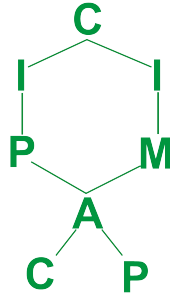




Food and Agriculture
Organization of the
United Nations



World Health
Organization

First joint CIPAC/FAO/WHO Open Meeting (48th CIPAC Meeting and 3rd JMPS Meeting)

2004

FIRST JOINT CIPAC/FAO/WHO OPEN MEETING (48th CIPAC Meeting and 3rd JMPS Meeting)

Czech Republic, Brno, 8 June 2004

1. Opening and welcome

Dr Müller, CIPAC Chairman, opened the meeting and welcomed participants to the first Joint CIPAC/FAO/WHO Meeting, incorporating the 48th CIPAC Meeting and 3rd Joint Meeting on Pesticide Specifications (JMPS).

Mr. Zdeněk Trnka, Head of the Division of Plant Commodities of the Ministry of Agriculture of the Czech Republic welcomed CIPAC and JMPS participants to Brno.

Dr Vaagt, FAO Joint Secretary of JMPS, in his introductory remarks thanked the Ministry of Agriculture of the Czech Republic and especially Mr. Jindrich Foltýn and his team as the main organizer and initiator of the JMPS and CIPAC meetings here in Brno. He drew attention to changes and progress in the previous 12 months.

- EC directive 91/414, which is now the legal guiding document for pesticide authorisation and management in 25 European countries, includes in its Annex 6 the requirement for compliance with FAO pesticide specifications.
- The Working Group on Plant Protection Products of the EU had adopted the FAO/WHO procedure for the determination of equivalence, to be incorporated into the 91/414 amendments.
- The Rotterdam Convention on the Prior Informed Consent (PIC) Procedure for Certain Hazardous Chemicals and Pesticides in International Trade became official on 24 February 2004. The Stockholm Convention on Persistent Organic Pollutants (POPs) similarly became official on 17 May 2004. FAO Pesticide specifications have already proved to be a supportive element for the implementation of these Conventions. Examples include the FAO specifications for maleic hydrazide as an alternative control to the PIC Procedure and the reduction of HCB impurity levels in chlorothalonil specifications (HCB, hexachlorobenzene, is a POPs chemical). Dr Vaagt noted that the Czech Republic had already ratified these two conventions.
- The Revised Version of the International Code of Conduct on the Distribution and Use of Pesticides includes various references to the specifications and supports the Rotterdam and Stockholm Conventions.

Dr Zaim, WHO Joint Secretary of JMPS, in his introductory remarks thanked the Division of Plant Protection of the Ministry of Agriculture of the Czech Republic, and especially Mr

Jindrich Foltýn, for their excellent support in facilitating the meeting, and for their warm hospitality.

Dr Zaim expressed his sincere thanks to the JMPS Panel Members for their invaluable technical support to the work of the two organizations for development of international standards for quality control of pesticides. He also extended his thanks to CIPAC for their support and assistance in development of test methods in support of the pesticide specifications.

Dr Zaim noted that the promotion of availability of quality pesticide products is a priority activity for WHO and that they are very pleased to note the interest shown by the Member States as well as industry in the FAO/WHO harmonized procedures for development of pesticide specifications. He emphasized the need for continued and close collaboration with industry and Member Countries on promoting effective management of public health pesticides, including their quality control.

2. Arrangements for chairmanship and appointment of rapporteurs

Dr Müller, CIPAC Chairman, advised the meeting of the arrangements for this joint open meeting. Chairmanship would rotate year by year beginning with CIPAC in 2004. Rapporteurs were appointed for the meeting: Mr Bura for CIPAC and Mr Hamilton for JMPS.

3. Adoption of the agenda

The agenda was accepted with the following modifications and amendments:
Add 9.3. Guidelines for TC/TK.

Treat items 11 and 12 together, i.e. the item becomes: *National and technical reports regarding CIPAC activities.*

Add:

- 14.1. Publication of test methods for impurities.
- 14.2. Method extension concept for impurities.
- 14.3. Clarification of procedure and timelines for adoption and publication of specifications following a JMPS review.
- 14.4. Report on closed Joint Meeting on Pesticide Specifications (JMPS).

4. Summary record of previous meetings

4.1 *47th CIPAC Meeting Technical Commission (12/13 June 2003, Bucharest, Romania)*
No comments. The Minutes of the 47th Annual Meeting as circulated by Mr L. Bura were accepted as correct without amendments.

4.2 *Second JMPS Open Meeting (10 June 2003, Bucharest, Romania)*
No comments other than editorial. The Summary Records of the 2nd JMPS were summarized by Mr J. Pim and were accepted as correct.

5. Summary of actions taken after the 47th CIPAC and 2nd JMPS meetings

5.1 CIPAC

Dr. Müller summarized relevant CIPAC activities:

Eight information sheets had been sent out and the results will be discussed during this year's meeting. Several pilot studies are under way. In the meantime, the latest CIPAC Handbook, K, was published. It contains 22 new or extended methods for determination of pesticides and 6 new or extended MTs.

Last year a considerable improvement of the CIPAC web site was realised facilitating access by potential users of the methods and ordering of Handbooks. Furthermore, CIPAC now accepts credit card payment, which significantly enhances the processing of orders.

The CIPAC handbooks J and K were prepared as a CD-ROM, with searching facilities, and are offered as a package with an appropriate cost to cover the publication and handling charges. The preparation of the CD-ROM was achieved without delaying the publication of Handbook K.

For the first time, CIPAC offered to provide adopted but not yet published methods under the so called prepublication scheme (see www.cipac.org/prepublished methods). They are available in a non-edited form. The method publication in a handbook no longer delays the publication of pesticide specifications.

5.2 FAO

Dr Vaagt summarized relevant FAO activities.

- The Manual on Development and Use of FAO and WHO Specifications for Pesticides has been translated into Spanish. French and Chinese versions will be available soon.
- JMPS has been established as a statutory body of FAO.
- Demand has risen to assist regulatory authorities and others with the procedure for equivalence determinations. Information has been disseminated at workshops, seminars and presentations:
 - July 2003: Workshop in Belize.
 - August 2003: Seminar in Kuala Lumpur, Malaysia.
 - October 2003: Seminar for representatives from CropLife Latin America, in Miami, USA.
 - November 2003: Presentation of the “new” procedure for the development of pesticide specifications during the International Conference of Pesticide Registration and Management, Beijing, People’s Republic of China.
 - December 2003: Presentation in Tokyo.
 - May 2004: Open presentation in Quito, Ecuador.

5.3 WHO

In order to assist the Member States in effective and sound management of public health pesticides, including their quality control, Dr Zaim summarized major activities carried out by WHO since the previous JMPS meeting.

