CIPAC

COLLABORATIVE INTERNATIONAL PESTICIDES ANALYTICAL COUNCIL LIMITED

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

Minutes of the 61th Annual meeting

The 61th meeting was held on Wednesday 14th June 2017 in FAO Headquarters, Rome, Italy.

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

1. Welcome and introductory remarks by the Secretary
The secretary, Mr L. Bura, opened the 61th CIPAC meeting, and welcomed all the participants. As Mr R. Hänel due to health problems could not attend the meeting, Mr Bura chaired the meeting. Mr T. De Rijk acted as assistant secretary and Mr L. Benke acted as temporary assistant secretary during the meeting.
In the last year three of our colleagues, Hans-Paul Bosshardt, Walter Dobrat and Shiv Pandey passed away. Mr. Bura asked the participants of the meeting to commemorate them.

2. Apologies
Apologies were received from: Mr R. Hänel, Mr M. Müller, Mr J. Garvey, Mr A. Martijn, Mr A. Plumb, Ms F. Mathieu and Mrs J. Schlosserova.

3. Adoption of the agenda
The following amendments were made to the agenda:
Item 4.1 (acephate) was removed, no data were received.
Item 5.7 was rescheduled to 5.1 on request of Mr Ramesh. The original items 5.1-5.6 were rescheduled accordingly. The modified agenda was adopted.

4. Reports of expert witnesses on collaborative trials

4.1 Chlorpyrifos-ethyl by Ms Yin Qing (5080, 5081)
Ms Yin Quin presented the results of a **full scale** collaborative study on the determination of chlorpyrifos-ethyl in long lasting insecticidal net samples.
Chlorpyrifos-ethyl was extracted from the long lasting insecticidal net by acetonitrile and determined by reverse phase high performance liquid chromatography using UV detection at 290 nm with 1,4-dibromonaphthalene as internal standard. The analyte solution contained about 10 mg of chlorpyrifos-ethyl and 10 mg of 1,4-dibromonaphthalene in 50 mL solution.
Fifteen laboratories received samples for this collaborative trial and all of them submitted results. The statistical evaluation was carried out according to DIN ISO 5725 and the guidelines in the CIPAC document “Guideline for CIPAC collaborative studies Procedure for Assessment of Performance of Analytical Methods”. The data were tested for outliers firstly using Cochran’s test on the within laboratory variance and then using Grubbs test on laboratory means to test for the between laboratory variance.
The data of laboratory 3 was considered invalid due to noncompliance with the reference method. The statistical evaluation was made using all data (15 participants) and after elimination of outliers

(14 participants).
The calculated RSD_R values met the Horwitz criteria for the tested samples in both cases.
The organizers of the trial proposed the analytical method for chlorpyrifos-ethyl in long lasting insecticidal net to become provisional.

The following comment was received from the meeting:

- Mr Manso commented that it would be useful to receive the report in time before the CIPAC meeting. Mr Bura answered that the reports were sent out in time, the raised problem was just an individual case.

4.2 d-tetramethrin by Mr Quibai Jiang (5101, 5102)
Mr Quibai Jiang presented the results of a **full scale** trial on the determination of the active ingredient content and isomer ratio of the diastereomers of d-tetramethrin in technical material. d-tetramethrin is a mixture of the isomers (1*R-trans*, *R*), (1*R-trans*, *S*), (1*R-cis*, *R*) and (1*R-cis*, *S*) of tetramethrin in an approximate ratio of 4:4:1:1. In practice the *trans* isomer range is 75-85 % and the *cis* isomer range is 15-25 %.
d-tetramethrin was determined by gas chromatography/flame ionization detection, with internal standardization. The isomer ratio of the diastereomers was determined by normal phase high performance liquid chromatography using a CHIRALPAK® AY-H HPLC column, 250×4.6 mm (i.d.), 5 µm particle size; *n*-heptane-ethanol-diethylamine, 930+70+1 (v/v) mobile phase, and detection wavelength of 230 nm.
Five TC samples were sent to fourteen laboratories. During the trial the participants conducted duplicate determinations on two different days with duplicate injections for each sample.
The statistical evaluation was carried out according to the CIPAC guidelines. The results were tested for outliers, firstly using Cochran's test on the within laboratory variance and then using Grubbs test on laboratory means to test the between laboratory variance. The tests were carried out at the alpha level of 0.01 for outliers and 0.05 for stragglers.
During the check of the GC results, three Cochran outliers occurred. After eliminating these data, no other outliers were found.
After the evaluation the calculated RSD_R fulfilled the Horwitz's criteria for all technical materials.
Based on these results, the organizers recommended that the d-tetramethrin method should progress to a fully validated CIPAC method.

