grassi asked whether the current CIPAC method distinguished between the different

isomers and Ms Song answered that the current method only is capable of analysing the total methoprene content.

### 4.5 Metolachlor by Ms Yue Wang (5335, 5336)

Ms Wang presented the results of a full scale collaborative study for the determination of the metolachlor content after methanol extraction in two TC materials, and three EC and three EW formulations by GC-FID on a capillary column, FID detection and internal standard correction. The aim of the method was to replace the current prescribed packed GC column with an HP-5 (or equivalent) capillary column. Laboratories from Europe, China and the USA participated and 18 returned results. Some laboratories reported minor deviations but they were not assessed as critical. And although three stragglers and one outlier were identified, the resulting HorRat values without eliminating them ranged from 0.2 to 0.8 therefore fulfilling the requested criteria.

Ms Wang recommended to accept the current method as provisional CIPAC method.

The following comments were received from the meeting:

- Mr Grassi asked whether metolachlor or S-metolachlor was analysed. Ms Wang confirmed that total metolachlor was analysed, not only the S-isomer.

### 4.9 Tebuconazole by Ms Yue Wang (5345, 5346)

Ms Wang (replacing Mr Terry Wang) presented the results of a small scale collaborative trial for the tebuconazole content in two TC materials, and two WP, two EC and two SC formulations. Four laboratories participated and all four reported their results in time. The aim of the method was to replace the current prescribed 5 m thick film capillary column (CIPAC 494, Handbook H) with a more common available 30 m narrow bore thin film capillary column. The sample preparation is identical to CIPAC 494 and the quantification was performed against dicyclohexyl phthalate as internal standard. The four participating laboratories strictly followed the prescribed procedure, however one laboratory used helium as carrier gas instead of the prescribed nitrogen. This was assessed as not critical. 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'. Low HorRat values were obtained ranging from 0.13-0.43 indicating high quality results. The proposed method is considered to be appropriate for the determination of tebuconazole in TC, WP, EC and SC formulations.

Ms Wang proposed to continue with a full scale collaborative study.

The following comments were received from the meeting:

- Ms Tessier asked what the aim of the study was: replacing CIPAC 494 or adding formulation types? Ms Wang confirmed that the only aim was to replace a less common GC column by a more readily available one.
- Mr Mink remarked that the method as proposed makes use of $N_2$ whereas CIPAC 494 makes use of He as carrier gas. Ms Wang will contact the laboratory responsible for the development of the method.

### 4.10 Tembotrione by Ms Yue Wang (5341, 5342)

Ms Wang presented the results of a small scale collaborative trial for the determination of the tembotrione content in two TC materials, and two SC and two OD formulations after extraction with acetonitrile by HPLC on a C18 column with UV detection at 284 nm and external standardization. Four laboratories participated and returned results in time. All participants strictly complied with the given analytical conditions and no deviation or remarks 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 0.2-0.6 indicating compliance to the test criteria. Ms Wang considered the method to be suitable validated and recommended continuation with full scale collaborative trial.

No questions were asked by the meeting.

### 4.2 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.

### 4.3 Gibberellic acid by Ms Xu Rong (5337, 5338)

Ms Rong presented the results of a small scale collaborative study for the determination of the gibberellic acid content of three TC materials by C18 HPLC with UV detection at 210 nm and quantification by external standardization. The extraction was performed with methanol followed by a dilution with HPLC eluent (acetonitrile: 0.05% phosphoric acid in water 33:67 (v/v)). Four laboratories participated, all laboratories reported in time and one laboratory reported that ultra sonication was performed to improve the dissolution. No further 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 0.4-0.5.

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

No questions were asked by the meeting.