- Held WHO Regional Workshops on the management of public health pesticides in Amman (December 2003) and Bangkok (April 2004) to develop regional strategies and national action plans.
- Initiated the development of "Quality control of pesticide products - Guidelines for national laboratories."
- Established a regional centre for quality control of pesticide in Africa, situated in Pretoria.

6. Technical liaison with other organizations

6.1 AOAC-International

Dr A. Hanks summarized the activities of the Committee on Pesticides and Disinfectant Formulations. It was pointed out that the fee required for validation studies has been a major concern and deterrent for those seeking collaborative validation. Methods for determining several herbicide active substances are continuing to be developed and improved, some with LC-MS technique. The study on acetanilide derived herbicides, glyphosate and pendimethalin formulations is continued and on hydrazine and maleic hydrazide will start.

6.2 CropLife International and European Crop Protection Association (ECPA)

Dr. T. Woods mentioned that CropLife International should not be abbreviated to CLI because CLI is already used for CropLife India.

He introduced and welcomed the representative of ECPA Secretariat, Ms D. Obierzynska and also recognized the the work of DAPF in method development and that of the specifications group.

6.3 American Society for Testing and Materials (ASTM)

Dr A. Viets made a short presentation on how ASTM is cooperating with CIPAC with respect to physical test methods.

6.4 European Crop Care Association (ECCA)

D. van Hoogstraten, representing many generic manufacturers from EU, presented the activities ECCA proposed independently last year to the EU.

6.5 United Nations Industrial Development Organization (UNIDO)

Mr K. Ziller presented a report prepared by Mr. B.Sugavanam:

UNIDO in association with Nanshen Pesticide Formulation Centre and supported by the Government of China organized a workshop on "Cleaner Production in Agrochemical Industry -Seed Dressing Technology". The workshop dealt with the various developments of formulation technology the history and the present status of seed treatment technology, and technology transfer to less developed regions of Southwest and Northwest China. It also dealt with the seed pelleting and seed coating technology which would be very valuable for China. Papers also dealt with import/export of pesticides in China and the registration aspects in China for pesticides. The workshop was attended by more than 20 countries from Asia and Europe. The message from the workshop was to promote safe and effective seed dressing formulations in developing countries, proper application methods with suitable and well maintained machines and good quality control. It also stressed the importance of neem trees for rural and forestry development with potential in pest control and other areas.

6.6 International Union of Pure and Applied Chemistry (IUPAC)

Mr D Hamilton reported that IUPAC information is available at www.iupac.org. Recently published papers and progress reports on current projects of the *IUPAC ADVISORY COMMITTEE ON CROP PROTECTION CHEMISTRY* are available from the internet. An IUPAC workshop on crop protection chemistry in Latin America is scheduled for 14-17 Feb 2005 in Costa Rica and the next IUPAC International Congress on Pesticide Chemistry will be held in Kobe, Japan from 6-11 Aug 2006.

6.7 European Commission (EC)

Nobody from the EC attended the meeting.

6.8 European Food Safety Authority (EFSA)

Mr R. Hänel gave a description of the history of the establishment of EFSA and a short description of the organizational structure. The EFSA was established because of a succession of food scares in Europe (e.g. BSE and dioxins), the creation of national food safety agencies and dissension within the EU over risk assessments. EFSA will provide scientific advice in the form of risk assessments and risk communication, but it is not responsible for risk management decisions. EFSA's web page: www.efsa.eu.int

6.9 Organization for Economic Co-operation and Development (OECD)

Dr. G. Vaagt informed the meeting that the OECD is interested in maintaining an information exchange with CIPAC and JMPS.

6.10 International Programme on Chemical Safety (IPCS)

Dr. M. Zaim reported that Dr. A. Aitio will represent IPCS.

6.11 International Organization for Standardization (ISO)

No representative.

7. Status, review and publication of CIPAC methods

Dr. M. Müller informed the meeting about the new guideline proposal on “method migration,” which will be discussed during the CIPAC technical meeting. The aim is to withdraw the old CIPAC methods, which are not in line with the requirements of the guidelines on chromatographic methods or which are otherwise obsolete, without leaving a data gap. CIPAC will try to speed up the process.

8. Review and publication of FAO and WHO specifications for pesticides**8.1 Status of FAO Specifications**

Dr Vaagt provided a progress report on previously evaluated pesticides.

Manufacturer	Product	FAO spec	Status
Agro Chemie	Difluvidazin (Flufenzine) TC, TK, SC	new	Specification and evaluation report published
Bayer CropScience	Cyfluthrin TC, WP, EW	new	In progress
	Imidacloprid TC, DT, FS, GR, PR, SC, SL, UL, WG, WS	new	In progress
	Iprodione TC, WP, WG, SC	new	In progress
Chlormequat Task Force (Nufarm, BASF, Ciba Specialty Chemicals, UCB SA)	Chlormequat chloride TK, SL	new	In progress
Drexel, Crompton, Fair Products	Maleic Hydrazide TC, SL, SG, SP, PD	new	Evaluation report published- publication of specifications subject to validation of analytical methods
DuPont	Hexazinone TC, WG, SP, GR	new	Evaluation report published – publication of specifications subject to validation of methods for the relevant impurity
	Chlorsulfuron TC, WP, WG	new	Ready for publication
Nufarm	Butralin	new	Was rescheduled for 2004, but withdrawn
Syngenta	Paraquat TC, TK, SG, SL	new	Specification (TK) and evaluation report published. Publication of formulation specifications is subject to validation of methods for impurities.
Trifolio M, EID Parry Fortune Biotech	Azadirachtin EC, TK	new	In progress, on 2004 agenda.

Dr Woods (CropLife International) noted that specifications were being held up by method requirements for relevant impurities and requested notification as early as possible about decisions on relevant impurities. Ideally, validation data should be provided at the time of data submission but companies could not always anticipate JMPS decisions on relevant impurities.

8.2 Status of WHO Specifications

Dr Zaim (WHO) reported that data submissions were received for 14 compounds in 2002-3, 7 of which were for joint FAO/WHO specifications and 7 specifically as WHO specifications. WHO aims to publish evaluation reports in the same year as the JMPS evaluation is completed.

The progress to publication is summarized in the tables below.

JMPS year	Compound	Proposer	WHO publication of evaluation report	specification
2002	d-allethrin	Sumitomo	yes	yes
2002	d-phenothrin	Sumitomo	yes	
2002	prallethrin	Sumitomo	yes	
2002	transfluthrin	Bayer Environmental Science		
2003	esbiothrin	Sumitomo		
2003	bioallethrin	Sumitomo		
2003	trans-cyphenothrin	Sumitomo		

8.3 Status of Joint FAO/WHO Specifications

JMPS year	Compound	Proposer	WHO publication of evaluation report	specification
2002	niclosamide	Bayer	yes	yes
2002	chlorpyrifos	Dow AgroSciences, Makhteshim	yes	
2003	lambda-cyhalothrin	Syngenta	yes	yes
2003	cyfluthrin	Bayer CropScience		
2003	propoxur	Bayer CropScience		
2003	novaluron	Makhteshim	yes	
2003	malathion	Cheminova		

9. Proposed new/amended specification guidelines

9.1 [Guidelines for mixed formulations ZC, ZE and ZW](#)

New codes and specifications for mixed formulations of CS and SC (ZC), CS and SE (ZE) and of CS and EW (ZW) were introduced at the 2003 meeting. Following the suggestion of Mr Hill, a clause for stability during freeze-thaw cycles (required for CS formulations) was introduced, which encompassed the requirement for storage stability at 0°C (required for liquid formulations).

