Food and Agriculture Organization of the United Nations

World Health Organization

Second Joint FAO/WHO Meeting on Pesticide Specifications (JMPS)

2003

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Tuesday, 10 June 2003
Bucharest, Romania
OPEN MEETING

1. Opening Speeches (From Romanian Ministry of Agriculture, FAO, WHO and CIPAC)

Mr Petre Daea, State Secretary of Ministry of Agriculture, Food and Forests, Romania, extended a warm welcome to participants of the 2nd FAO/WHO Joint Meeting on Pesticide Specifications and the 47th Collaborative International Pesticide Analytical Council Meeting. Mr Daea emphasized the importance and need for the use of quality and effective pesticides in ensuring food safety and considered the existing international cooperation to develop quality pesticides very important and valuable. He continued by stating that the use of quality pesticides contributed towards the effective control of pests and at the same time minimising the adverse effects on man and the environment. Mr Daea concluded by thanking and wishing participants, especially representatives of FAO and WHO and Chairman of CIPAC, a successful meeting.

Dr Gero Vaagt, speaking on behalf of the Food and Agriculture Organisation of the United Nations (FAO), welcomed all participants to this year's meeting on the development and use of FAO/WHO Pesticide Specifications. He then thanked the host Romanian Ministry of Agriculture, Food and Forests and especially Mrs Teodora Iurascu for the excellent support in facilitating the meeting. Dr Vaagt explained that this was his first visit to Romania and that coming from Rome and then travelling to Romania was a very interesting and curious situation. He said he was looking forward to getting to know Romanians and to get a better understanding of the relationship between Romans and Romanians.

Dr Vaagt explained that this was a very special meeting in Bucharest, being the second FAO/WHO joint meeting on Pesticide Specifications. In fact this would be the 33rd FAO meeting to develop and discuss pesticide specifications. He gave a brief history of the specification setting process and explained the importance of setting pesticide specifications as an international reference point against which to judge the quality of pesticide products for regulatory purposes or in commerce.

Dr Vaagt stated that the work on Pesticide Specifications is a good example of the cooperation between the private sector and international / intergovernmental organisations. The development of specifications is based on proposals and data submitted by the pesticide industry. Under the new

procedure, FAO/WHO specifications apply only to the products of those manufactures whose technical materials have been evaluated.

Dr Vaagt hoped that companies in the region will become more active in the future. Up to now only one application had been received from a company in this region. He hoped the meeting would strengthen the understanding of the process and the participation of the private sector.

It was assured that the FAO will continue to support the pesticide specification procedure, in particular to implement the newly revised version of the International Code of Conduct on the Distribution and use of Pesticides. A revised version was adopted in November 2002. It examines pesticide specifications in various Articles and clearly outlines the responsibilities of governments and the pesticide industry. The Code clearly defines not only the specific responsibilities but also the joint efforts necessary on the part of the major stakeholders, governments and the pesticide industry.

Dr Vaagt stated that the first edition of the Manual on Development and Use of FAO and WHO Specifications had been published at the end of 2002 and is also available on the FAO website. At the moment the manual is only available in English but it will also be published in French and Spanish. It may also be published in other languages if there is sufficient demand. The new manual has been of great interest to regulatory authorities: parts of it will be used in the EU for equivalence of technical materials and the Central American member countries of OIRSA are proposing to integrate the equivalence criteria in to their registration scheme. Dr Vaagt explained that the FAO is very proud to have set up the new procedure and he expressed thanks to all those who were involved in the lengthy negotiation process. He stated that there is growing recognition of the new procedure and it is hoped that it will help to harmonise procedures on a worldwide scale.

Dr Vaagt finished by again thanking the organisers and everyone present at the meeting.