The following comments were received from the meeting:

- Mr Bura drew the attention on the problem of the anonymity of the participating laboratories and asked that in the future the organisers should respect this demand.
- One participant asked whether it was necessary to use dichloromethane as solvent.
- Mr Bura showed that even without eliminating the outliers the results of the measurements met the Horwitz's criteria.
- The remarks about the quality of the results of laboratory 5 in the conclusion of the presentation were based on an assumption, not on actual data.

4.3 Flupyradifurone by Mr Michael Haustein (5094, 5095)
Mr Michael Haustein presented the results of a **full scale** study carried out by 22 laboratories on a TC and six different formulation samples (AL; EC; EW; FS; SL; WG) to demonstrate that the method is suitable for the determination of flupyradifurone in technical and in main formulation types. The homogenized samples containing flupyradifurone were dissolved in a solvent mixture of acetonitrile/purified water, followed by active ingredient determination using gradient reversed phase high performance liquid chromatography, UV detection at 280 nm with an external standard calibration, using a Phenomenex Kinetex C18, 50 mm x 4.6 mm, 2.6 µm particle size column. 22 laboratories sent back the results, 13 participants used the column material described in the CIPAC trial, 8 laboratories used a different column type and data from one laboratory were not considered due to significant changes applied to the method.

In summary it can be stated that the method deviations, noted by the 13 participants, who used the Kinetex C18 column, did not affect the analytical results significantly and therefore all data sets were included within the statistical assessment.
The EW formulation showed crystallization due to insufficient cold stability of the formulation. The crystallization was reported by several participants. As a result of the observed crystallization only eight results were used in the evaluation of the EW formulation.
In the first round the statistical evaluation was carried out on the data of 13 laboratories which used the column described in the CIPAC method.
In addition the data of all the 21 laboratories participating in the CIPAC collaboration trial have been evaluated.
The data presented in the statistical summary showed that the method was suitable to gain acceptable and reproducible results for all samples tested and was therefore regarded to be robust.
The following comments were received from the meeting:

- It was asked whether sonication for 50 min was necessary and in which cases would be also shorter sonication time enough. The answer was that in case of FS and SE formulations it was necessary to sonicate for a longer time.
- One participant commented that the selection of 13 participants from the 21 (who used exactly the prescribed column during the trial) is not encouraged by CIPAC. Were there any specific reasons that the organisers excluded from the statistical calculation the other laboratories which did not use the Kinetex column?
- Mr Bura told that the use of the Kinetex column was a recommendation and in the final method it will be mentioned that similar columns can also be used.
- One participant asked if there is any explanation for the different behaviour of the WG formulation. The answer was that probably the insufficient extraction could cause the problem, however no investigation was performed to detect its cause.

4.4 Mancozeb by Mr Li Liunhu (5103, 5104)

Mr Li Liunhu presented the results of a **full scale** trial for the determination of mancozeb in TC and WP formulations by HPLC. 18 laboratories sent back the results in time; the statistical evaluation was carried out based on their results.
Five samples were sent to the participants, two technical materials and three WP formulations.
Mancozeb was determined by reversed phase high performance liquid chromatography using UV detection at 282 nm and external standardization.
After the elimination of outliers and stragglers the between laboratory experimental relative reproducibility standard deviation (% RSD_R) was below the calculated acceptable value based on the Horwitz's curve calculation for the mancozeb technical and WP samples.
Therefore the organizer of this trial considered that the method is suitable for the intended purpose and recommended accepting it as a provisional CIPAC method for the determination of mancozeb in TC and WP formulations.

The following comments were received from the meeting:

- One participant asked if purified standard material was used and if yes, than how did the laboratory purify the standard? Technical standard was used and was not further purified.
- It was asked whether a commercially available standard was tested and compared with the technical material. The laboratory compared their standard with the commercially available reference material.
- One participant expressed some concerns regarding the stability of mancozeb and mentioned that in some cases it was difficult to solubilise the sample. Degradation during analysis was also observed.
- Big variation between the two days measurements data was observed by several participants
- What was the purity of used standard? The purity of standard was 86%.
- Did the laboratory use own produced reference material and if this was compared with the commercially available? The laboratory used their own production standard and compared

the used standard with the commercially available one.
- Did the laboratory use the presented HPLC method for characterising the standard material? The HPLC method was used.