After the introduction of this additional clause, the new guidelines were accepted by the meeting and are attached in its final version as [Annex 1](#). FAO and WHO will publish these new guidelines on their website.

9.2 Guidelines for long lasting insecticidal nets

The draft guidelines specifications for long-lasting insecticidal nets (LN) was presented by industry. Mr Hill requested the inclusion of "free from damage (such as splitting or tearing)" in the description. After the introduction of this additional clause the new guidelines were accepted by the meeting and are attached in its final version as annex 2.

9.3 Guidelines for TK

Dr Grohs (Bayer CropScience) suggested that the concept and definition of TK could be extended to include salts or complexes of the active ingredient as well as just the active ingredient with residual moisture or solvents from the production process. The definition of TK could be amended by adding the words "or salt or complex of the active ingredient."

Proposed amended definition (underlined words are added).

TK Technical concentrate. A material resulting from a manufacturing process comprising the active ingredient, or salt or complex of the active ingredient, together with associated impurities. This may contain small amounts of necessary additives and appropriate diluents. A note would be added at the end of paragraph 5.2.2.2 of TK specifications stating. " (Note 2): In the case of salts or complexes only a minimum content (not less than.... g/kg) needs to be declared."

The Meeting was concerned about the consequences, which were not all immediately clear, of extending the meaning of TK and, at least at this time, declined to make a change. Mr Hill proposed alternative definitions of TC and TK and industry representatives agreed to consider them.

10. Proposed new/extended CIPAC analytical and physical test methods and CIPAC workplan for 2004/5

Dr. M. Müller informed the meeting that there are several MT and analytical methods under revision and development.

11 and 12. National and technical reports regarding CIPAC activities

The following reports were presented:

Argentina – H. di Loretto in the name of SENASA
Australia – P. Sethi
Belgium – M. Galoux
Brasil – M. Fuentes Piedade
Czech Republik – J. Foltýn
Cyprus - written report by A. Kashouli-Kouppari
Denmark – T. Krongaard
El Salvador – E. de Aguila
France – A. Venant
Germany – G. Menschel
Greece – A. Hourdaki
Hungary – L. Bura
India – G. Mukherjee
Ireland – J. Garvey
Italy –R. Dommarco
Japan - T. Fujita
Korea – K. Oh
Netherlands – E. van der Wal
P.R.China – J. Ying
Romania – T. Iuraşcu
Slovenia – A. Gregorčič
Slovakia – written report by J. Schlosserova
Spain – L. Manso
South Africa – S. Marais
Switzerland – M. Müller
China, Province of Taiwan – S.-S. Wong
Thailand – N.Tayapuch
Ukraine – V. Chmil
United Kingdom – R. Fussell
USA – V. Goodwin

The following national laboratories reported on their participation in CIPAC collaborative studies and provided a summary of quality control analysis carried out on pesticide samples during 2003-2004:

Region	Reporting laboratory	No. of samples tested	Non-compliance	
			No.	%
Africa	South Africa	12	3	25
Americas	El Salvador	662	83	13
	Indiana, USA	262	21	8
Europe	Czech Republic	29	4	14
	Cyprus	84	6	7
	Denmark	45	7	16
	France	80	16	20
	Germany	13	1	8
	Greece	158	5	3
	Hungary	1585	81	5
	Italy	298	9	3
	Romania	2494	50	2
	Slovakia	197	9	5
	Slovenia	11	2	18
	Spain	518	48	9
	UK	102	10	10
Asia	China, Taiwan	1240	250	20
	China	710	130	18
	Thailand	4589	100	2
Total		13089	835	6.4

13. FAO/WHO priority list for the development of specifications (2005-2007)

Dr Vaagt provided the priority list of specifications for evaluation in 2005 and a tentative list for 2006. It was suggested that the permethrin/S-bioallethrin combination is not used in agriculture and may not require an FAO specification.

Compounds to be evaluated by JMPR (Joint Meeting on Pesticide Residues) in 2006 should have been previously evaluated by JMPS. However, national governments have tended to treat residue issues as the priority, so the 2006 requirement will be treated as voluntary.

Products	Proposer(s)
2005	
FAO	
Clofentezine TC, SC	Makhteshim
Chlorothalonil	Caffaro SpA, Vischim Srl, SDS Biotech K.K.
Copper, cupric hydroxide and oxychloride (to include copper calcium oxychloride), Bordeaux mixture, tribasic copper sulphate and cupric oxide.	European Union Copper Task Force
Diquat dibromide, TC, SL	Syngenta
Ethofumesate TK, SC, EC, SE, OD	Bayer
Nicosulfuron TC, WG	Dupont
Pendimethalin TC, TK, EC	Industria Prodotti Chimici
Rimsulfuron TC, WG	Dupont
WHO:	
IR3535	Merck
Permethrin long-lasting insecticidal net	Sumitomo
Temephos	BASF
Permethrin/S-bioallethrin TC, EW	Bayer
FAO & WHO:	
Alpha-cypermethrin TC, SC, WP	BASF, Tagros
Bendiocarb TC, WP	Agros
Deltamethrin TC, WP	Agros
Deltamethrin TC, SC, WT, WG, WP, EC	Tagros
Permethrin TC	Sumitomo
Permethrin TC, EC	Tagros
Pyriproxyfen TC, GR	Sumitomo
Spinosad TC, GR, SC	DAS
2006 Tentative	
FAO	
Propaquizafop	Makhteshim
Oxamyl TK, SL, G	Dupont
FAO & WHO:	
Lambda-cyhalothrin TC, CS, WP	Tagros

14. Any other matters

14.1. Publication of test methods for impurities

Peer validation of methods to determine relevant impurities in technical material and in formulations: a proposal for development of guidelines for and organization of, peer validation studies through CIPAC. Proposal to publish such methods by CIPAC.

14.2. Method extension concept for impurities

JMPS asked CIPAC to develop guidelines for extension of methods for impurities.

14.3. Clarification of procedure and timelines for adoption and publication of specifications following a JMPS review

The Meeting discussed the timing for publication of specifications. It was suggested that a formal process be established to keep the proposer aware of progress and obstacles that have cropped up.

14.4. Report on closed Joint Meeting on Pesticide Specifications (JMPS)

Mr Hill (Chairman of JMPS) summarized the discussions on general items in the 2004 JMPS.

