CIPAC

COLLABORATIVE INTERNATIONAL PESTICIDES ANALYTICAL COUNCIL LIMITED

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

Minutes of the 57th Annual meeting

The 57th meeting was held on Wednesday 12th June and on Thursday 13th June 2013 in Hotel 'Rus', Kyiv

Those attending
- Items 1 to 6 on 12th and 13th June: members, correspondents, observers and expert witnesses.
- Items 7 to 14 on Thursday 13th June: members, correspondents and observers (representatives of industry and commercial laboratories, by special invitation only)

1. Welcome and introductory remarks by the chairman
The acting chairman, Mr László Bura, opened the 57th CIPAC meeting, and welcomed all the participants. He expressed his thanks to industry for presenting the information today.

2. Apologies
Apologies were received from:
Mr Ralf Hänel, Mr Walter Dobrat, Mr Albertus Martijn, Mr Francisco Sánchez-Rasero, Mrs Julianna Schlosserova.

3. Adoption of the agenda
The following amendments were made to the Agenda:
Points 4.3 and 4.5 were presented together.

4. Reports of expert witnesses

4.1 Amisulbrom by Mr Hiroaki Takahashi (4883, 4884)
Mr Takahashi presented the results of a **full scale** collaborative study on the determination of amisulbrom in technical product (TC), water dispersible granule (WG) and suspension concentrate (SC) formulations using HPLC-UV, detection at 254 nm and external standard calibration. The trial was organised by JAPAC. Two samples of TC, one sample of WG and two samples of SC were provided. 22 laboratories offered to participate and results and data were received from 20.

Mr Takahashi remarked that a YMC Pack column is the same as an ODS column but is supplied by a Japanese company. Various types of different ODS column were used by the laboratories, and some slightly changed the mobile phase composition. Some laboratories also used shorter columns – but even when these were used no interference from other peaks was noted for the TC and WG and the retention time for amisulbrom fell within the acceptable range. For the SC there was a larger interfering peak observed but it was well separated from the amisulbrom peak. It could be concluded therefore that there were no problems with using a shorter column.
One laboratory remarked that TC-1 and TC-2 were analysed on Day 3 because of low repeatability on Day 2.

Three laboratories reduced the amount of sample weighed out but maintained the same concentration levels as describe in the method.
The statistical evaluation was carried out according to the CIPAC guidelines.
For TC 1 Labs 6, 7, 8, 10 and 17 were Cochran's outliers
For TC 2 Labs 7, 8 and 10 were Cochran's outliers and Lab 12 was identified as a Grubb's outlier
For WG 1 Lab 12 was identified as a Cochran's outlier
For SC 1 Lab 13 was identified as a Cochran's straggler
For SC 2 Lab 10 was identified as a Cochran's straggler
No data were excluded from the initial evaluation. When all the data were included the Horwitz criteria were met in all cases.

Mr Takahashi concluded that JAPAC propose the method is appropriate for the determination of amisulbrom in TC, WG and SC and that it is adopted as a provisional CIPAC method.

The following comments were received from the meeting:

- How important it is to use a YMC column? Could you describe what is meant by YMC column or equivalent? It would be desirable to have some sort of check for the user to do so that they can determine if their column is equivalent. Mr Takahashi replied that the YMC column is manufactured by a Japanese company. But the column is of a type (C18) that is used worldwide so he believes there are equivalent columns available.
- In the presentation you mentioned possible interferences as shown in some example chromatograms and also provided information on the sum of total inferences as a % of the active ingredient peak. Perhaps this type of chromatogram and information could be used to indicate what labs should look for to see if their column is equivalent
- 9 out of 20 labs have used the recommended column and 11 used variations. One of the labs that participated in the trial commented that they used a Zorbax C18 150 mm column with no problems at all. It should be possible to determine an equivalent column from all those used by the labs.