4.5 Metofluthrin by Mrs Kiyoko Miyakawa (5082, 5083)
Mrs Kiyoko Miyakawa presented the results of a **small scale** trial for the determination of metofluthrin in technical material and formulations using gas chromatography.
Four laboratories took part in the small scale trial and five samples were sent out to the laboratories: three technical materials and two emulsions, oil in water.
Metofluthrin was analysed by capillary gas chromatography using flame ionization detector and fluoranthene as internal standard.
The RSD_R values were smaller than those calculated by Horwitz's equation.
The GC method proposed was considered appropriate for determination of metofluthrin in TC and EW formulations. JAPAC recommended proceeding to a full collaborative trial.

The following comments were received from the meeting:
- One participant asked about the number of injections which can be performed until the liner should be changed. The answer was that it depends on the sample injected on the GC.
- One participant commented that probably the large solvent peak may cause the frequent contamination and change of the liner.

4.6 Prothioconazole by Mr Friedhelm Schulz (5095, 5096)
Mr Friedhelm Schulz presented the results of a small scale collaborative trial carried out by DAPA with 8 laboratories on one TC and four different formulation samples (EC (250 g/L prothioconazole); FS (100 g/L prothioconazole); FS (250g/L pencycuron + 8 g/L prothioconazole) and SC (480 g/L prothioconazole).
The homogenized samples containing prothioconazole were dissolved in solvent mixture of acetonitrile/purified water followed by active ingredient determination using gradient reversed phase high performance liquid chromatography with UV detection at 254 nm and external standard calibration.
After the evaluation the calculated RSD_R fulfilled the Horwitz's and HorRat criteria for all analysed samples.
Based on these results, the organizers recommended that the prothioconazol method should progress to a full scale CIPAC trial.

The following comments were received from the meeting:
- It was asked if sonication for 50 min was necessary and in which cases a shorter sonication time would be enough? The answer was that for this type of formulation the relatively long sonication time was needed.
- Some participants to the trial filtered, other centrifuged the samples. Was the centrifugation necessary? Both methods can be used.

5. Reports of expert witnesses on other matters

5.1 Extension of the scope of CIPAC 454/LN/M/3.2 to LN (incorporated type) by Mr Atmakuru Ramesh (5107)
Mr Atmakuru Ramesh presented a study of **method extension** of existing CIPAC methods for pyriproxyfen and alpha-cypermethrin in long lasting insecticidal nets.
Two laboratories took part in the study and each laboratory received 3 samples of Royal Guard®, 120 denier and 3 samples of Royal Guard® 150 denier, each containing five 25 cm x 25 cm pieces.
The method CIPAC 715/TC/M/3 was used to determine the pyriproxyfen content without any modifications.
The extraction method CIPAC/4887 (extension of CIPAC 715/TC/M/3) was used to determine the

alpha-cypermethrin (454/LN/M/3.2) with minor modifications (CIPAC/5043) using dicyclohexyl phthalate as internal standard instead of dioctyl phthalate.
The pyriproxyfen content in LN was determined by reverse phase high performance liquid chromatography, after evaporating an aliquot of the heptane extract and dissolving it in acetonitrile, using UV detection at 254 nm with di-cyclohexyl phthalate as internal standard (CIPAC 715/TC/M/3).The same sample extracted with heptane was used for the determination of alpha-cypermethrin by GC-FID (CIPAC 454/LN/M/3.2).
The values of RSD_R were smaller than those calculated by Horwitz's equation.
The modifications were considered to be minor modifications and the method extension was proposed to be accepted.

The following comments were received from the meeting:

- No questions were received.