- In updating evaluations and specifications, editorials and minor changes will be introduced as date-controlled corrigenda. New information that may have an impact on decisions or recommendations will be put on the agenda of a JMPS for consideration. Borderline cases, e.g. receipt of expected method validation data, may be circulated by email for consideration by JMPS Panel members.
- If the specification 'description' clause states the identity and quantity of a stabilizer, it becomes part of the specification and a validated analytical method is needed for stabilizer analysis and identification. If the specification 'description' clause refers to a NOTE about the stabilizer, it is not part of the specification and an analytical method is not required.
- When isomer composition is specified for TC or TK, it will also be specified for formulations. Isomer composition does not need to be specified if the ISO name already defines the isomer composition.
- Products from different manufacturers may be substantially different even though having the same active ingredient and being covered by the same technical or formulation code, i.e. TC, SC, CS, LN, etc. The differences mean that there could be more than one TC, TK, etc specification for the same active ingredient. This situation can occur for traditional synthetic chemicals, but is likely to occur more frequently for botanicals, microbials and products such as LN (long-lasting insecticidal nets). The JMPS is considering how to handle this situation and how to identify different specifications that have the same nominal identifying code. The views of industry were also sought.
- The Manual includes a definition for relevant impurities and a process for the determination of equivalence. The JMPS, in the light of experience, is developing more detailed guidelines on deciding on whether or not an impurity is relevant. A delay in the process may occur if JMPS decides that an impurity is relevant, but the proposer had not anticipated the decision and had not validated an analytical method suitable for the formulations as well as the technical material. The FAO and WHO Secretaries should keep the proposer informed of the situation to minimize delays in publishing the specifications.

15. Date and Venue of next meeting

The Netherlands is the likely venue for the 2005 JMPS-CIPAC meetings.

Mixed formulation of CS and SC (ZC)

Introduction

A mixed formulation of CS and SC is a stable suspension of microcapsules of the active ingredient and fine particles of active ingredient(s) in fluid, normally intended for dilution with water before use. In the case of microcapsules, the active ingredient is present inside discrete, inert, polymeric microcapsules. The formulation is intended for dilution into water prior to spray application. Mixtures of active ingredients one of which is encapsulated are used to provide a broader spectrum of pest control. Formulating the active ingredients together eliminates the need for tank mixing (which can lead to incompatibilities). Like other aqueous liquid formulation, ZC formulations are easy to handle and measure, dust free, nonflammable and offer good miscibility with water.

Different reasons for the encapsulation of active ingredient may exist, for instance

- To increase the residual biological activity
- To reduce the acute toxicity
- To obtain a physical or chemically stable water-based formulation

This purpose determines whether the “free” active ingredient and the “release rate” are relevant properties of a specific product.

Mixed formulations of CS and SC are not stable indefinitely and therefore it is necessary to ensure that, after transportation and storage, the formulation remains suitable for use. Quantification of the following parameters, particularly after high and low temperature stability tests, serves this purpose.

- Active ingredient, determined and expressed as “total”, “free” (non-encapsulated) and “release rate” (“total” is required in all cases; “free” and “release rate” are dependent upon the intended application, see above).
- Pourability test
- Spontaneity of dispersion, suspensibility, re-suspensibility, wet sieve and persistent foam tests (to ensure the sprayability of the diluted ZC formulation)

Information about other properties may also be given, e.g. mass per milliliter and flash point (if relevant), but these parameters do not normally constitute essential parts of the specification. However, some other physical properties, especially particle size distribution and viscosity, are excluded from the specification for the following reasons.

- Particle size distribution (CIPAC MT 185). There is no internationally accepted, simple method for determination of the particle size distribution of ZC formulations. Moreover, particle size distribution is described and limited in the specification by easily quantifiable parameters which are influenced by it. These parameters are the performance in wet sieve analysis and the suspensibility tests.
- Viscosity. Although viscosity is a very important property, it cannot be described simply, as most ZC formulations show non-Newtonian flow characteristics. In the specification, the pourability and spontaneity of dispersion adequately describe the flow (rheological) properties.

[ISO Common name] Mixed formulation of CS and SC

[CIPAC number]/ZC

1.1. Description

The material shall consist of a suspension of fine particles of technical [ISO common name] complying with the requirements of the FAO/WHO specification....., in the form of(section 4.2), combined with a suspension of microcapsule of technical [ISO common name] complying with the requirements of FAO/WHO specification..., in the form of ...(section 4.2), in an aqueous phase together with suitable formulants. After gentle agitation the material shall appear homogeneous (Note 1) and be suitable for dilution in water.

1.2. Active ingredients

1.2.1. Identity test (Note 2)

The active ingredients shall comply with identity tests and, where an identity remains in doubt, it shall comply with at least one additional test.

1.2.2. [ISO common names] content

1.2.2.1. Total content (Note 2)

The [ISO common names] content shall be declared (g/kg or g/l at 20 ± 2 °C, Note 3) and, when determined, the average contents measured shall not differ from those declared by more than the appropriate tolerances, given in the table of tolerances, section 4.3.2.

1.2.2.2. Free, non-encapsulated content (if relevant, Note 4)

The free[ISO common names] average content measured shall not exceed% of the determined total content.

1.2.2.3. Release rate (if relevant, Note 4)

The release rate measured shall comply with the following criteria:

1.3. Relevant impurities

1.3.1. By-products of manufacture or storage (Note 5)

Maximum: ... % of the [ISO common name] content found under 1.2.2.1.

1.4. Physical properties

1.4.1. Acidity or alkalinity (MT31) or pH range (MT 75.3) (Note 6)

Maximum acidity: ...g/kg calculated as H₂SO₄

Maximum alkalinity: ...g/kg calculated as NaOH

pH range: ...to...

1.4.2. Pourability (MT 148.1)

Maximum "residue":%

1.4.3. Spontaneity of dispersion (MT 160) (Note 7)

A minimum of ...% of the [ISO common name] content found under 1.2.2.1. shall be in suspension after 5 min in CIPAC Standard Water D at 30 ± 2 °C (Note 8)

1.4.4. Suspensibility (MT 161, MT 184) (Note 7)

A minimum of ...% of the ..[ISO common name] content found under 1.2.2.1. shall be in suspension after 30 min in CIPAC Standard Water D at 30 ± 2 °C (Note 8).

1.4.5. Wet sieve test (MT 185) (Note 9)

Maximum: ...g/kg of the formulation shall be retained on a ... μ m test sieve, at the dilutions specified.

1.4.6. Persistent foam (MT 47.2) (Note 10)

Maximum ...ml after 1 min

1.5. Storage stability

1.5.1 Freeze/thaw stability (Note 11)

After undergoing ... freeze/thaw cycles and following homogenization, the formulation shall continue to comply with the clauses for: acidity/alkalinity/pH range, pourability, spontaneity of dispersion, suspensibility and wet sieve test, as required.

An increase in the free [ISO common name] content shall be allowed to an extent of ..% (absolute) of that found under 1.2.2.2.