4.2 Nicosulfuron by Mr Ronald Chen (4903, 4904)

Mr Chen presented the results of a validation study for the **extension** of the CIPAC method 709/TC/M/3 for determination of nicosulfuron in oil dispersion (OD) formulations. The existing CIPAC method 709/TC/M/3 is suitable and validated for the determination of nicosulfuron in TC and water dispersible granules (WG)
Validation data in accordance the CIPAC guideline for a method extension were presented. The method extension met these criteria.
Mr Chen concluded that the method is appropriate and proposed that the method extension be adopted by CIPAC.
The following comments were received from the meeting:

- Can you tell me what sample preparation was used, as this will be different to that given in the CIPAC method for the solid preparation? Mr Chen replied that the sample preparation involved dissolving the formulation in acetonitrile.
- Were there any issues with the compatibility of the oil in the OD and the mobile phase of the method? Were there any issue with or limits to the solubility of the formulation? Mr Chen replied that the acetonitrile mixed well with the mineral oil.
- Do you have draft of the method extension for the OD? Mr Chen replied that this will be drafted and provided shortly.
- There will need to be an identity test included in the draft in accordance with the CIPAC style.

- Did you do any statistical analysis without the internal standard? Is the internal standard necessary? Mr Chen replied that they used an internal standard as the purpose of the study was to validate and an extension of an existing CIPAC method and the original method contains the internal standard.

4.3 and 4.5 Permethrin/Pyriproxyfen by Ms Makiko Mukumoto (4885)

Ms Mukumoto presented the results of a validation study for the **extension** of the CIPAC method 331/LN/(M2)/3 for determination of permethrin and CIPAC method 715/TC/M/3 for the determination of pyriproxyfen to include a LN formulation containing both permethrin and pyriproxyfen. The study was organised with JAPAC.

Validation data in accordance the CIPAC guideline for a method extension for permethrin were presented. The method extension met these criteria. JAPAC concluded that the method is appropriate and proposed that the permethrin method extension be adopted by CIPAC.

For pyriproxyfen some modifications to the extraction procedures were needed. A change in the extraction procedure and solvent was needed to take account of the nature of LN formulations. Heptane was used as an extraction solvent instead of acetonitrile and the samples were extracted for 45 minutes at 85-90°C.

The solvent used to prepare the internal standard solution was changed from acetonitrile to the 1-propanol. This was considered to be a minor modification.

Validation data in accordance the CIPAC guideline for a method extension for pyriproxyfen were presented. The method extension met these criteria. JAPAC concluded that the method is appropriate and proposed that the pyriproxyfen extension be adopted by CIPAC.

The following comments were received from the meeting:

- Did you try to develop a single method for both a.s. in the LN? Ms Mukumoto replied that pyriproxyfen can be determined by the CIPAC permethrin method but the separation was not satisfactory and example chromatograms showed many impurities. Therefore they concluded that it was not really appropriate to determine both together.

4.4 Pyraoxystrobin by Ms Wang Haixia (4905)

A **small scale** collaborative study on the determination of pyraoxystrobin in technical product (TC) and suspension concentrate (SC) formulations using HPLC-UV, detection at 280 nm and external standard calibration was presented at the 56th CIPAC meeting in Dublin 2012.

Following on from this there were four key questions that needed to be resolved before a full scale trial could be conducted. Ms Wang Haixia presented the progress made with the method since last year.

One issue was the need to define a standard temperature for the HPLC analysis rather than room temperature. The column temperature was fixed at 30°C.

Adjustments were made to the sample and standard preparation to address some issues notices with dissolution.

It was also questioned whether the method could resolve the *E*- and *Z*- isomers of pyraoxystrobin, if there was any *Z*-isomer present in the TC sample (the ISO common name refers to *E*-isomer only). Further data was presented to the meeting to demonstrate that the *Z*-isomer could not be detected in the TC using several different analytical techniques and that synthesis of the *Z* -isomer was not possible.

Ms Haixia proposed that a full scale trial could now be conducted.

No comments were received from the meeting.