5.2 Alpha-cypermethrin and/or chlorfenapyr in interceptor and interceptor G2 by Mr Nicolas Mabon (5089, 5090)

Mr Nicolas Mabon presented the results of a **small scale** trial for the determination of alpha-cypermethrin and chlorfenapyr content in technical material (TC) and long lasting insecticidal net (LN). Five laboratories took part in the small scale trial, two TC samples and five LN samples were sent out to the laboratories. The scope of the trial was to test the method for the determination of the active ingredient content of alpha-cypermethrin and chlorfenapyr in technical grade active ingredients and in long lasting insecticidal nets.
Alpha-cypermethrin and chlorfenapyr were determined by gas chromatography with flame ionisation detection with internal standardization.
During the statistical evaluation of the results one Grubbs' outlier occurred, the data of two different days were both too low. According to the head of this laboratory, it was due to abnormal turbidity of TC 1 solution. After eliminating the outlier data, the calculated reproducibility standard deviation (RSD_R) met the Horwitz's criteria for all technical grade materials and for all long lasting insecticidal nets.
Based on these results, it was recommended that the analytical method for determination of the active ingredient content should progress to a large scale collaborative trial.

The following comments were received from the meeting:

- Laboratory 5 duplicated the measurements and got better results.

5.3 Dinotefuran method extension to RB by Mr Onie Tsabari (5097, 5098)

Mr Onie Tsabari presented a study of the **method extension** for determination of dinotefuran in bait samples formulations by HPLC/UV.
Five fresh samples of bait formulations with the active ingredient dinotefuran and five samples after accelerated storage of 8 weeks at 40 °C were analysed using the HPLC method.
The CIPAC LC/UV method was modified for dinotefuran concentration in formulation samples. Calculations were carried out using UV detector at λ=270 nm. The results and the chromatograms of the analysis were presented.

The following comments were received from the meeting:

- One comment was received: the study was carried out in just one laboratory.
- No questions were received.

5.4 Imidacloprid method extension to UL by Mr John Dawson (5108, 5109)

Mr John Dawson presented the **method extension** of CIPAC 582/TC/M2/ to the UL formulation type with imidacloprid content, with a few minor modifications. This report was prepared to demonstrate the validity of the extension of the CIPAC 582/TC/M2/- for imidacloprid in UL formulations.

Imidacloprid was determined by reversed phase high performance liquid chromatography using UV detection at 260 nm and external standardization.
In order to apply the CIPAC 582/TC/M2/- to UL formulations containing imidacloprid, the method required an increase in the analytical run time of the HPLC measurement. This was considered to be a minor modification.
The data shown demonstrated that the method is specific and has acceptable precision (repeatability, r). Therefore, the modified method was considered appropriate for the determination of imidacloprid in a UL formulation and the extension of CIPAC 528/TC/M2/3- to UL formulations was proposed by Clarke International.

The following comments were received from the meeting:

- One observation was made regarding the assessment of the precision of the method. It was noted that the modified Horwitz equation was not used and Mr Dawson agreed to recalculate the data.

5.5 Prallethrin applicability of method to UL by Mr John Dawson (5110)
Mr John Dawson presented the **extension of method** CIPAC 743/LV/M/- total prallethrin content, to the UL formulation type, which contains prallethrin, with a few modifications. This report was prepared to demonstrate the validity of the extension of the CIPAC 743/LV/M/- for total prallethrin to UL formulations.
In order to apply the CIPAC 743/LV/M/- methodology to the formulation of interest, (CMP123-004), the following modifications were applied:

- Detector temperature changed from 270 ºC to 320 ºC
- Column oven temperature changed from 245 ºC isothermal, to 50 ºC for 0.5 min then 40 ºC/min up to 240 ºC for 15min. This was necessary in order to ensure complete separation of formulation components from the prallethrin peak.
- Injection port temperature changed from 270 ºC to 275 ºC
- Carrier gas flow changed from 35 cm/s to 41.541 cm/s (flow rate 2 ml/min)

Furthermore, due to a combination of the large sample weight required, and other ingredients of the formulation, separation of the internal standard triphenyl phosphate in the sample solution was not achievable. The calculations to determine the amount of prallethrin content were changed to external standard quantitation.
The data shown demonstrated that the method is linear, specific, and has acceptable precision (repeatability, r). Therefore, the modified method was considered appropriate for the determination of total prallethrin in a UL formulation and the extension of CIPAC 743/LV/M/- to UL formulations was proposed by Clarke International.

The following comments were received from the meeting:

- Observations were made by several participants of the meeting regarding the use of internal standard and different conditions in the method. It was a controversial issue whether these changes were minor or major changes. Finally it was considered that there were so many changes that the method could be considered as an extension of the existing method.