1.5.2 Stability at elevated temperature (MT 46.3)

After storage at $54 \pm 2^{\circ}\text{C}$ for 14 days (Note 12), the determined average total active ingredient content must not be lower than ...% relative to the determined average content found before storage (Note 13) and the formulation shall continue to comply with the clauses for: free active ingredient content (1.2.2.2.), (an increase in the free [ISO common name] content shall be allowed to an extent of ...% (absolute) of that found under 1.2.2.1.), by-products of manufacture or storage (1.3.1.), acidity/alkalinity/pH range (1.4.1), pourability (1.4.2), spontaneity of dispersion (1.4.3.), suspensibility (1.4.4.), and wet sieve test (1.4.5.), as required.

Note 1 All physical and chemical tests listed in this specification are to be performed with a laboratory sample taken after the recommended homogenization procedure. Before sampling to verify formulation quality, inspect the commercial container carefully. On standing mixed formulation of CS and SC usually develop a concentration gradient from the top to the bottom of the container. This may result in the appearance of a clear liquid on the top and/or sediment on the bottom. Therefore before sampling, the formulation must be homogenized according to the instructions given by the manufacturer or, in the absence of such instructions, by gentle shaking of the commercial container (for example by inverting the closed container several times).

After this procedure the container shall not contain a sticky layer of non-dispersed matter at the bottom (if the ZC has flocculated it may not be possible to re-disperse this sticky layer). A suitable and simple method of checking for a non-dispersed sticky layer “cake” is by probing with a glass rod or similar device adapted to the size and shape of the container.

Note 2 Method(s) of analysis must be CIPAC, AOAC or equivalent. If the methods have not yet been published then full details, with appropriate method validation data, must be submitted to FAO/WHO by the proposal.

Note 3 Unless homogenization is carried out carefully, it is possible for the sample to become aerated. This can lead to errors in the determination of the mass per ml, and in calculation of the active ingredient content (in g/l), if methods other than MT 3.3

are used. If the buyer requires both g/kg and g/l at 20°C, then in case of dispute the analytical results shall be calculated as g/kg.

- Note 4 The need for clauses to limit the free active ingredient content and release rate of the active ingredient depends on the intended properties of the formulation. A clause to control release rate is likely to be required for formulations intended to show slow- or controlled-release properties. A clause to control free active ingredient is likely to be required where encapsulation is intended to control the release or stability of the active ingredient, or to decrease the risk to users from (accidental) exposure to the active ingredient.
- Note 5 This clause should include only relevant impurities. Method(s) of analysis must be peer validated.
- Note 6 The method to be used shall be stated. If several methods are available, a referee method shall be selected.
- Note 7 Chemical assay is the only fully reliable method to measure the mass of active ingredient still in suspension. However, simpler methods such as gravimetric and solvent-extraction determination may be used on a routine basis provided that these methods have been shown to give equal results to those of the chemical assay method. In case of dispute, the chemical method shall be the “Referee method”.
- Note 8 Unless another temperature is specified.
- Note 9 This test detects coarse particles (e.g. oversize capsules, crystals) or agglomerates (of capsules or from crust formation). or extraneous materials which could cause blockage of spray nozzles or filters in the spray tank.
- Note 10 The mass of sample to be used in the test should be specified at the application rate of use recommended by the supplier.
- Note 11 After manufacture and during shipping it is often impossible for buyer or seller to guarantee that the formulation has not been exposed to freezing temperatures. As freezing of a ZC formulation may result in undesirable, irreversible changes, including (but not limited to) capsule failure caused by crystallization of the active ingredient, the ability of the formulation to successfully withstand repeated freezing and thawing is an important property. Unless otherwise agreed, the freeze/thaw stability test shall cycle the formulation between room temperature (e.g. 20 ± 2°C) and -10 ± 2°C on 18-hour-freeze/6-hour-melt cycles for a total of 4 cycles.
- Note 12 Unless other temperatures and/or times are specified. Refer to section 4.6.2 of the FAO/WHO Manual for alternative storage conditions.
- Note 13 Samples of the formulation taken before and after the storage stability test should be analyzed concurrently after storage in order to reduce the analytical error.

Mixed formulation of CS and SE (ZE)

Introduction

A mixed formulation of CS and SE is a stable dispersion of microcapsules and a mixture of active ingredient(s) dispersed in an aqueous solution, where one (or more) of the active ingredients is in suspension form and one (or more) of the active ingredients is in emulsion form. The formulation is normally intended for dilution with water before use. In the case of microcapsules, the active ingredient is present inside discrete, inert, polymeric microcapsules. The formulation is intended for dilution into water prior to spray application. Mixtures of active ingredients one of which is encapsulated are used to provide a broader spectrum of pest control. Formulating the active ingredients together eliminates the need for tank mixing (which can lead to incompatibilities). Like other aqueous liquid formulations, ZE formulations are easy to handle and measure, dust free, nonflammable and offer good miscibility with water. Different reasons for the encapsulation of active ingredient may exist, for instance

- To increase the residual biological activity
- To reduce the acute toxicity
- To obtain a physical or chemically stable water-based formulation

This purpose determines whether the “free” active ingredient and the “release rate” are relevant properties of a specific product.

Mixed formulations of CS and SE are not stable indefinitely and therefore it is necessary to ensure that, after transportation and storage, the formulation remains suitable for use. Quantification of the following parameters, particularly after high and low temperature stability tests, serves this purpose.

- Active ingredient, determined and expressed as “total”, “free” (non-encapsulated) and “release rate” (“total” is required in all cases; “free” and “release rate” are dependent upon the intended application, see above).
- Pourability test
- Dispersion stability, wet sieve and persistent foam tests (to ensure the sprayability of the diluted ZE formulation);

Information about other properties may also be given, e.g. mass per milliliter and flash point (if relevant), but these parameters do not normally constitute essential parts of the specification. However, some other physical properties, especially particle size distribution and viscosity, are excluded from the specification for the following reasons.

- Particle size distribution (CIPAC MT 185). There is no internationally accepted, simple method for determination of the particle size distribution of ZW formulations. Moreover, particle size distribution is described and limited in the specification by easily quantifiable parameters which are influenced by it. These parameters are the performance in wet sieve analysis and the dispersion stability tests.
- Viscosity. Although viscosity is a very important property, it cannot be described simply, as most ZE formulations show non-Newtonian flow characteristics. In the specification, the pourability and dispersion stability adequately describe the flow (rheological) properties.

[ISO Common name] Mixed formulation of CS and SE

[CIPAC number]/ZE

1.5. Description

The material shall consist of an emulsion of fine droplets of technical [ISO common name] complying with the requirements of the FAO/WHO specification..., in the form of(section 4.2), and a suspension of fine particles of technical [ISO common name] complying with the requirements of the FAO/WHO specification..., in the form of(section 4.2), combined with a suspension of microcapsule of technical [ISO common name] complying with the requirements of FAO/WHO specification..., in the form of ...(section 4.2), in an aqueous phase together with suitable formulants. After gentle agitation the material shall appear homogeneous (Note 1) and be suitable for dilution in water.