Dr Morteza Zaim, speaking on behalf of the Executive Director of the WHO Programme on Communicable Diseases, welcomed all participants to the Open Meeting of the 2nd FAO/WHO Joint Meeting on Pesticide Specifications. He then thanked the Romanian Ministry of Agriculture, Food and Forests and especially Mrs Teodora Iurascu for the excellent support in facilitating the meeting. Dr Zaim stated that the WHO was highly committed to promote appropriate management of public health pesticides and had recently increased its efforts by developing detailed guidelines on management of these chemicals, in collaboration with pesticide registration authorities and national disease control programmes, as well as FAO, the International Programme on Chemical Safety and Industry. He continued by stating that WHO would actively promote the adaptation and implementation

of these guidelines which, among other things, contained elements such as quality control of pesticides and appropriate use of WHO recommendations and specifications by Member States.

Dr Zaim further stated that the WHO, in its efforts to assist Member States better manage public health pesticides, was in the process of expanding the network of WHO Designated Collaborating Centres for quality Control of pesticides. He expressed appreciation for the assistance provided by CIPAC in the review of current as well as potential WHO collaborating centres.

Dr Zaim further expressed his appreciation for the keen interest shown by the Industry and Member States in the FAO/WHO harmonized procedures for development of pesticide specifications and the Industry in the development of alternative pesticides and application technologies for vector control. He concluded by stating that he looked forward to a continued and fruitful collaboration with Industry, registration authorities, national quality control laboratories, FAO and CIPAC on the development of quality standards for pesticides.

Dr Markus Müller, as the chairman of CIPAC, expressed his honour in having his inaugural meeting in Bucharest and he extended a warm welcome to all participants. He expressed his gratitude and thanks to the Ministry of Agriculture, Food and Forests and especially to Mrs Teodora Iurascu and her colleagues of the Central Laboratory for Phytosanitary Quarantine, for all the hard work that had gone in to the organisation of these meetings.

Dr Müller explained that this would be the first CIPAC meeting in Romania and it has always been a CIPAC tradition to hold meetings in countries where Members live and work. Dr Müller realised that the local organisation of the meeting was a challenge; however he felt that it was an extremely valuable experience for both the inviting party and participants. It gives the opportunity to get to know each other much better and helps to spread the word on the work of FAO, WHO and CIPAC.

Dr Müller went on to explain that CIPAC is a unique organisation with scientists from governments and industry working together to produce CIPAC methods, which underpin pesticide specifications and pesticide regulations. He stated that this cooperation is a model of how the various parties could work together to produce sound schemes to assist both industrialised and developing countries. By working together FAO, WHO and CIPAC could help to provide higher quality pesticides for use in agriculture and public health. Dr Müller finished by again thanking the host nation and wished the meeting great success.

2. Appointment of Chairman and Rapporteurs

Mr Alan Hill was appointed as Chairman and Mr. Jeff Pim and Mr Tan Soo Hian as rapporteurs.

3. Adoption of agenda

The agenda was adopted with the inclusion of an item on the Establishment of Equivalence in the EU under item 9.

4. Summary Record of the 1st Open Joint FAO/WHO Meeting on Pesticide Specifications (Rome, Italy 18 June 2002)

There were no amendments to the record of the meeting.

5. Summary of action taken after the 1st JMPS Meeting

5.1 Progress in revising the International Code of Conduct on the Distribution and Use of Pesticides

Dr Vaagt reported that the revised International Code of Conduct had been adopted by the FAO Council in November 2002. The Code is now available in print as well as from FAO web site. He further informed the meeting that FAO was in the process of translating it into Arabic, Chinese, French and Spanish. The meeting was further informed that the original structure of the Code had been retained but many of the articles had been revised, including the incorporation of all relevant international policy instruments related to the management of pesticides. Of great significance was the article requesting governments to use the principles for determining equivalence of pesticides as described in the Manual on the Development and Use of FAO and WHO Specifications for Pesticides.

Dr Vaagt also addressed the concern of the Industry that making specifications developed by JMPS a prerequisite for the JMPR evaluations might delay the JMPR process. He informed the meeting that the requirement would be phased over a number of years, beginning in 2006. Under this arrangement, specifications for pesticides to be evaluated by JMPR should begin to be developed by 2004. He urged the Industry to coordinate and support the new procedure. The meeting agreed that the development of specifications should not delay the JMPR process.