4.5 Pyriproxyfen by Ms Makiko Mukumoto (4886, 4887)

See point 4.3

5. Reports of expert witnesses on other matters

5.1 Determining ETU in formulations containing ethylene-bis-(dithiocarbamate) by Mr Carel Diepenhorst (4899, 4900)

Mr Diepenhorst presented the results of a pilot trial and full scale study on a change of the methodology for CIPAC MT 162 for the determination of ETU in TC and formulated products. The current methodology may lead to the formation of ETU during the extraction phase; therefore the results obtained are dependent on the solvent used and the extraction time. EBDC's deocompose rapidly in protic polar solvents such as water and methanol. The rate is much lower in aprotic polar solvents such as acetone and acetonitrile. A revised method has been developed to more accurately determine the ETU content and to hopefully be applicable to all relevant EDBC product types (TC, TK, WP, WG and SC).
The revised method uses acetonitrile as extraction solvent and has standardised the extraction time. Granular samples require milling before sample extraction. An internal standard is used (*N,N,N'*-trimethylthiourea). Samples are analysed by HPLC-UV with detection at 230 nm.
A specialised column is required: Long carbon chain RP column with enhanced polar retention/separation e.g. Zorbax Bonus (polar amide group embedded in long C-chain) Agilent. Alternatives are available (Supelco RP18 Amide column, Altima HP C18 Amide column)
Initial validation data for the pilot study indicated acceptable linearity and accuracy. Repeatability for products from two different EDBCs formulation was tested and gave reasonable results. It was noted that the calibration solutions were stable for at least 1 week.

For the full scale trial 7 samples were provided: for maneb 1 x TC, 1 x WP, 1 x WG; for mancozeb 1 x TK, 1 x WP, 1 x WG, 1 x SC.
30 labs volunteered to take part; however there was only sufficient sample material for 15 labs.
The ETU content in the mancozeb WG samples was much lower than expected and so was close to the limit of the method.
Some chromatographic interference with the internal standard was noted for the SC formulation; however there was no significant difference when the results were recalculated without the internal standard.
2 participants did not report results in time, 1 did not provide information on the HPLC column used and 4 reported data from either C 8 of C18 column. Only 8 labs reported data obtained on the prescribed columns, of these the result from 2 labs were discounted as the variation between individual results, including for the standard solutions was high.
The statistical evaluation was carried out according to the CIPAC guidelines, initially with all 12 laboratories that had reported results; however in all cases the Horwitz values were not met. Therefore the statistical analysis was recalculated using results from the 6 laboratories that used the correct HPLC column.
Mr Diepenhorst concluded that:
Method is repeatable, reproducible enough, linear, specific and interference is not significant
He noted however that it is important to closely follow the method particularly in the timing of the extraction procedure i.e. analysis immediately after extraction.
Mr Diepenhorst proposed that the method be adopted as a provisional CIPAC method.

The following comments were received from the meeting:

- The retention time for ETU is quite long. And we note that the extraction time of 15 minutes has to be respected to ensure consistent results. Would it be possible to shorten the run time so that the overall method does not take too long? Mr Diepenhorst replied that it is possible to analyse for ETU without using a gradient mobile phase to shorten the run time, however experience has shown that there are interferences that can elute 6 or 7 injections later if an isocratic mobile phase is used. Also for combination products a

gradient mobile phase is needed to ensure separation from other compounds

- You based the concentration range of the method on the specification limits for ETU in each product, but we saw in practice that in some samples the level was much lower. Could you adjust the concentration of the calibration standards to give a larger range? Mr Diepenhorst replied that as linearity was demonstrated over a wide range it should be possible to determine at differ levels within this range.
- The content of ETU in the SC samples was quite low but this contradicts with the data that showed rapid ETU formation in water; can you explain why? Mr Diepenhorst replied that there is a stabilizer in the SC formulation that prevents further ETU formation.
- Do you expect to receive results from the labs that did not finish the analytical work in time? Mr Diepenhorst replied that he hoped this would be the case.
- Is this method applicable to metiram products? Mr Diepenhorst replied that unfortunately this was not tested as he did not know the specification limits for metiram or the composition of the formulated products to predict if there would be any issues. In addition as the extraction time needs to be respected conducting the analysis for 7 samples takes 1 day so adding more samples would have made the trial take too long. In theory the method should work.
  Would it be possible to extract the samples using an ultrasonic bath instead of a magnetic stirrer? Mr Diepenhorst replied that the energy put into an extract by using an ultrasonic bath could stimulate the production of ETU therefore stirring was the preferred procedure