1.6. Active ingredients

1.6.1. Identity test (Note 2)

The active ingredients shall comply with identity tests and, where an identity remains in doubt, it shall comply with at least one additional test.

1.6.2. [ISO common names] content

1.6.2.1. Total content (Note 2)

The [ISO common names] content shall be declared (g/kg or g/l at 20 ± 2 °C, Note 3) and, when determined, the average contents measured shall not differ from those declared by more than the appropriate tolerances, given in the table of tolerances, section 4.3.2.

1.6.2.2. Free, non-encapsulated content (if relevant, Note 4)

The free[ISO common names] average content measured shall not exceed% of the determined total content.

1.6.2.3. Release rate (if relevant, Note 4)

The release rate measured shall comply with the following criteria:

1.7. Relevant impurities

1.7.1. By-products of manufacture or storage (Note 5)

Maximum: ... % of the [ISO common name] content found under 1.2.2.1.

1.8. Physical properties

1.4.1. Acidity or alkalinity (MT31) or pH range (MT 75.3) (Note 6)

Maximum acidity: ...g/kg calculated as H₂SO₄

Maximum alkalinity: ...g/kg calculated as NaOH

pH range: ...to...

1.4.2. Pourability (MT 148.1)

Maximum "residue":%

1.4.3. Dispersion stability (MT 180) (Note 7)

The formulation, when diluted at $30 \pm 2 \text{ }^\circ\text{C}$ (Notes 8) with CIPAC Standard Waters A and D, shall continue to comply with the following:

Time after allowing the dispersion to stand	Limits of stability
0 h 0.5 h	initial dispersion complete "cream", maximum: ...ml "free oil", maximum: ...ml sediment, maximum: ...ml
24 h 24.5 h	Re-dispersion complete "cream", maximum: ...ml "free oil", maximum, ...ml sediment, maximum: ...ml

1.4.4. Wet sieve test (MT 185) (Note 9)

Maximum:g/kg of the formulation shall be retained on a ...micro m test sieve, at the dilutions specified.

1.4.5. Persistent foam (MT 47.2) (Note 10)

Maximumml after 1 min

1.6. Storage stability

1.6.1. Freeze/thaw stability (Note 11)

After undergoing ... freeze/thaw cycles and following homogenization, the formulation shall continue to comply with the clauses for: acidity/alkalinity/pH range, pourability, dispersion stability, and wet sieve test, as required.

An increase in the free [ISO common name] content shall be allowed to an extent of ..% (absolute) of that found under 1.2.2.2.

1.6.2. Stability at elevated temperature (MT 46.3)

After storage at $54 \pm 2^\circ\text{C}$ for 14 days (Note 12), the determined average total active ingredient content must not be lower than ...% relative to the determined average content found before storage (Note 13) and the formulation shall continue to comply with the clauses for: free active ingredient content (1.2.2.2.), (an increase in the free [ISO common name] content shall be allowed to an extent of ...% (absolute) of that found under 1.2.2.1., by-products of manufacture or storage (1.3.1.), acidity/alkalinity/pH range (1.4.1), pourability (1.4.2), dispersion stability (1.4.3.), and wet sieve test (1.4.4.), as required.

Note 1 All physical and chemical tests listed in this specification are to be performed with a laboratory sample taken after the recommended homogenization procedure. Before sampling to verify formulation quality, inspect the commercial container carefully. On standing ZE formulations usually develop a concentration gradient from the top to the bottom of the container. This may result in the appearance of a clear liquid on the top and/or sediment on the bottom. Therefore before sampling, the

formulation must be homogenized according to the instructions given by the manufacturer or, in the absence of such instructions, by gentle shaking of the commercial container (for example by inverting the closed container several times).

After this procedure the container shall not contain a sticky layer of non-dispersed matter at the bottom (if the ZE has flocculated it may not be possible to re-disperse this sticky layer). A suitable and simple method of checking for a non-dispersed sticky layer “cake” is by probing with a glass rod or similar device adapted to the size and shape of the container.

- Note 2 Method(s) of analysis must be CIPAC, AOAC or equivalent. If the methods have not yet been published then full details, with appropriate method validation data, must be submitted to FAO/WHO by the proposer.
- Note 3 Unless homogenization is carried out carefully, it is possible for the sample to become aerated. This can lead to errors in the determination of the mass per millilitre, and in calculation of the active ingredient content (in g/l), if methods other than MT 3.3 are used. If the buyer requires both g/kg and g/l at 20°C, then in case of dispute the analytical results shall be calculated as g/kg.
- Note 4 The need for clauses to limit the free active ingredient content and release rate of the active ingredient depends on the intended properties of the formulation. A clause to control release rate is likely to be required for formulations intended to show slow- or controlled-release properties. A clause to control free active ingredient is likely to be required where encapsulation is intended to control the release or stability of the active ingredient, or to decrease the risk to users from (accidental) exposure to the active ingredient.
- Note 5 This clause should include only relevant impurities. Method(s) of analysis must be peer validated.
- Note 6 The method to be used shall be stated. If several methods are available, a referee method shall be selected.
- Note 7 The test will normally be carried out after the stability at elevated temperatures test (1.5.1). The test should be carried out at 2% dilution or, alternatively, at the highest and lowest recommended rates of use.
- Note 8 Unless another temperature is specified.
- Note 9 This test detects coarse particles (e.g. oversize capsules, crystals) or agglomerates (of capsules or from crust formation) or extraneous materials that could cause blockage of spray nozzles or filters in the spray tank.
- Note 10 The mass of sample to be used in the test should be specified at the application rate of use recommended by the supplier.
- Note 11 After manufacture and during shipping it is often impossible for buyer or seller to guarantee that the formulation has not been exposed to freezing temperatures. As freezing of a ZE formulation may result in undesirable, irreversible changes, including (but not limited to) capsule failure caused by crystallization of the active ingredient, the ability of the formulation to successfully withstand repeated freezing and thawing

is an important property. Unless otherwise agreed, the freeze/thaw stability test shall cycle the formulation between room temperature (e.g. $20 \pm 2^{\circ}\text{C}$) and $-10 \pm 2^{\circ}\text{C}$ on 18-hour-freeze/6-hour-melt cycles for a total of 4 cycles

Note 12 Unless other temperatures and/or times are specified. Refer to section 4.6.2 of the FAO/WHO manual for alternative storage conditions.

Note 13 Samples of the formulation taken before and after the storage stability test should be analyzed concurrently after storage in order to reduce the analytical error.