### 4.1 Bifenthrin, chlorfenapyr, pyriproxyfen and PBO by Ms Marie Baes (5347, 5348)

Ms Baes presented the results of a full scale collaborative trial for the determination of bifenthrin, chlorfenapyr, pyriproxyfen and piperonyl butoxide (PBO) in two bifenthrin TC materials, two pyriproxyfen TC materials, two chlorfenapyr TC materials, two PBO TC materials and four LN formulations by GC/FID on a DB-210 (or equivalent) capillary column and internal standard quantification. 16 Laboratories from Europe, Asia and USA participated and 13 laboratories reported results in time. Five laboratories reported having difficulties with the ultra-sonication step (one hour at 80°), especially for not being able to reach the requested temperature. This was regarded as a major deviation. Other comments to the method were regarded as not critical. The statistical evaluation was performed according to the 'Guidelines for CIPAC Collaborative Study Procedure for Assessment of the Performance of Analytical Methods'. In all TC materials the HorRat values were good (range 0.46-0.95) or acceptable (1.3-1.8). However, in the four LN formulations the HorRat values were less satisfactory with one good result (0.87), three acceptable results (1.3-2.0) and six not acceptable results (2.1-7.1). Ms Baes therefore recommended to conduct a second full scale trial while taking into account the following adjustments: a higher analytical standard weight (25-50 mg) and to take care that the required temperature and energy dissipation of the sonication equipment is reached as the extraction of the incorporated active

substances is difficult. Monitoring of the true temperature was deemed essential. Furthermore, Ms Baes suggested that the second trial should contain only the LN samples as otherwise probably not enough participants would join and that the LN samples should be prepared and homogenized in the laboratory of the organizer before dispatching to the participating laboratories.

The following comments were received from the meeting:

- Mr Hänel responded that he was not happy with the argumentation of requesting to leave out the TCs for the next full scale trial.
- Mr Benke recommended to use the graduated flask in the sample preparation phase instead of just adding exactly 1 ml of internal standard. Ms Baes replied that because of the accurate addition of the internal standard the use of a graduated cylinder was of no influence to the outcome of the trial.
- Mr Di Loreto asked whether the extraction was optimized as one hour seemed to long? Ms Baes confirmed that this was done in preparation of the interlaboratory trial and that after 45 min the full extraction was not reached, and 1 h was needed.
- Mr Treutwein suggested the use of very strong solvents which are capable of dissolving the polymer net material. Ms Baes replied that was not considered because of the toxic nature of these solvents and because of the possible occurrence of changes of the actives substances. It was demonstrated during the validation that heptane was extracting the actives.
- Mr Ramesh asked what the usefulness of this trial was as already five out of eight participants encountered major difficulties in the extraction process and suggested as a next step to perform a small scale trial focussing on the extraction only.
- Mr Bura suggested to repeat the analysis with the five laboratories who reported extraction problems. Ms Baes responded by telling that the organizers already had decided to repeat the trial because of the bad overall results.
- Ms Baes also mentioned that the bad HorRat scores could be caused by sample inhomogeneity, underperforming extraction conditions or a combination of both.

### 4.4 Isocycloseram by Mr Christian Mink (5349, 5350)

Mr Mink presented the results of a full scale collaborative trial for the determination of isocycloseram in two TC materials and three WP formulations after dissolving in or extraction with acetonitrile, reversed phase HPLC, UV-detection at 265 nm and external standardization. 16 Laboratories from Europe and Asia participated and all laboratories reported in time. One laboratory used a different stationary phase which could influence the outcome of the trial, the other reported deviations were assessed as not critical. The statistical evaluation resulted in HorRat values of 0.3-0.6, which included one outlier and included the laboratory which applied another stationary phase.

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

No questions were asked by the meeting.