5.2 Deltamethrin enantioselective identity test by Mr Gerhard Krautstrunk (4907, 4908)

Mr Krautstrunk presented the peer validation of a new chiral analytical method for deltamethrin. The deltamethrin FAO specification defines a minimum purity for the technical active ingredient as a single isomer. The method is designed to separate all eight deltamethrin stereoisomers and is suitable for the determination of the ratio of deltamethrin and its enantiomer in combination with the chemical purity determined using method CIPAC333/TC/M2.
The method uses HPLC with UV detection, and requires a Phenomenex LUX Cellulose-1 column and pre-column. Use of the pre-column is strongly recommended to protect the main column and to improve the resolution.
Two labs took part in the peer validation and two samples of TC were used.
Mr Krautstrunk proposed that the method be adopted by CIPAC and also that current FAO specifications were revised to reflect the new method.

The following comments were received from the meeting:

- When you look at assignment of the peaks you can see it's not easy to get separation. Did you considerer the temperature of the columns as resolution improves with lower temperatures for chiral columns? Mr Krautstrunk replied that the temperature used was 25°C this was considered to strike the right balance between good resolution and avoiding impracticalities of lowering the temperature below "ambient".
- If this is the case should a temperature range be specified or is it sufficient to just state or 25°C? Mr Krautstrunk replied that he would need to check the raw data for the method development to confirm this point.
- The assumption is made that the UV absorption is the same for all stereoisomers – could it be made clear in the method that this is the general assumption but that we do not know for sure. Mr Krautstrunk replied that they had only validated the 2 enantiomers of concern so the other information provided for the other isomer pairs is only qualitative rather than quantitative. This will be made clear in the method.

Mr Gerhard Krautstrunk then made a proposal that the deltamethrin CIPAC methods should be revised and requested that the other data owners of deltamethrin TC and products contacted him to begin discussions.

The following comments were received from the meeting:

- The official version of the method for coated LNs is in Handbook M and was provided several years ago; later an improvement developed using a calibration curve + internal standard as used for the deltamethrin methods for the 2 other LN types. This method is not published in a handbook but is available on the website. The web version of the method is being used by labs. This also needs to be considered. Mr Krautstrunk replied that his proposals were presented on the basis of what is published in the handbook but agreed that there will also need to be a consideration of those methods on the website.

5.3 Degree of dissolution and solution stability by Mr Franz Wochner (4891, 4892)

Mr Wochner presented a proposal for a method extension to CIPAC method MT 179, dissolution degree and solution stability to increase the scope of the method. Currently the scope of the method is only applicable for water soluble granules. The proposal is to change the scope to solid, water soluble formulations.

Other proposals included were to increase the standing time to 24 h and to harmonise the general procedures and temperatures in line with other CIAPC methods.

It was noted that the size of the sieve states in the published method is no longer available (i.e. the diameter of the sieve) therefore an alterative was proposed. The mesh size was not changed. As the changes did not make a significant alternation to the original method no data were provided to support the proposals.

Mr Wochner proposed that the extended version be adopted by CIPAC.

The following comments were received from the meeting:

- Can you clarify what the original standing time that was in the method? Mr Wochner replied that the original standing time was 16 hours. This timing can limit the flexibility of the laboratories conducting the test therefore a proposal of 24 h is made.
- At what temperature is the method currently conducted at? Mr Wochner replied that the test is currently conducted at 25°C and they have not proposed to change this.

5.4 Disintegration of tablets by Mr Bruno Patrian (4893, 4894)

Mr Patrian on behalf of DAPF presented the proposal and validation for a new MT method for disintegration of tablets. The method is designed to determine the completeness of disintegration of tablets which are dissolved or dispersed in water independent of the use rate. The method is designed for, but not limited to, the disintegration of effervescent tablets.