Mixed formulation of CS and EW (ZW)

Introduction

A mixed formulation of CS and EW is a stable dispersion of microcapsules and active ingredient(s) in an emulsion form, normally intended for dilution with water before use. In the case of microcapsules, the active ingredient is present inside discrete, inert, polymeric microcapsules. The formulation is intended for dilution into water prior to spray application. Mixtures of active ingredients one of which is encapsulated are used to provide a broader spectrum of pest control. Formulating the active ingredients together eliminates the need for tank mixing (which can lead to incompatibilities). Like other aqueous liquid formulation, ZW formulations are easy to handle and measure, dust free, nonflammable and offer good miscibility with water.

Different reasons for the encapsulation of active ingredient may exist, for instance

- To increase the residual biological activity
- To reduce the acute toxicity
- To obtain a physical or chemically stable water-based formulation

This purpose determines whether the “free” active ingredient and the “release rate” are relevant properties of a specific product.

Mixed formulations of CS and EW are not stable indefinitely and therefore it is necessary to ensure that, after transportation and storage, the formulation remains suitable for use. Quantification of the following parameters, particularly after high and low temperature stability tests, serves this purpose.

- Active ingredient, determined and expressed as “total”, “free” (non-encapsulated) and “release rate” (“total” is required in all cases; “free” and “release rate” are dependent upon the intended application, see above).
- Pourability test
- Dispersion stability, wet sieve and persistent foam tests (to ensure the sprayability of the diluted ZW formulation)

Information about other properties may also be given, e.g. mass per milliliter and flash point (if relevant), but these parameters do not normally constitute essential parts of the specification. However, some other physical properties, especially particle size distribution and viscosity, are excluded from the specification for the following reasons.

- Particle size distribution. (CIPAC MT 185). There is no internationally accepted, simple method for determination of the particle size distribution of ZW formulations. Moreover, particle size distribution is described and limited in the specification by easily quantifiable parameters which are influenced by it. These parameters are the performance in wet sieve analysis and the dispersion stability tests.
- Viscosity. Although viscosity is a very important property, it cannot be described simply, as most ZW formulations show non-Newtonian flow characteristics. In the specification, the pourability and dispersion stability adequately describe the flow (rheological) properties.

[ISO Common name] Mixed formulation of CS and EW

[CIPAC number]/ZW

1.9. Description

The material shall consist of an emulsion of fine droplets of technical [ISO common name] complying with the requirements of the FAO/WHO specification....., in the form of(section 4.2), combined with a suspension of a microcapsule of technical [ISO common name] complying with the requirements of FAO/WHO specification..., in the form of ... (section 4.2), in an aqueous phase together with suitable formulants. After gentle agitation the material shall appear homogeneous (Note 1) and be suitable for dilution in water.

1.10. Active ingredients

1.10.1. Identity test (Note 2)

The active ingredients shall comply with identity tests and, where an identity remains in doubt, it shall comply with at least one additional test.

1.10.2. [ISO common names] content

1.10.2.1. Total content (Note 2)

The [ISO common names] content shall be declared (g/kg or g/l at 20 ± 2 °C, Note 3) and, when determined, the average contents measured shall not differ from those declared by more than the appropriate tolerances, given in the table of tolerances, section 4.3.2.

1.10.2.2. Free, non-encapsulated content (if relevant, Note 4)

The free[ISO common names] average content measured shall not exceed% of the determined total content.

1.10.2.3. Release rate (if relevant, Note 4)

The release rate measured shall comply with the following criteria:

1.11. Relevant impurities

1.11.1. By-products of manufacture or storage (Note 5)

Maximum: ... % of the [ISO common name] content found under 1.2.2.1.

1.12. Physical properties

1.4.1. Acidity or alkalinity (MT31) or pH range (MT 75.3) (Note 6)

Maximum acidity: ...g/kg calculated as H₂SO₄

Maximum alkalinity: ...g/kg calculated as NaOH

pH range: ...to...

1.4.2. Pourability (MT 148.1)

Maximum "residue":%

1.4.3. Dispersion stability (MT 180) (Note 7)

The formulation, when diluted at $30 \pm 2 \text{ }^\circ\text{C}$ (Notes 8) with CIPAC Standard Waters A and D, shall continue to comply with the following:

Time after allowing the dispersion to stand	Limits of stability
0 h 0.5 h	initial dispersion complete "cream", maximum: ...ml "free oil", maximum: ...ml sediment, maximum: ...ml
24 h 24.5 h	Re-dispersion complete "cream", maximum: ...ml "free oil", maximum, ...ml sediment, maximum: ...ml

1.4.4. Wet sieve test (MT 185) (Note 9)

Maximum:g/kg of the formulation shall be retained on a ...micro m test sieve, at the dilutions specified.

1.4.5. Persistent foam (MT 47.2) (Note 10)

Maximumml after 1 min

1.7. Storage stability

1.7.1. Freeze/thaw stability (Note 11)

After undergoing ... freeze/thaw cycles and following homogenization, the formulation shall continue to comply with the clauses for: acidity/alkalinity/pH range, pourability, dispersion stability, and wet sieve test, as required.

An increase in the free [ISO common name] content shall be allowed to an extent of ..% (absolute) of that found under 1.2.2.2.

1.7.2. Stability at elevated temperature (MT 46.3)

After storage at $54 \pm 2^\circ\text{C}$ for 14 days (Note 12), the determined average total active ingredient content must not be lower than ...% relative to the determined average content found before storage (Note 13) and the formulation shall continue to comply with the clauses for: free active ingredient content (1.2.2.2.) (an increase in the free [ISO common name] content shall be allowed to an extent of ...% (absolute) of that found under 1.2.2.1.), by-products of manufacture or storage (1.3.1.), acidity/alkalinity/pH range (1.4.1), pourability (1.4.2), dispersion stability (1.4.3.), and wet sieve test (1.4.4.), as required.

Note 1 All physical and chemical tests listed in this specification are to be performed with a laboratory sample taken after the recommended homogenization procedure.

Before sampling to verify formulation quality, inspect the commercial container carefully. On standing mixed formulation of CS and EW usually develop a concentration gradient from the top to the bottom of the container. This may result in

the appearance of a clear liquid on the top and/or sediment on the bottom. Therefore before sampling, the formulation must be homogenized according to the instructions given by the manufacturer or, in the absence of such instructions, by gentle shaking of the commercial container (for example by inverting the closed container several times).

After this procedure the container shall not contain a sticky layer of non-dispersed matter at the bottom (if the ZW has flocculated it may not be possible to re-disperse this sticky layer). A suitable and simple method of checking for a non-dispersed sticky layer “cake” is by probing with a glass rod or similar device adapted to the size and shape of the container.