5.2 Roster Call for Experts for JMPS by FAO

Dr Vaagt reported that there was a very good response to the roster call for experts for the JMPS by the FAO. To date, 78 applications had been received of which two thirds were from developing countries with the remainder from industrialized countries. The meeting was informed that FAO and WHO had

set up a panel to evaluate the applications and had each identified 5 potential candidates to join the JMPS.

5.3 Publication of the Manual on Development and Use of FAO and WHO Specifications for Pesticides, 1st Edition

Dr Vaagt informed the meeting that the Manual had been published and copies are available from FAO and WHO. It is also available on the web sites of the FAO and WHO. The meeting was further informed that a number of additional recommended formats such as the letter of access to confidential data and proposer data entry had been incorporated in the Manual to assist the Industry in their submissions.

Dr Zaim informed the meeting that, in addition to the Manual, publications by WHOPES could also be obtained from the WHO web site (http://www.who.int/ctd/whopes). He further informed the meeting of the plan to translate the Manual into French and Spanish and invited the Industry to help in the preparation of the draft. WHO and FAO would then bear the cost of the publication of the Manual. Translation into other languages would also be considered but would have to be based on demand.

6. Review and Publication of Specifications

6.1 Status of FAO Specifications

Dr Vaagt presented the status of FAO specifications that had been evaluated by the 1st JMPS meeting. The status of these specifications is as detailed in the table below.

Manufacturer	Product	FAO specification	Status
Dupont	Bensulfuron-methyl TC, WP, WG	New	Specification and Evaluation report published
	Methomyl TC, TK, SL, SG	Revised	Specification and Evaluation report published
	Tribenuron-methyl TC, WG	New	Specification and Evaluation report published

Manufacturer	Product	FAO specification	Status
BASF	Quinclorac TC, WP, WG, SC	New	Specification and Evaluation report published
Syngenta	Glyphosate SL	Extension of specification	Extended specification and amended evaluation published
BASF; Gharda; Syngenta	Dicamba TC, SL, WG	Revised	Specification and Evaluation report published
Crompton Corp. (Uniroyal)	Maleic hydrazide TC, TK, SL, SG	New	Evaluation report published – publication of specifications subject to validation of analytical methods
Fortune; Godrej; Trifolio M	Azadirachtin (TC), TK, EC	New	In progress, scheduled again for 2003
Agro-Chemie	Flufenzine (diflovidazin) TC, TK	New	In progress, scheduled again for 2003
	Beta-Cypermethrin	New	Insufficient data, removed from the programme
Bayer CropScience	Iprodione TC, SC, WG, WP	New	Withdrawn, rescheduled for 2003
Nufarm	Butralin	New	Withdrawn, rescheduled for 2004

6.2 Status of WHO Specifications

Dr Zaim expressed concern that the Industry had been rather slow in some instances in the validation of the test methods proposed for support of specifications and requested that this problem be addressed. The Chairman said that, although there had been many excellent submissions, a few proposers had been slow in their responses to the queries posed by the reviewers.

Dr Thomas Woods, speaking on behalf of Crop Life International, thanked FAO and WHO for the efficiency of the review process as well as the improved dialogue between the two parties. He also apologised on behalf of the Industry for some of the problems faced in the evaluation process and stated that the Industry would look into overcoming them. It was proposed that a status report on the work of the JMPS be posted on the FAO and WHO web site. Dr Zaim stated that there was already an existing section in the WHO web site that showed the progress of the development of WHO specifications. Dr Vaagt stated that it should not be a problem and would look into its implementation by FAO.

The Chairman reminded the meeting that, although the minutes of the Open Meeting are available on the net, minutes of the Closed Meeting would however not be published as they include confidential information.

Dr Zaim presented the status of WHO specifications reviewed in 2002, as shown below.