One entire tablet is added to a defined volume of CIPAC standard water D and mixed by gentle stirring for the specified disintegration time of the tablet. The suspension is then passed through a 2000 μm sieve. The absence of a residue on the screen indicates complete disintegration of the tablet.

12 laboratories particpaited and 3 x ST samples from different suppliers were tested.

For the 1st sample tested no outliers were identified. For the 2nd sample, one outlier was identified, however the lab concerned noted that the appearance of the tablets was unusual and that during the test the tablet floated with no evolution of gas. It was concluded that the package of the tablet or the tablet itself had been damaged during shipping.

For the 3rd sample, 3 labs reported residue on the sieve. These 3 labs received the samples at least 2 months later than all other participants. Therefore it was concluded that the repackaging and the long transport (high temperature, humidity) had a significant influence of the results and are responsible for the residues observed.

Mr Patrian concluded that:

- Water temperature, speed of the stirrer and stirrer positions had no significant effect on the results

- storage problems during the transport of the tablets were detected by the new method: demonstrating its purpose and usability
- The collaborative test demonstrates the applicability and robustness of the proposed method to determine the completeness of the disintegration of tablets.

DAPF propose the method is adopted as a provisional method.
No comments were received from the meeting.

5.5 Wash resistance index of LN: validation of the new CIPAC washing method by Mr Olivier Pigeon (4909)

Mr Pigeon provided a summary of the work conducted to date on the validation of the LN and the conclusions of the previous CIPAC meetings. At the CIPAC meeting in 2012 the method was adopted as provisional, however CIPAC concluded that the method should remain provisional until further validation is provided to give a sufficiently large data set.
Mr Pigeon presented additional validation data for 8 different LN products in order to validate the method further and to allow revision of the relevant interim WHO specifications.
The additional data provided showed:

- Acceptable repeatability and/or reproducibility
- Results consistent with those of the CIPAC Small Scale Collaborative Trial or with those of the WHOPES Phase I studies (WHO method)
- Results consistent with / supporting the limits of the WHO specifications

Mr Pigeon proposed that the method be adopted as a full method, subject to some minor editorial changes.
The following comments were received from the meeting:

- One of the editorial changes is to remove one of the sources of the washing agent. Could you explain further? Mr Pigeon replied one of the two sources is not available anymore. In addition although the chemical composition seems to be the same the two were clearly different as there was a difference in pH between the two. Even though they were the same substance they had 2 CAS numbers as they were prepared in different ways which lead to the differences. Mr Pigeon states that it had already been agreed not to include the name of the supplier of the washing agent in the method but he would also propose to include a note to check the pH of the washing agent before starting the work.

5.6 Relevant impurity toluene in formulations by Mr Bruno Patrian (4895, 4896)

Mr Patrian on behalf of DAPA presented the results of a small scale trial for the determination of toluene as a relevant impurity in formulations. The method uses headspace GC-FID with a J&W DB-624 column.
8 laboratories participated in the small scale trial. 5 samples were tested: 2 x EC, 1 x FS, 1 X SC and 1x SC. Many different headspace systems were used.
One laboratory only sent data from 1 day so they were excluded from the statistical analysis. All other data were initially used.
The Horwitz criteria were not met for any samples initially. After removal of outliers RSD(R) of 1 sample meets the Horwitz criterion, RSD(R) of 3 samples is close to Horwitz criterion and RSD(R) of 1 sample is far above Horwitz criterion.
The reasons for deviations are not obvious, however given that the method is a headspace technique some variations are to be expected. The DAPA-group concluded that the method is able to quantify toluene in many formulation types and can be used with different headspace-sampling devices and FID or MS detection.
Mr Patrian proposed that the method could go for a full scale trial.
The following comments were received from the meeting:

- Why is toluene a relevant impurity? Mr Patrian replied that in the EU toluene is considered a relevant impurity even if the limits are low. Therefore a monitoring method

is needed in the EU. For FAO & WHO specifications the way of describing an impurity as relevant is not the same as in the EU.