- Note 2 Method(s) of analysis must be CIPAC, AOAC or equivalent. If the methods have not yet been published then full details, with appropriate method validation data, must be submitted to FAO/WHO by the proposer.
- Note 3 Unless homogenization is carried out carefully, it is possible for the sample to become aerated. This can lead to errors in the determination of the mass per millilitre, and in calculation of the active ingredient content (in g/l), if methods other than MT 3.3 are used. If the buyer requires both g/kg and g/l at 20°C, then in case of dispute the analytical results shall be calculated as g/kg.
- Note 4 The need for clauses to limit the free active ingredient content and release rate of the active ingredient depends on the intended properties of the formulation. A clause to control release rate is likely to be required for formulations intended to show slow- or controlled-release properties. A clause to control free active ingredient is likely to be required where encapsulation is intended to control the release or stability of the active ingredient, or to decrease the risk to users from (accidental) exposure to the active ingredient.
- Note 5 This clause should include only relevant impurities. Method(s) of analysis must be peer validated.
- Note 6 The method to be used shall be stated. If several methods are available, a referee method shall be selected.
- Note 7 This test will normally be carried out after the stability at elevated temperatures test (1.5.1). The test should be carried out at 2% dilution or, alternatively, at the highest and lowest recommended use rates.
- Note 8 Unless another temperature is specified.
- Note 9 This test detects coarse particles (e.g. oversize capsules, crystals) or agglomerates (of capsules or from crust formation) or extraneous materials which could cause blockage of spray nozzles or filters in the spray tank.
- Note 10 The mass of sample to be used in the test should be specified at the application rate of use recommended by the supplier.
- Note 11 After manufacture and during shipping it is often impossible for buyer or seller to guarantee that the formulation has not been exposed to freezing temperatures. As

freezing of a ZW formulation may result in undesirable, irreversible changes, including (but not limited to) capsule failure caused by crystallization of the active ingredient, the ability of the formulation to successfully withstand repeated freezing and thawing is an important property. Unless otherwise agreed, the freeze/thaw stability test shall cycle the formulation between room temperature (e.g. $20 \pm 2^{\circ}\text{C}$) and $-10 \pm 2^{\circ}\text{C}$ on 18-hour-freeze/6-hour-melt cycles for a total of 4 cycles.

Note 12 Unless other temperatures and/or times are specified. Refer to section 4.6.2 of the FAO/WHO manual for alternative storage conditions.

Note 13 Samples of the formulation taken before and after the storage stability test should be analyzed concurrently after storage in order to reduce the analytical error.

Draft Guideline for Specifications of Industrial Long Lasting Insecticidal Net

Long-Lasting Insecticidal Netting [CIPAC number]/LN

Preface

This working document provides reasonable basis for further evaluation and discussion; however it reflects currently available LLIN technology. A guideline for specifications must be equally suitable for any LLIN technology, and any net type specification used.

1. Description

The product shall consist of netting, formed from[type and mono-/poly-filament, DEN, specific weight] fibers, treated with technical[ISO common name] complying with WHO specification, or formulation thereof, together with any necessary stabilizers, plasticisers, other formulants and synergists, if required. The product shall appear clean, free from visible extraneous matter, free of damage (such as splitting or tearing) and shall be suitable for use as an insecticidal net with long lasting activity (Note 1).

2. Active ingredient

2.1 Identity tests

The active ingredient shall comply with an identity test and, where the identity remains in doubt, shall comply at least one additional test (Note 2,3).

2.2 Total content of active ingredient

The[ISO common name] content shall be within the range tog/kg and, when determined, the average content shall not differ from that declared by more than \pm g/kg, or \pm % (Note 2,3).

2.3 Initial surface concentration of active ingredient

The initial surface amount of[ISO common name] on the yarn, determined by the method described in Note 4, shall be not more thanmg/m² or g/kg of netting (Note 2,4).

2.4 Release or retention index

The release or retention index of[ISO common name] from the netting, when determined by the method described in Note 5, shall be within the range to

3. Relevant impurities

3.1 Relevant impurities of manufacturing or storage

If required,

Maximum:% of the[insert common name and/or chemical name] content found under 2.2.

4. Physical properties

Physical properties of netting must comply with the WHO specifications for netting material (WHO/CBS/RBM/2001.28, June 200), namely in the following aspects:

4.1 Netting mesh size

4.2 Dimensional stability of netting to washing (Note 6)

4.3 Mass per m² of netting (Note 7)

4.4 Bursting strength (Note 8)

4.5 Tearing strength (Note 9)

5. Storage stability

5.1 Stability at elevated temperature (MT 46.3)

After storage at $54 \pm 2^\circ\text{C}$ for 2 weeks, the determined total active ingredient content shall not be lower than% relative to the determined average content found before storage (Note 10) and the product shall continue to comply with clauses for initial surface concentration (2.3), release or retention index (2.4), dimensional stability (4.2), bursting strength (4.4) and tearing strength (4.5).

- Note 1 Long-lasting insecticidal netting is expected to retain its insecticidal activity during its lifespan and through a given number of washes. Washing should be done according to WHO guidelines. For bioassays, samples should be taken according to WHO recommendation. The long-lasting insecticidal effect may be produced by incorporation or coating of pesticide in/on the yarn.
- Note 2 Sampling. A sufficient quantity of sample is acquired by taking an appropriate procedure. For example, cut out at least one full-width strip, at least 20 cm wide, across the shortest dimension and not less than 100 cm from the end of the longest dimension of a net or the netting. Roll up the strip(s) and place it/them in a labeled, new, clean aluminum foil prior to analysis. Sub-samples for testing should be taken as described in each test method.
- Note 3 Methods must be CIPAC, AOAC or equivalent. If the methods have not yet been published then full details, with appropriate method validation data, must be provided.
- Note 4 A full description of the method for the determination of initial surface concentration must be provided or, if an appropriate method has been published, a reference must be given, or if the method has not yet been published then full details, with appropriate method validation data, must be provided. The method is expected to distinguish good and bad products of the same type, using an extraction procedure designed for the product. For this reason, a method intended for impregnated nets must not be used with coated nets, or *vice versa*, and the method must be specific to a particular product.
- Note 5 A full description of the method for release or retention index must be provided or, if an appropriate method has been published, a reference must be given, or if the method has not yet been published then full details, with appropriate method validation data, must be provided. The method is expected to enforce high quality products. For this reason, the method must be specific to a particular active ingredient or type of product. The method used for durability washing must be suitable to differentiate between initial surface load and total content without regard to any type of impregnation technology or coating and washing procedure shall be done following WHO recommendations (standard washing procedures).
- Note 6 The dimensional stability should be determined according to the method of ISO 5077 (1984) or equivalent and an appropriate reference or a particular description to the method must be provided.
- Note 7 The mass/m² should be determined according to the method of ISO 3801 (1977) or equivalent and an appropriate reference or a particular description to the method must be provided.
- Note 8 The minimum bursting strength must be measured according to ISO 13938-1 (1999) or equivalent and an appropriate reference or a particular description to the method must be provided.

- Note 9 The minimum tearing strength must be measured according to ISO 13934-2 (2000) or equivalent and an appropriate reference or a particular description to the method must be provided.
- Note 10 Samples of the product taken before and after the storage stability test should be analyzed concurrently in order to reduce the analytical error.