Manufacturer	Product	WHO specification	Status
Sumitomo	d-allethrin TC	New	Evaluation report published – publication of specification subject to validation of analytical methods
	d-phenothrin TC	New	Evaluation report published – publication of specification subject to validation of analytical methods
	Prallethrin TC	New	Evaluation report finalised – publication of specification subject to validation of analytical methods
Bayer	Transfluthrin TC	New	Evaluation report finalised – publication of specification subject to validation of analytical method

6.3 Status of FAO/WHO Specifications

Dr Zaim reported the status of FAO/WHO specifications reviewed in 2002, as shown below.

Bayer	Niclosamide TC, WP, EC	Revised/Joint	Evaluation report and specifications finalised – await decision by JMPS and Industry on definition of TC/TK
Dow AgroScience, Makhteshim & Gharda (withdrew)	Chlorpyrifos TC, EC	Revised/Joint	Evaluation report published - publication of specifications subject to validation of analytical method for the relevant impurity

7. Proposed new/amended specification guidelines for:

7.1 Insecticide-incorporated mosquito nets

The meeting was informed that annually more than 250 million people were infected with malaria and out of that a million would die. One of the best tools for personal protection was the insecticide treated mosquito net which WHO had been promoting but had however, faced many problems related to the treatment and re-treatment of these nets. To address this problem, WHO in collaboration with the Industry had developed and recently completed an evaluation of factory-treated insecticidal nets. These nets, manufactured by incorporation of the insecticide into the fibre of the net, would not require any treatment or re-treatment by users and would be effective throughout the life of the net.

The Industry in responding to the request of the WHO, had developed a draft Guideline for Specifications of Long Lasting Insecticidal Net (Annex 1). Dr Itoh Takaaki of Sumitomo Chemical Company presented the manufacturing process of these nets as well as the draft guideline.

There was concern expressed by the Industry that the storage stability test at $54 + 2\ ^{o}C$ could be too drastic for some active ingredients and hence there should be provision for other lower temperatures. It was however pointed out that such mosquito nets would generally be used in hot climates for a period of up to 5 years and a less severe condition of testing might not be appropriate. It was generally felt that, if there were to be such a need for different test conditions, decisions should be on a case by case basis. Industry was requested to consider the proposed draft guideline which should also include sampling. Comments should be sent to Dr Itoh who would coordinate and submit the revised draft guideline to the WHO for

consideration by the JMPS at next year's meeting. The final draft guideline should be submitted to the WHO before 31 December 2003.

7.1 Mixed formulation of CS and SC (New Code ZC)

7.2 Mixed formulation of CS and SE (New Code ZE)

7.3 Mixed formulation of CS and EW (New Code ZW)

These three new specification guidelines were given in a single presentation, the justification for these specifications and the draft guideline specifications themselves are detailed in Annex 2.

The Chairman questioned why there were no clauses in the specifications for free active ingredient bearing in mind that one of the reasons for the introduction of these formulations is to reduce the risk to users. He accepted that it may be difficult to determine free active but if the function of the encapsulation is to reduce operator exposure then free active ingredient would be very important for the specification. It was also recognised that, for fast release capsules, release rate as part of a specification may not be so important. The Chairman requested that CropLife International act as the coordinator to receive further comments on the draft guidelines. The amended draft guidelines should then be submitted to FAO and WHO by 31 December 2003 for consideration at next year's meeting.

8. Priority list for development of specifications and three-year programme (2004-2006)

See Annex 3.

9. Any other matters

Dr Ada Hourdaki briefed the meeting on the establishment of equivalence in the EU. The Meeting was informed that the EU had adopted the FAO/WHO process for the determination of equivalence of pesticide technical materials. It was pointed out that the determination of equivalence of active substances would be needed when:

- A large scale production had been established (pilot plant → large scale production);
- The applicant had changed the synthesis pathway/manufacturing process;
- The applicant had purchased the active ingredient from a new source in terms of new producer and/or new location; and

- A subsequent applicant had requested an authorisation.

Dr Hourdaki further informed the meeting that the EU Working Group on Plant Protection Products was in the process of preparing the Guidance Document on the Assessment of Equivalence of Technical Materials of Substances Regulated under Council Directive Dir. 91/414 EEC.