5.6 Extension of CIPAC/804/EW/(M) for metofluthrin/d,d-trans-cyphenothrin/PBO EW by Mrs Makiko Mukumoto (5082, 5083)
Mrs Makiko Mukumoto presented the **extension** of CIPAC/804/TC/(M) for d,d-*trans*-cyphenothrin oil in water emulsion EW. The report was prepared to demonstrate the validity of the extension method for the d,d-*trans*-cyphenothrin.
The cyphenothrin content was determined by capillary gas chromatography using flame ionization detector and triphenyl phosphate as internal standard.

In order to extend the CIPAC method a short temperature program was added to assure that all formulants elute from the analytical column. This modification was considered as a minor change. The data shown demonstrated that the method is appropriate for the determination of , d,d-*trans*-cyphenothrin in EW formulation. JAPAC proposed the extension of CIPAC 804/TC/(M)3 for d,d-*trans*-cyphenothrin to the EW formulation.

The following comments were received from the meeting:

- No other questions were received.

5.7 Extension of CIPAC/33/TC/M for metofluthrin/d,d-*trans*-cyphenothrin/PBO EW by Mrs Makiko Mukumoto (5084, 5085)

Mrs Makiko Mukumoto presented the **extension** of CIPAC/33/EW/M/3 for the determination of piperonyl butoxyde in metofluthrin/d,d-*trans*-cyphenothrin/PBO oil in water emulsion (EW).
The piperonyl butoxide content was determined by capillary gas chromatography using flame ionization detection and triphenyl phosphate as internal standard. The original method used heptadecane as internal standard, but due to separation problems it was changed to triphenyl phosphate. This modification was considered as minor change.
The data shown demonstrated that the method is appropriate for the determination of piperonyl butoxide in metofluthrin/d,d-*trans*-cyphenothrin/piperonyl butoxide EW.
JAPAC proposed the extension of CIPAC/33/EW/M/3 for metofluthrin/d,d-*trans*-cyphenothrin/piperonyl butoxyde EW formulation.

The following comments were received from the meeting:

- No other questions were received.

5.8 Determination of relevant impurities in Triflumuron TC& SC formulations by Mr Michael Haustein (5091, 5092)

Mr Michael Haustein presented a study on CIPAC **peer validation** of the analytical method for the determination of 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-trifluoromethoxyaniline in triflumuron TC and SC formulations by high performance liquid chromatography.
The peer validation was conducted with four independent laboratories through the network of DAPA and two samples were analysed (one TC and one SC).
The 1,3-bis(4-trifluoromethoxyphenyl)urea content and 4-trifluoromethoxyaniline content were determined using isocratic elution, UV detection (226 nm and 258 nm) and external standard calibration.
For all samples, the analytical method was peer-validated in terms of specificity, linearity, precision, accuracy and quantitation limit. The RSDs of repeatability for technical material and SC formulation were found to be smaller than 20% for all laboratories participating in this peer validation.
In conclusion, the proposed method was successfully peer-validated and it was considered suitable for the determination of 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-trifluoromethoxyaniline in technical material and SC formulations.

The following comments were received from the meeting:

- How was the LOQ calculated? The LOQ was calculated based on the lowest fortification level.

5.9 Retention properties of pyriproxyfen MR formulations by Mrs Makiko Mukumoto (5088)

The method for the determination of retention properties of pyriproxyfen matrix release formulations (CIPAC/4999) was accepted as a tentative CIPAC MT method at the 59th meeting in Athens (2015) with the request of additional validation data. The status of the method remained tentative as it was mentioned that data from additional 4 laboratories will be presented in the next CIPAC TC meeting. It was proposed to use an extraction at room temperature for 24 hours or the

same with a footnote of the other option. The data from the additional 4 laboratories were presented in Tokyo.

The following comments were received from the meeting:

- Mr Bura remarked that the method should have been promoted to full during the Tokyo meeting of 2016.No other questions were received.

6. Revision/update of CIPAC guidelines

Mr Bura remarked that the process of revising and/or updating of our CIPAC guidance has started. The meeting was requested to send in comments on all guidelines available on the website.

6.1 HorRat-values as criteria for the assessment of the reproducibility in the CIPAC collaborative trials

The proposal was accepted by the meeting.

6.2 Extension of the methods

The following comments were received from the meeting:

- In the CIPAC guidelines should be clarified what is considered minor and what is considered as major change because there are different interpretations.
- Changing the measurement conditions in the method is a borderline between minor and major changes.
- The problem of multi active methods was raised.
- Mr Bura asked the participants to send him all the proposals and comments by the end of the year.