### 4.8 PBO by Mr Molingzhi (5343, 5344)

Mr Molingzhi presented the results of the CIPAC 33/LN/(M)/3 method extension for PBO. CIPAC 33/LN/(M)/3 is suitable for PBO impregnated insecticidal nets in the presence of deltamethrin. The extension focusses on the suitability of CIPAC 33/LN/(M) for determining PBO in coated insecticidal nets in the presence of deltamethrin. Furthermore, some modifications were applied: xylene was replaced by acetone and the injection split ratio was adjusted. After reflux extraction for 30 minutes with acetone PBO is determined by capillary GC/FID and internal standardization. Five samples of coated insecticidal nets were tested by two laboratories resulting in HorRat values ranging from 0.31-0.91.

Mr Molingzhi recommends the extension CIPAC 33/LN/(M)/3 to coated insecticidal nets.

The following comments were received from the meeting:

- Mr Patrian remarked that a split ratio of 0:1 (as mentioned in the presentation) is not possible. Mr Molingzhi remarked that this is written in the Handbook. Mr Patrian suggested that this was a typo and that it should have been 10:1.
- Mr Pigeon also remarked that the replacement of xylene by acetone was a welcome improvement of the method.

### 4.11 Solvents in chlorantraniliprole by Ms Mary Ellen McNally (5351, 5352)

Ms McNally presented the results of a small scale trial for the determination of two solvents in chlorantraniliprole one TC material and one WG and three SC formulations by GC-FID on a capillary WAX column. Six laboratories participated resulting in seven datasets as one laboratory reported two independent results. All laboratories reported deviations but none were assessed as critical. After removing outliers/stragglers the HorRat ratios ranged from 0.46-1.6. The results from acetonitrile deviated a little bit more with a HorRat range of 1.4-4.3. Ms McNally explained the larger acetonitrile HorRat range by the low concentration of acetonitrile in the investigated products: in the WG formulation the encountered concentration was approximately 30 times lower than regulated and in the SC formulations the encountered concentration was approximately 10 times lower. Previous internal method validation with spiked solvents resulted in recoveries between 95% and 105% with low RSDs (0.3-8.0%).

Ms McNally therefore recommended continuation with a full scale collaborative trial.

No questions were asked by the meeting.

## 5. Reports of expert witnesses on other matters

**5.1 MT 148.2 Pourability by Mr Burkhard Wiese (5355)** was cancelled and will be on the agenda of the 68th CIPAC annual meeting in 2024.

### 5.2 MT 185 Wet sieve test by Ms Claudia Vinke (5353)

Ms Vinke could not attend the meeting and presentation was given by Mr Patrian. The aim of the proposal was to combine MT 182 with MT 185 while introducing editorial adjustments and removing obsolete references. The most important adjustment is the way in which no remaining visible residues will be reported: it is proposed to report this as '<0.1%'. Furthermore, it is tressed that the use of the 75 µm mesh is obligatory.

Mr Patrian recommends to combine MT 182 with MT 185 in 185.1 which supersedes both MT182 and MT 185.

No questions were asked by the meeting.

### 5.3 Density of solids and liquids with automated systems by Mr Jonas Treutwein (5356)

Mr Treutwein reported about the reasoning behind the addition of a new procedure for the determination of the density of solids and liquids. The current methods (MT 3, MT 186, A3 and OECD 109) do not include the current practice of the majority of laboratories of applying fully automated gas pycnometers for solids or oscillating density meters for liquids. Also MT 3 is already very complicated therefore a new method incorporating the new techniques is more efficient than rewriting MT 3. It was also stated by Mr Treutwein that an interlaboratory proficiency test would not be required as the equipment is fully automated and therefore can only lead to erroneous results if operated in a not correct way.

Mr Treutwein therefore recommends that the new method 'Density of Solids and Liquids with automated systems' should be adopted as provisional CIPAC method.
The following comments were received from the meeting:

- Mr Wolffram asked whether the proposed method is related to a certain instrument brand. This was not the case, no CIPAC endorsement will be granted.