- Would it be possible to do the method without headspace equipment? Mr Patrian replied that at the beginning they had tried using a classical GC technique it was not sure if this would work for all the different formulating types. Therefore they tried headspace techniques.
- Have you tried SPME rather then headspace? It may be easier to do with SPME. Mr Patrian replied that the group did not have experience with this technique but it may be possible.
- Could you clarify how much sample you need to use and what the concentration is in the final vial? Mr Patrian replied that the idea is to use a sample size that gives you a concentration of 20 mg toluene in the final vial. The calculation is based on the concentration of the active ingredient in the formulation and the maximum amount of toluene permitted in the active ingredient.

5.7 Determination of quaternary ammonium compounds in disinfectant formulations by Mr Adrian Burns (4910)

Mr Burns discussed the results of a collaborative study that has been conducted in conjunction with CIPAC. A poster was also presented at the symposium.
Quaternary ammonium compounds (QUATs) have many other uses but are mainly used as antimicrobial disinfectants.
A previous AOAC method was a titrimetric method using dichloromethane, with a colour change as the end point. A new potentiometric method with an ionic surfactant electrode has been developed and has been collaboratively tested.
Hydrochloric acid and alcohols can interfere with the electrode which means that the method may not be applicable to some product types. Also electrodes are expensive which may limit the use of the new method - for the collaborative study the manufacture provided the electrode to the laboratories.
12 laboratories participated in the study and 7 samples were tested covering concentrations of QUAT from 0.5-30%. None of the samples contained hydrochloric acid or alcohols.
All results for the collaborative study are within the Horwitz value. There were no outliers.

Mr Burns proposed that the method is adopted as a provisional CIPAC method.
No comments were received from the meeting.

6. Replacement of obsolete methods

6.1 Comments on existing methods

Mr Ramesh presented some proposals to correct CIPAC 288/TC (M)
A couple of technical errors were noted in terms of nomenclature. In addition he proposed changing the internal standard name to the correct IUPAC name as there are inconsistencies throughout the method.

Mr Ramesh also presented proposals to correct CIPAC 494
A couple of technical errors were noted in terms of nomenclature. In addition he proposed changing the internal standard name to the correct IUPAC name as there are inconsistencies throughout the method.
Mr Bura remarked that comments and corrections with CIPAC methods are always welcomed and that errata can be made on the web site. He asked the meeting if there were any other comments on existing methods.
Mr Bascou informed the meeting that they had note for the clothianidin (CIPAC 738) methods,

in the method for suspensibility there is a paragraph describes the usual suspensibility test for SCs when in fact clothianidin is really an FS. He proposed that a correction is needed to reflect this and offered to provide a proposal to the secretary in writing.

6.2 Harmonisation of the temperatures of the MT methods from 30°C to 25 +/- 5°C
During the JMPS a proposal was made to consider revise and harmonise the temperatures in CIPAC methods. Mr Bura requested comments about how this change in temperature would affect laboratories and/or if there was any experience of where this may not be appropriate. Comments should be sent in writing to CIPAC.

Mr Bura declared the open meeting closed.

7. Minutes of the 56th meeting (4880/P)
The minutes were circulated to members by e-mail. No corrections or comments were received therefore the minutes were accepted.

8. Secretary's report (4881/P)
Mr László Bura presented the Secretary's report for the period from the 56th CIPAC meeting held in Dublin, covering the attendance, number of trials conducted, the decisions taken concerning the methods and the election of correspondents and members of CIPAC.
No comments were received.

9. Discussion of individual compounds

454: alpha-cypermethrin (wash method)
At the 56th meeting, 2012 in Ireland it was agreed that the soap washing method for the determination of remaining active ingredient concentration remains a tentative MT method because of the ongoing general work on LN washing method(s).
The meeting agreed that this method is considered no longer relevant as a new wash method has been adopted.

570: Chlorfenapyr
At the 56th meeting, 2012 in Ireland the method was adopted as provisional. No further comments were received
The method can be promoted to a **full CIPAC method**.