6.3 DAPA proposal concerning the conduction of collaborative trials

Mrs Claudia Vinke presented the DAPA proposal concerning the performance of CIPAC collaborative trials, suggesting the use of the HorRat values as an acceptability criterion and drawing the attention on the responsibility of the organiser of the trial.

The following comments were received from the meeting:

- One participant agrees with the suggestion on choosing the participants in the second round, it would be useful if participants were selected from different countries.
- The transparency of the collaborative trials should be kept and a fair chance for the participating laboratories should be assured.
- Mr Bura asked the participants to send him all the proposals and comments in written form.

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

7.1 Comments to existing methods

Pyraclostrobin
d-phenotrin
MT 190 –determination of release properties of lambda-cyhalothrin CS formulations
MT 178.2

- Mr Bura presented the comments received concerning the mentioned methods. The errors will be mentioned in the errata on the CIPAC website.

7.2 Revision of MT methods

It is in progress.

- No remarks, comments, questions were received.

8. Minutes of the 60th meeting (5077/P)

The minutes were circulated to the participants by e-mail and were available on the website. No comments were received, as a conclusion the minutes are accepted as a true record of the last year meeting.

- No remarks, comments, questions were received.

9. Secretary's report (5078/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.

- No remarks, comments, questions were received.

10. Discussion of individual compounds

794: Chlorantraniliprole

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**.
The reversed phase HPLC method using internal standard (CIPAC/5034) for the determination of chlorantraniliprole in TC, FS, WG and SC formulations was accepted as a full CIPAC method.

738: Clothianidin, (ext.WP)

The method was provisional. It was promoted to **full CIPAC method**.
The extension of the scope (CIPAC/5051) of CIPAC method 738/WG/M/ for the determination of the clothianidin content of WP formulations was accepted as a full CIPAC method

693: Fenazaquin

The method was provisional. It was promoted to **full CIPAC method**.
The reversed phase HPLC method using external standardization (CIPAC/5036) for the determination of fenazaquin in TC and SC formulations was accepted as a full CIPAC method

454: Alpha-cypermethrin (ext. LN)

The method was tentative. It was promoted to **full CIPAC method**.
The extension of the scope (CIPAC/5043) of CIPAC method 454/LN/M/3.2 for the determination of the alpha-cypermethrin content of the long lasting insecticidal mosquito net (incorporated type) containing alpha-cypermethrin and pyriproxyfen, with the modification of having di-cyclohexyl phtalathe as internal standard instead of dioctyl phtalathe, was accepted as a full CIPAC method.

MT 46.3 Accelerated storage procedure of the MR formulations

The method was tentative. It was promoted to **full CIPAC method.**
The extension of the scope (CIPAC/5045) of CIPAC method MT 46.3 for the accelerated storage procedure of the MR formulations regarding determination of active ingredient content and retention index was accepted as a full CIPAC MT method.

Active substances discussed this year:

338 acephate

It was postponed to the next year.

221.202 chlorpyrifos ethyl

A full scale collaborative trial was presented, the method was accepted as **provisional**.
The reversed phase HPLC method using internal standard (CIPAC/5080) for the determination of chlorpyrifos ethyl in long lasting insecticidal nets was accepted as a provisional CIPAC method.

989 d-tetramethrin

A full scale collaborative trial was presented, the method was accepted as **provisional**.

(One remark was received: To contact ISO for ISO common name.)
The capillary GC method (CIPAC/5101) using internal standard for the determination of d-tetramethrin in TC formulations and the chiral phase HPLC method (CIPAC/5101) for the determination of the isomer ratio of d-tetramethrin in TC formulations were accepted as provisional CIPAC methods.

987 flupyradifurone
A full scale collaborative trial was presented, the method was accepted as **provisional**.
(One remark was received: A note should be inserted in the method regarding the sample preparation of WG formulations)
The reversed phase HPLC method (CIPAC/5094) for the determination of flupyradifurone in TC, AL, EC, EW, FS, SL and WG formulations was accepted as a provisional CIPAC method with the need to insert a comment concerning the sample preparation for the WG formulation.