- Mr Di Loreto asked why a collaborative trial is not needed? Mr Treutwein answered that a collaborative trial is not needed because you have just to use the instrument according to its instructions.
- Mr Benke remarked that traditional methods, e.g. by using a pycnometer, is more accurate. Mr Treutwein answered that the accuracy of the automated systems is sufficient and that the current methods stay in place.
- Mr Hänel asked why an automated method should be registered as a CIPAC method. Mr Treutwein answered that it necessary because of requests from some authorization bodies. Nevertheless, the question remains why a fully automated method should be a CIPAC method.
- Ms Tessier asked whether the equipment is widely available. Mr Treutwein answered that they were.

### 5.4 MT 36.3. Emulsion stability and re-emulsification by Mr Paolo Grassi (5361)

Mr Grassi presented a proposal to supersede MT 36.3 by a new proposal (to be registered as 36.4). The revision is intended to give better indications on the evaluation of initial emulsification and emulsion stability and is harmonised with similar methods regarding the stability of formulations in water. In the proposal the single inversion is omitted and a more specific description of the visual interpretation is given. Furthermore, the two hour waiting period is mandatory and the other waiting periods will only be needed when the two hour waiting period results in an out of specification determination. The temperature is changed from 30 ± 2 °C to 25 ± 5 °C.

The following comments were received from the meeting:

- Ms Tessier asked why the 0.5 hour waiting period has to be removed. Mr Grassi replied that it will be optional and the 2 hours waiting period will be mandatory and that the current procedure is complicated and that a simplification is needed.
- Mr Pigeon remarked that in the actual proposal CIPAC water D is mentioned, however it might be useful to specify two types of water. Mr Grassi suggested that it might be better to say to use any type of water or what the authority requests.
- Mr Dubois remarked that both conical flasks and emulsion tubes are mentioned. However, they differ considerably in appearance and will lead to different interpretations of the emulsion stability. Mr Grassi answered that this is why it is considered optional, to be used for development, as it is not possible to compare the results.
- Ms Breedt asked whether conical flasks as emulsion tubes were tested and compared. This was not the case. She also asked if the flasks cannot be inverted 30 times? The answer was that the change from 1 to 30 would be too big.

## 6. Revision/update of CIPAC guidelines

### 6.1 CIPAC Guideline by Mr Dirk Wolffram (5254)

Mr Wolffram presented a proposal to unify all individual CIPAC Guidelines into one new document, as the current documents contain redundant and/or obsolete information. The aim of the new unified document is to restructure all information in a more comprehensible way, to make it a ‘living’ document that is easily adaptable (including version history) and to combine both the quantitative methods and the MT methods. A draft proposal will be sent to all CIPAC members for comment within a four months’ reaction time with the aim to finalise it as provisional document during the 2024 CIPAC meeting.

The following comments were received from the meeting:

- Ms Tessier mentioned that this was a good idea and this opinion was underlined by other members of the meeting.

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

Mr Bura mentioned that no replacement of obsolete methods or comments to existing methods were reported to him. One erratum in Handbook F was mentioned and dealt with.

**8. Minutes of the 66th meeting (5332/P)**

The minutes were circulated to the participants by e-mail. After comments were received and agreed upon no further comments were sent, so as a conclusion, the minutes were accepted as a true record of the last year meeting.

**9. Secretary's report (5333/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**

**1023 14-hydroxylated brassinosteroid**

It appeared that a question from Mr Watanabe has not been answered yet, however the Company clarified the issues raised in the question, but the answer was not forwarded in time by the secretary to Mr Watanabe.
The reversed phase HPLC method (CIPAC/5311) for the determination of 14-hydroxylated brassinosteroid in TK and SL formulations was accepted as **full** CIPAC method after additional justification for the Horrat >1 and for the eliminations.

**687 Difenoconazole**

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 considering the data sets using hydrogen or helium as eluent gas with a stricter description of the method.

**373 Ethephon**

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.

**578 Fumioxazin (extension)**

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.

**1024 Matrine**

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.

**414 Methoprene (extension)**

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.