There were no other matters not covered by the agenda. The Chairman closed the meeting by thanking the organisers, the rapporteurs and the meeting participants.

Annex 1

Draft Guideline for Specifications of Long Lasting Insecticidal Net

Long-lasting insecticidal netting
[CIPAC number]/LN

1. Description

The product shall consist of netting, formed from ….. [type and mono-/poly-filament] fibres, treated with technical ……. [ISO common name] complying with WHO specification …., together with any necessary stabilizers, plasticisers, other formulants and synergists, if required. The product shall be suitable for use as an insecticidal net and shall have long-lasting activity (Note 1).

2. Active ingredient

2.1 Identity tests **(Note 2)**

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

2.2 Total content of active ingredient **(Note 2)**

The …. [ISO common name] content shall be within the range ….. to g/kg and, when determined, the average content shall not differ from that declared by more than ± ..... g/kg or ± 15% (homogenous product) or ± 25% (heterogeneous product) if the declared content is lower than 25 g/kg.

2.3 Any other relevant clause

Such as isomer ratio or synergist content, if relevant.

2.4 Initial surface concentration of active ingredient on yarn **(Note 3)**

The initial surface amount of [ISO common name] on the yarn, determined by the method described in Note 2, shall be not less than ..... mg/g of netting.

2.5 Release index or durability to washing **(Note 4)**

The …… [ISO common name] release index or durability to washing, when determined by the method described in Note 3 shall be within the range …. to ……

3. Relevant impurities

3.1 By-products of manufacture or storage [insert common name and/or chemical name]
If required,
Maximum: …… % of the ……. [ISO common name] content found under 2.2.

4. Physical properties

4.1 Fibre composition **(Note 5)**

The fibres shall be of a …..type.
If required, melt index shall be in the range .... to ….

4.2 Netting mesh size

The mesh size shall be uniform and with a minimum of complete holes per square inch.

4.3 Dimensional stability of netting to washing **(Note 6)**

The dimensional stability (length and width) shall be ± 10% of initial dimensions.

4.4 Mass per m^2 of netting **(Note 7)**

The mass/m^2 shall be … ± ……g/m^2.

4.5 Bursting strength **(Note 8)**

The minimum bursting strength shall be .....

5. Storage stability

5.1 Stability at elevated temperature (MT 46.3, CIPAC J, pp.128-130)

After storage at 54 ± 2 ^{O}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 9) and the product shall continue to comply with clauses for:
isomer ratio (2.3),
initial surface concentration (2.4),
release index (2.5)
dimensional stability (4.3) and
bursting 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. The long-lasting insecticidal effect may be produced by incorporation or coating of pesticide in/on the yarn. Although flammability of the product is not part of this specification, it should be measured by 16CFR Part 1610 and the result presented on the package.

Note 2 Sampling. 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 aluminium 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 and an appropriate reference to the method must be provided.

Note 4 A full description of the method for determination of initial surface concentration must be provided or, if the method has been published, an appropriate reference must be given. 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 may be specific to a particular product.

Note 5 A full description of the method for release index or durability to washing must be provided or, if the method has been published, an appropriate reference must be given. 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 may be specific to a particular product.

Note 6 The melt index should be determined according to the method of ISO (1997).

Note 7 The dimensional stability should be determined according to the method of ISO 5077 (1984).

Note 8 The mass/m^2 should be determined according to the method of ISO 3801 (1977).

Note 9 The minimum bursting strength must be measured according to ISO 93938-2 (1999), using a 7.3 cm^2 sample.

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.

Annex 2

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 “release rate” is a relevant property 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” and “release rate” (“total” is required in all cases and “release rate” is dependent upon the intended application).

- Pourability test.

- Dispersion stability, wet sieve and persistent foam tests (to ensure the sprayability of the diluted ZE formulation).

- Rate of release. In assessing performance of a capsule, the rate of release of the active ingredient after application may be considered an important property (see above).

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).

- 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 water dispersibility adequately described the flow (rheological) properties.