653: Cyazofamid
At the 56th meeting, 2012 in Ireland the method was adopted as **provisional** with a proposal to the company to include a footnote about a column flush gradient programme.
The reversed phase HPLC method (CIPAC/4833) for the determination of cyazofamid in TC and SC formulations was accepted as a **full** CIPAC method with the amendments in the description of the method concerning the gradient flush after the elution of the a.i.

333: Deltamethrin (LN extension, wash method)
At the 56th meeting, 2012 in Ireland it was agreed that the soap washing method for the determination of remaining active ingredient concentration remains a tentative MT method because of the ongoing general work on LN washing method(s).
The meeting agreed that this method is considered no longer relevant as a new wash method has been adopted.

The extension of the scope of CIPAC method 333 (CIPAC/4673) for the determination of the total content of deltamethrin in incorporated PE LN formulations remains as a **provisional** CIPAC method. The extension of the scope of CIPAC method CIPAC/4673 333/LN/(M)/3 (CIPAC/4797)for the determination of the total content of deltamethrin in incorporated polypropylene LN formulations was accepted as a **full** CIPAC method, with the unequivocal definition of the scope.
CIPAC will, in cooperation with Bayer CS, review the data and clarify the situation.

595: Flazasulfuron
At the 56th meeting, 2012 in Ireland the method was adopted as provisional. No further comments were received
The method can be promoted to a **full CIPAC method**.

585: Fosthiazate
At the 56th meeting, 2012 in Ireland the method was adopted as provisional subject to the additional notes needed in the method, and to the provision of the statistical analysis with and without the internal standard.
The reversed phase HPLC method (CIPAC/4829) for the determination of fosthiazate in TC and GR formulations was accepted as a **full** CIPAC method with the necessary amendments in the description of the method and the note concerning the diastereomers.

767: 1-MCP
At the 54th meeting, 2010 in Slovenia it was agreed that the method should remain provisional as further feedback was required from laboratories using the method. The method is difficult and uses specific equipment - therefore there needs to be more experience gained. The method is not routinely used and for that reason until more experience is gained it should remain provisional. No new information has been received there the meeting agreed the situation remains as stands. The method **remains provisional**. The meeting also agreed that discussion of 1-MCP should be removed from the agenda until further data are received.

331: Permethrin (LN extension, wash method)
At the 56th meeting, 2012 in Ireland it was agreed that the soap washing method for the determination of remaining active ingredient concentration remains a tentative MT method because of the ongoing general work on LN washing method(s).
The meeting agreed that this method is considered no longer relevant as a new wash method has been adopted.
The extension of the scope (CIPAC/4841) of CIPAC method 331/LN/M/3 for the determination of the total content of permethrin in long lasting insecticidal mosquito net (incorporated type) containing permethrin and PBO was accepted as a **full** CIPAC method.

636: Spinosad
At the 56th meeting, 2012 in Ireland the method was adopted as provisional. No further comments were received.
The method can be promoted to a **full CIPAC method**.

637: Thiamethoxam
At the 56th meeting, 2012 in Ireland the method was adopted as provisional. No further comments were received
The method can be promoted to a **full CIPAC method**.

Extension of CIPAC MT 46.3 for the accelerated storage stability of LN
At the 55th meeting, 2011 in China the method was adopted as tentative with the requirement of some further work on validating the method.
No validation data has been received as the focus for the "LN group" has been on finalising and collaboratively testing the washing method for LNs. Now that the washing method has been finalised, work to validate this method further is on-going.
The method remains **tentative.**

789 Amisulbrom
The meeting discussed the comments received during the open meeting. Written remarks from Mr Martijn were also received.
10 different types of column were used – the meeting considered how far a laboratory can deviate from the method that is proposed. It was agreed that to such problems in the future the procedure should be written in a way that makes it clear when deviations are necessary/acceptable.
The meeting noted that even when there were 10 types of column used, the results were acceptable. This demonstrates the ruggedness of the method.
The meeting agreed the method could be **adopted as provisional**

709 Nicosulfuron
The meeting discussed the comments received during the open meeting.
The meeting discussed further the choice of solvent used for sample preparation. The miscibility of acetonitrile with the OD formulation was questioned, as it was mineral oil based. It was noted that some OD formulations are vegetable oil based and miscibility of acetonitrile vegetable oil is better than for mineral oils. The meeting agreed further clarification is needed. Based on the experience of one laboratory THF could be used as an alternative. The meeting considered that a bringing study comparing MeCN and THF might be useful.
The meeting also agreed that there may be a need to change the suspensibility method for nicosulfuron.
Information on the additional identity test and a description of the method need to be provided.
The meeting agreed that pending the comparison of extraction solvents and provision of the additional information the extension can be adopted **as provisional**.