**239 Pirimiphos-methyl**

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.

**183 Trifluralin**

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.

**333+570 Deltamethrin + chlorfenapyr**

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 with modification of the description of the method considering the column and specifying the resolution.

**MT 178.3 Attrition resistance (supersedes 178.1 and 178.2)**

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 with the editorial changes and with the remark that MT 178.3 supersedes MT 178 and MT 178.2.

**MT 201 Discharge rate of trigger dispenser**

Clarification from DAPF will be requested by Mr Hänel and Mr Bura. If the clarification is sufficient then the method can be promoted to a **full** CIPAC method. It was clarified that Technical monograph No.2 (8th edition) contains the formulation type TD (trigger dispenser).

**MT 202 Discharge rate of aerosol dispenser**

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.

**MT 160.1 Spontaneity of dispersion of suspension concentrates (supersedes 160)**

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 with the remark that MT 160.1 supersedes MT 160.

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**Active substances discussed this year:**

**4.12 Clethodim**

It was clarified that the name clethodim was originally approved for the substance with (*E*)-stereochemistry at the C=N double bond, but in 2008 the definition was changed including also the (*Z*)-isomer. No further comments were received, the method was recommended to a **full scale** CIPAC trial.

**4.6 Methoprene**

Mr Hänel remarked that the presenter was very optimistic about the results of the method. Mr De Rijk added that already very slight variations of the extraction procedure resulted in large result deviations. Ms Karassali added that in her laboratory they strictly followed the method however were not able to report reproducible results. Mr Benke remarked that statistics should not have been performed for this data set as the results were too variable. Mr Pigeon added that the extraction procedure is not exact enough. Mrs Baes mentioned that the uncertainty is higher than 50% of the tolerance. Mr Hänel summarized the reactions of the meeting by stating that the method was not robust, that the number of participants was lower than eight and statistics should not have been performed. The meeting recommended to develop a more robust method. Mr Hänel and Mr Bura will contact Ms Song.

**4.7 S-Methoprene**

Mr Hänel suggested that a decision should be postponed as the results from Lab 9 should be discarded and statistics should be recalculated. If the recalculated results are within the relevant criteria the method can be promoted to **provisional** CIPAC method. Mr Hänel and Mr Bura will contact Ms Song.

**4.5 Metolachlor**

The method is intended as a replacement of the current method. Mr Pigeon remarked that the proposed method is much better compared to the current method.

No further comments were received, the method can be promoted to **provisional** CIPAC method with the remark that the new method supersedes the current method.

### 4.9 Tebuconazole

The proposal relates to the replacement of a less common GC capillary column with a more common one. The new method is a step forward but covers less formulation types when compared to the current method. Therefore, the applicability of the new method will be reduced when the new method replaces the current method. Mr De Rijk remarked that a suggestion could be given to the organizers to enlarge the scope of the new method to encompass the scope of the current method.

No further comments were received, the method was recommended to a **full scale** CIPAC trial including the suggestion to enlarge the scope of the new method.

### 4.10 Tembotrione

No further comments were received, the method is recommended to a **full scale** CIPAC trial.

### 4.2 Coronatine

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.

### 4.3 Gibberellic acid

No further comments were received, the method was recommended to a **full scale** CIPAC trial.

### 4.1 Bifenthrin, chlorfenapyr, pyriproxyfen and PBO

Mr Hänel proposed that both Mrs Baes and Mr Pigeon do not have to leave the room under the condition that they would not interfere with the discussion. This was accepted by the meeting. In her presentation Ms Baes mentioned that the results of the presented full scale trial were not acceptable and that a second trial should be performed. It was proposed to omit the TC materials which was accepted by the meeting. However, the second suggestion related to the sample pretreatment to be performed by the organizing laboratory instead of all individual participating laboratories encountered some opposition of the meeting as Mr Hänel, Mr De Rijk, Mr Di Loreto and Mrs Breedt remarked that the extraction was a crucial step in the method and should therefore be carried out by each individual laboratory. Mr Di Loreto also remarked that it is the duty of the organizer of the trial to deliver homogeneous sample material. Ms Nováková suggested that both intact LN nets and homogenized LN nets could be sent to all participants. Mr Hänel summarized all reactions by allowing that a second **full scale** CIPAC trial can performed under strict control of the sample pre-treatment by the individual participating laboratories and strict monitoring of the temperature and energy dissipation of the ultrasonic extraction (as suggested by Ms Baes during her presentation).