[ISO Common name] Mixed formulation of CS and SE

[CIPAC number]/ZE

1.1. 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.

1.2. Active ingredients

1.2.1.1. 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.

1.2.1.2. [ISO common names] content

1.2.1.3. Total content (No. 2)

The [ISO common names] content shall be declared (g/kg or g/l at 20 ± 2 ? , 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.

1.2.1.4. Release rate (if relevant, see introduction)

1.3. Relevant impurities

1.3.1.1. By-products of manufacture or storage (Note 4)

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 5)

Maximum acidity: … g/kg calculated as H_2SO_4
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 6)

The formulation, when diluted at 30 ± 2 ? (Notes 7 and 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	initial dispersion complete
0.5 h	"cream", maximum: … ml "free oil", maximum: … ml sediment, maximum: … ml
24 h	Re-dispersion complete
24.5 h	"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)

Maximum ….ml after 1 min

1.5. Storage stability

1.5.1.1. Stability at elevated temperature (MT 46.3)

After storage at 54 ± 2? for 14 days (Note 11), the determined average total active ingredient content must not be lower than … % relative to the determined average content found before storage (Note 12) 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 ?, then in case of dispute the analytical results shall be calculated as g/kg.

Note 4 This clause should include only relevant impurities. Method(s) of analysis must b peer validated.

Note 5 The method to be used shall be stated. If several methods are available, a referee method shall be selected.

Note 6 The test will normally be carried out after the stability at elevated temperatures test (7.41.5.2). The test should be carried out at the highest and lowest recommended rates of use.

Note 7 Unless another temperature is specified.

Note 8 The formulation should be tested at 2% dilution or, alternatively, at the highest and lowest rates of use recommended by the supplier.

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 Unless other temperatures and/or times are specified. Refer to section 4.6.2 of this Manual for alternative storage conditions.

Note 12 Samples of the formulation taken before and after the storage stability test should be analyzed concurrently after the test in order to reduce the analytical error.

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 “release rate” is a relevant property 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” and “release rate” (“total” is required in all cases and “release rate” is dependent upon the intended application).
- Pourability test.
- Dispersion stability, suspensibility, re-suspensibility, wet sieve and persistent foam tests (to ensure the sprayability of the diluted ZC formulation).
- Rate of release. In assessing performance of a capsule, the rate of release of the active ingredient after application may be an important property (see above).

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).
- 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 water dispersibility adequately described the flow (rheological) properties.

[ISO Common name] Mixed formulation of CS and SC

[CIPAC number]/ZC

1.5. 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.

1.6. Active ingredients

1.6.1.1. 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.

1.6.1.2. [ISO common names] content

1.2.2.1. Total content (No. 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. Release rate (if relevant, see introduction)

1.7. Relevant impurities

1.7.1.1. By-products of manufacture or storage (Note 4)

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 5)

Maximum acidity: … g/kg calculated as H_2SO_4
Maximum alkalinity: … g/kg calculated as NaOH
pH range: …to…

1.4.2. Pourability (MT 148.1)

Maximum “residue”: …..%

1.4.3. Dispersion stability (CIPAC MT 180)

The formulation, when diluted at 30 ± 2 ? (Notes 7 and 8) with CIPAC Standard Waters A and D, shall continue to comply with the following:

1.4.5. Wet sieve test (MT 185) (Note 8)

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

1.4.6. Persistent foam (MT 47.2) (Note 9)

Maximum …. ml after 1 min

1.6. Storage stability

1.6.1.1. Stability at elevated temperature (MT 46.3)

After storage at 54 ± 2? for 14 days (Note 10), the determined average active ingredient content must not be lower than … % relative to the determined average content found before storage (Note 11) 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 … % of that found under 1.2.2.1., by-products of manufacture or storage (1.3.1.), acidity/alkalinity/pH range (1.4.2), pourability (1.4.3), spontaneity of dispersion (1.4.4.), suspensibility (1.4.5.), and wet sieve test (1.4.6.), 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 This clause should include only relevant impurities. Method(s) of analysis must be peer validated.