331 Permethrin
The meeting discussed the comments received during the open meeting.
It was confirmed (CIPAC/4885) that the existing method for the determination of permethrin in LN formulations (331/LN/(M2)/3) is applicable for the long lasting insecticidal mosquito net (incorporated type) containing permethrin and pyriproxyfen

715 Pyriproxyfen
The extension of the scope (CIPAC/4887) of CIPAC method 715/TC/M/2 for the determination of the pyriproxyfen content of a the long lasting insecticidal mosquito net (incorporated type) (LN) containing permethrin and pyriproxyfen was accepted as a **provisional** CIPAC method

964 Pyraoxystrobin
The meeting agreed that the company had made every effort to demonstrate that the pyraoxystrobin was present as predominantly *E*-isomer. The meeting agreed that a full scale trial could be conducted.

Determining ETU in formulations containing ethylene-bis-(dithiocarbamate)
The meeting considered if there was enough data generated. It was noted that the method is for a relevant impurity, and that CIPAC do not require data from 8 labs for a relevant impurity

method.
One member that took part in the study and had used the right column commented that the problem with the stability of the samples means the samples have to be determined one at a time.
The meeting agreed that the method was progress on previous methods and agreed that the method could **be adopted.**

Deltamethrin- enantioselective identity test
The meeting noted that the column proposed for the new method is expensive and this may cause problems. It was also noted that the column used in the old method was also an expensive column.
It was noted that even though eight peaks are separated (all the enantiomers) only 2 are of interest and relate to deltamethrin
The meeting agreed that the method can **be adopted** as a quantitative stereo specific identity method for deltamethrin; however it has to be made clear that the older method is no longer supported.
The meeting agreed with the proposals to harmonise and update the deltamethrin methods in conjunction with other data owners. It was noted that the coated LN method that is published in the Handbook is not performing as well as the modified version on the web. It would be useful to discuss with Vestergaard before taking any action. CIPAC agreed to contact.

Degree of dissolution and solution stability
The meeting agreed that the extension could be accepted **as provisional**, becoming MT 179.1

Disintegration of tablets
The meeting agreed that the method could be accepted **as provisional**.

Wash resistance index of LN: validation of the new CIPAC washing method
The meeting noted that the new data that was provided shows the method seems to be working well. The meeting considered if there would be problems with the availability of the washing agent in the future and agreed it should be checked if there are other sources available.
The meeting agreed that it would be useful to allocate an MT number to the wash method for ease of reference.
Pending confirmation that the reagent is more widely available, and the other editorial changes proposed the method can be adopted **as full.**

Relevant impurity toluene in formulations
The meeting agreed that a full scale trial could be conducted, but noted there may not be enough labs that have the necessary headspace analysis equipment.

Determination of quaternary ammonium compounds in disinfectant formulations
The meeting felt it was difficult to discuss the method without the full set of data available. It was a full study and conducted well. It was felt that it was unfair to wait for a year because the results seem very good. Scientifically there are probably no issues but the meeting agreed that this is politically sensitive issue and did not want to cause embarrassment to CIPAC, its members or any other organisation by discussing the method further.

The weight of opinion of the meeting was in favour of postponement.

10. Matters related to FAO and WHO specifications
Dr Müller informed the meeting that as in previous years comments have been made by the SEG on proposed revisions to the FAO/WHO Manual. There were typographical issues and some

technical issues. These were considered by the JMPS and the amendments will be published on the FAO and WHO websites.

11 Any other business
None

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

Sonia Tessier	László Bura
Assist. Secretary	Secretary