### 4.4 Isocycloseram

It was not clear to the meeting if an identity test was available or performed.

The method can be promoted to a **provisional** CIPAC method with the remark that an identity test should be available.

### 4.8 PBO

The extension of the current method can be promoted to a **provisional** CIPAC method

### 4.11 Solvents in chlorantraniliprole

Mr De Rijk mentioned that some of the HorRat values were far beyond the acceptable range. According to the presenter this was caused by the very low concentrations of the investigated solvents in the sample material. Although not relevant in a small scale trial high HorRat values will

be relevant in a full scale trial.

The method was recommended to a **full scale** CIPAC trial. Mr De Rijk contacted the organizers in order to discuss other validation options.

## 5. Reports of expert witnesses on other matters

### 5.1 MT 148.2

The item was postponed to 2024.

### 5.2 MT 185.1 Wet sieve test

No further comments were received, the new method can be promoted to **provisional** CIPAC method under the prerequisite that it supersedes both MT 182 and MT 185.

### 5.3 Density of solids and liquids with automated systems

Mr Hänel asked the meeting whether the handling of a fully automated system should be included in a CIPAC method. It could be judged as an endorsement of a certain brand of equipment. Mrs Nováková suggested to put the method on the website only, but without CIPAC 'recognition'. Mr Pigeon suggested to incorporate the automated system in the current MT method. Mr Bura and Mr De Rijk stressed the importance of performing an interlaboratory test as correct calibration of the system was vital for obtaining correct results and because very viscous materials require a dilution step in order to obtain correct results.

It was decided that DAPF would be contacted by Mr Hänel and that no promotion could be granted. The request will return on the agenda of the 68$^{th}$ CIPAC annual meeting in 2024.

### 5.4 MT 36.3. Emulsion stability and re-emulsification

Mrs Tessier had concerns about the proposed method. It is not up to the CIPAC method to decide whether or not a time point is necessary. Regulatory authorities can decide whether this time point (30 min) is needed or not but it should be included in the test method. Also in the FAO manual in chapter 4.5.45 the description of the Emulsifiability clause still states after 0.5 hrs as a result that needs reporting. The use of the graduated tubes – this is given as optional but the discussion in the meeting indicated that inversion procedure was different using these tubes – this needs further consideration.

Mr Plumb mentioned that oil was not mentioned anymore in the new proposal as was the possibility of adding a colourant to enhance the visibility. A good justification as to why this is not needed anymore is needed. Mr Pigeon remarked that the type of water should be clearly defined as there are differences between the use of types of water between the FAO/WHO method and the proposed CIPAC method.

Mr Hänel remarked that further explanation from DAPF is required before a decision can be made.

## 6. Revision/update of CIPAC guidelines

### 6.1 CIPAC Guideline

Mr Hänel and Mr Bura proposed to send the first draft of the new guideline also to the correspondent CIPAC members and to industry representatives. Mr De Rijk asked whether CIPAC independence could be challenged if industry is participating in the new Guideline document. Mr Hänel responded by stating that it was a first draft only and that the industry input was needed as they have to perform the majority of the laboratory work. Furthermore, no pressure from industry is expected. It was agreed that the first draft version will also be available from the website for comments with a defined deadline.

## 11. Matters related to FAO and WHO specifications

There were no matters related to FAO and WHO specifications.

**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.

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