Note 5 The method to be used shall be stated. If several methods are available, a referee method shall be selected.

Note 6 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 7 Unless another temperature is specified.

Note 8 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 9 The mass of sample to be used in the test should be specified at the application rate of use recommended by the supplier.

Note 10 Unless other temperatures and/or times are specified. Refer to section 4.6.2 of this Manual for alternative storage conditions.

Note 11 Samples of the formulation taken before and after the storage stability test should be analyzed concurrently after the test 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 “release rate” is a relevant property 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” and “release rate” (“total” is required in all cases and “release rate” is dependent upon the intended application).

- Pourability test.

- Dispersion stability, wet sieve and persistent foam tests (to ensure the sprayability of the diluted ZW formulation).

- Rate of release. In assessing performance of a capsule, the rate of release of the active ingredient after application may be an important property (see above).

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).
- 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 water dispersibility adequately described 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 (No. 2)

The [ISO common names] content shall be declared (g/kg or g/l at 20 ±2 ? , 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. Release rate (if relevant, see introduction)

1.10.3. Relevant impurities

1.10.4. By-products of manufacture or storage (Note 4)

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

1.10.5. Physical properties

1.4.1. Acidity or alkalinity (MT31) or pH range (MT 75.3) (Note 5)

Maximum acidity: …g/kg calculated as H_2SO_4
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 6)

The formulation, when diluted at 30 ± 2 ? (Notes 7 and 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	initial dispersion complete
0.5 h	“cream”, maximum: … ml “free oil”, maximum: … ml sediment, maximum: … ml
24 h	Re-dispersion complete
24.5 h	“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)

Maximum …. ml after 1 min

1.7. Storage stability

1.7.1.1. Stability at elevated temperature (MT 46.3)

After storage at 54 ± 2? for 14 days (Note 11), the determined average active ingredient content must not be lower than … % relative to the determined average content found before storage (Note 12) 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 … % 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 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 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?, then in case of dispute the analytical results shall be calculated as g/kg.

Note 4 This clause should include only relevant impurities. Method(s) of analysis must be peer validated.

Note 5 The method to be used shall be stated. If several methods are available, a referee method shall be selected.

Note 6 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 7 Unless another temperature is specified.

Note 8 The formulation should be tested at 2% dilution or, alternatively, at the highest and lowest rates of use recommended by the supplier.

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 Unless other temperatures and/or times are specified. Refer to section 4.6.2 of this Manual for alternative storage conditions.

Note 12 Samples of the formulation taken before and after the storage stability test should be analyzed concurrently after the test in order to reduce the analytical error.

PROGRAMME FOR DEVELOPMENT OF FAO AND WHO SPECIFICATIONS FOR PESTICIDES

Year	*Products*	*Proposer(s)*
2004	**FAO:**	
	Azadirachtin	Fortune Biotech
	Butralin	Nufarm
	Chlorothalonil TC,SC,WG,WP	Syngenta
	Cymoxanil TC,WP,WG	Dupont
	Guazatine TC, LS	Makhteshim
	Picloram	DAS
	Prochloraz TC, EC, SC	Makhteshim
	Propanil	Propanil Task Force (DAS; Riceco)
	WHO:	
	Bacillus thuringiensis israelensis TK, WG	Valent BioSciences
	Deltamethrin long-lasting insecticidal net	Vestergaard
	Icaridin (KBR 3023)	Bayer
	FAO & WHO:	
	Bifenthrin TC, WP	FMC
	Deltamethrin TC, DP, SC, UL, WG, WP, WT	Bayer
	Diflubenzuron TC, WP	Crompton Corp.
	Fenthion TC, EC, WP	Bayer
	Pirimiphos-methyl TC, EC, WP	Syngenta
2005 tentative	**FAO:**	
	Azimsulfuron	Dupont
	Nicosulfuron	Dupont
	WHO:	
	Permethrin long-lasting insecticidal net	Sumitomo
	FAO & WHO:	
	Permethrin TC	Sumitomo
	Pyriproxyfen TC,GR	Sumitomo