34 mancozeb
A full scale collaborative trial was presented, the following comments were received:
-The proposed method had lots of problems, the standard couldn't be properly dissolved in the indicated solvent, the peak areas were not stable, the measurements carried out on consecutive days were not similar.
Other participants on the trial agreed with the first comment and further observations were received:
-FTIR technique is not an acceptable tool for quantitative analysis of the reference material?
-For stability reasons and to reduce hydrolysis, the pH should be kept at higher values, instead of 9 at values of 9.5-9.9.
-Due to the stability problems in many cases differences between the two days measurement were observed
-Mancozeb is a complicated mixture which is very sensitive to temperature and pH
Some recommendations were received:
-To increase the sonication time to 30 min.
-To adjust the pH to 9.9 for stability reasons.
The opinion of the meeting was that additional data will be requested for this method.

649 metofluthrin
The method was proposed for a full scale collaborative study.

745 prothiaconazole
The method was proposed for a full scale collaborative study.

5.1 Extension of the scope of CIPAC 454/LN/M/3.2 to LN (incorporated type) (5107)
The decision of last year was confirmed.

5.2 Alpha-cypermethrin and/or chlorfenapyr in interceptor and interceptor G2 (5089, 5090)
Comments of the meeting:
-Lots of plant protection products with two or more active ingredients will appear in the future
-Further discussions will be needed for the multi-active formulations and methods
-Methods should be adapted to the matrixes
-Mr Bura asked the participants to make suggestions via e-mail.

749 dinotefuran
The extension of the scope (CIPAC/5097) of CIPAC method 749/TC/M/3 for the determination of the dinotefuran content in bait formulations, with the modification of the eluent profile and sample preparation, was accepted as a **tentative** CIPAC method, with the need for the provision of a second data set according to the provisions of the CIPAC guideline.

582 imidacloprid
The extension of the scope (CIPAC/5108) of CIPAC method 582/TC/M2/ for the determination of the imidacloprid content in UL formulations, with the modification of the run time, was accepted as a **tentative** CIPAC method, with the need for the provision of a second data set according to the provisions of the CIPAC guideline.

743 prallethrin (applicability of method to UL (5110))
The following comments were received from the meeting:
Observations were made regarding the necessity to use of external standardisation instead of internal standardisation, different temperature program. It was a controversial issue whether these changes were minor or major changes. Finally it was considered as a major change and the meeting did not agree to consider it as a method extension. As a consequence the study has to be repeated with a proper internal standard and in at least two different laboratories.

804 d,d-*trans*-cyphenothrin (5.6 Extension of CIPAC/804/EW/(M) for metofluthrin/d,d-*trans*-cyphenothrin/PBO EW (5082, 5083))
The extension of the scope (CIPAC/5082) of CIPAC method 804/EC/(M)/ for the determination of the d,d-*trans*-cyphenothrin content in Metofluthrin/d,d-*trans*-Cyphenothrin/Piperonyl butoxide EW formulations was accepted as a **provisional** CIPAC method.

33 piperonyl butoxide (5.7 Extension of CIPAC/33/TC/M for metofluthrin/d,d-trans-cyphenothrin/PBO EW (5084, 5085))
The extension of the scope (CIPAC/5084) of CIPAC method 33/EW/M/3 for the determination of the piperonyl butoxidecontent in Metofluthrin/d,d-*trans*-Cyphenothrin/Piperonyl butoxide EW formulations, with the use of triphenyl phosphate as internal standard, was accepted as a provisional CIPAC method.

5.8 Determination of relevant impurities in Triflumuron TC& SC formulations (5091, 5092)
No remarks, comments, questions were received.
The reversed phase HPLC method for the determination of the relevant impurities 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-trifluoromethoxyaniline in triflumuron TC and SC formulations (CIPAC/5091) was noticed and adopted.

5.9 Retention properties of pyriproxyfen MR formulations (5088)
The method for the determination of retention properties of pyriproxyfen matrix release formulations (CIPAC/4999) was accepted as a **full** CIPAC MT method

6.1 HorRat-values as criteria for the assessment of the reproducibility in the CIPAC collaborative trials
The proposal was accepted by the meeting and modification of the CIPAC guidance document will be proposed. Mr Bura asked the meeting to present comments to him before the end of the year. Comments were requested also concerning the DAPA proposal on how to conduct the collaborative trials.

11. Matters related to FAO and WHO specifications
No remarks, comments, questions were received.

12. Any other business
No remarks, comments, questions were received.

13. Closure

The Secretary thanked the organising team and the participants for their contribution to the success of the meeting and closed the meeting.

László Bura	Theo de Rijk	Lajos Benke
secretary	assist. secretary	temp. assist. secretary