

**WHO SPECIFICATIONS AND EVALUATIONS
FOR PUBLIC HEALTH PESTICIDES**

DELTAMETHRIN

**(S)- α -cyano-3-phenoxybenzyl (1R,3R)-3-(2,2-
dibromovinyl)-2,2-dimethylcyclopropane
carboxylate**



**World Health
Organization**

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Disclaimer¹

WHO specifications are developed with the basic objective of promoting, as far as practicable, the manufacture, distribution and use of pesticides that meet basic quality requirements.

Compliance with the specifications does not constitute an endorsement or warranty of the fitness of a particular pesticide for a particular purpose, including its suitability for the control of any given pest, or its suitability for use in a particular area. Owing to the complexity of the problems involved, the suitability of pesticides for a particular purpose and the content of the labelling instructions must be decided at the national or provincial level.

Furthermore, pesticides which are manufactured to comply with these specifications are not exempted from any safety regulation or other legal or administrative provision applicable to their manufacture, sale, transportation, storage, handling, preparation and/or use.

WHO disclaims any and all liability for any injury, death, loss, damage or other prejudice of any kind that may be arise as a result of, or in connection with, the manufacture, sale, transportation, storage, handling, preparation and/or use of pesticides which are found, or are claimed, to have been manufactured to comply with these specifications.

Additionally, WHO wishes to alert users to the fact that improper storage, handling, preparation and/or use of pesticides can result in either a lowering or complete loss of safety and/or efficacy.

WHO is not responsible, and does not accept any liability, for the testing of pesticides for compliance with the specifications, nor for any methods recommended and/or used for testing compliance. As a result, WHO does not in any way warrant or represent that any pesticide claimed to comply with a WHO specification actually does so.

¹ This disclaimer applies to all specifications published by WHO.

INTRODUCTION

WHO establishes and publishes specifications* for technical material and related formulations of public health pesticides with the objective that these specifications may be used to provide an international point of reference against which products can be judged either for regulatory purposes or in commercial dealings.

From 2002, the development of WHO specifications follows the **New Procedure**, described in the 1st edition of Manual for Development and Use of FAO and WHO Specifications for Pesticides (2002). This **New Procedure** follows a formal and transparent evaluation process. It describes the minimum data package, the procedure and evaluation applied by WHO and the experts of the “FAO/WHO Joint Meeting on Pesticide Specifications” (JMPS).

WHO Specifications now only apply to products for which the technical materials have been evaluated. Consequently, from the year 2002 onwards the publication of WHO specifications under the **New Procedure** has changed. Every specification consists now of two parts, namely the specifications and the evaluation report(s):

Part One: The Specification of the technical material and the related formulations of the pesticide in accordance with chapters 4 to 9 of the 1st edition of the “FAO/WHO Manual on Pesticide Specifications.”

Part Two: The Evaluation Report(s) of the pesticide, reflecting the evaluation of the data package carried out by WHO and the JMPS. The data are provided by the manufacturer(s) according to the requirements of chapter 3 of the “FAO/WHO Manual on Pesticide Specifications” and supported by other information sources. The Evaluation Report includes the name(s) of the manufacturer(s) whose technical material has been evaluated. Evaluation reports on specifications developed subsequently to the original set of specifications are added in a chronological order to this report.

WHO specifications under the **New Procedure** do not necessarily apply to nominally similar products of other manufacturer(s), nor to those where the active ingredient is produced by other routes of manufacture. WHO has the possibility to extend the scope of the specifications to similar products but only when the JMPS has been satisfied that the additional products are equivalent to that which formed the basis of the reference specification.

Specifications bear the date (month and year) of publication of the current version. Dates of publication of the earlier versions, if any, are identified in a footnote. Evaluations bear the date (year) of the meeting at which the recommendations were made by the JMPS.

* Footnote: The publications are available on the Internet under (<http://www.who.int/quality/en/>).

PART ONE
SPECIFICATIONS

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WHO SPECIFICATIONS FOR AGRICULTURAL PESTICIDES

DELTAMETHRIN

INFORMATION

ISO common names

Deltamethrin (BSI, draft E-ISO), deltaméthrine ((f) draft F-ISO)

Synonyms

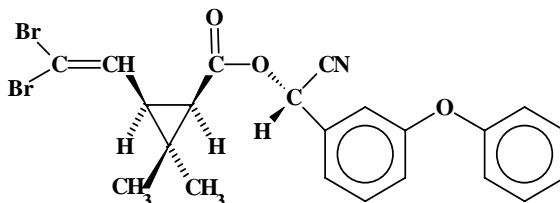
Decamethrin (rejected common name)

Chemical names

IUPAC (S)- α -cyano-3-phenoxybenzyl (1R,3R)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxylate

CA [1R-[1 α (S*),3 α]]-cyano(3-phenoxyphenyl)methyl 3-(2,2-dibromoethenyl)-2,2-dimethylcyclopropanecarboxylate

Structural formula



Empirical formula

C₂₂H₁₉Br₂NO₃

Relative molecular mass

505.2 g/mol

CAS Registry number

52918-63-5

CIPAC number

333

EEC number

258-256-6

Identity tests

HPLC retention time; TLC; IR, NMR and mass spectra

WHO SPECIFICATIONS FOR PUBLIC HEALTH PESTICIDES

DELTAMETHRIN TECHNICAL MATERIAL

WHO specification 333/TC (April 2005*)

This specification, which is PART ONE of this publication, is based on an evaluation of data submitted by the manufacturers whose names are listed in the evaluation reports (333/2004, 333/2005, 333/2006.2). It should be applicable to TC produced by these manufacturers but it is not an endorsement of those products, nor a guarantee that they comply with the specifications. The specification may not be appropriate for TC produced by other manufacturers. The evaluation reports (333/2004, 333/2005, 333/2006.2), as PART TWO, form an integral part of this publication.

1 Description

The material shall consist of deltamethrin together with related manufacturing impurities and shall be a white to cream coloured crystalline powder, free from visible extraneous matter and added modifying agents.

2 Active ingredient

2.1 Identity tests (333/TC/M/2, CIPAC Handbook L, p.45, 2006)

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

2.2 Deltamethrin content (333/TC/M/3, CIPAC Handbook L, p.45, 2006)

The deltamethrin content shall be declared (not less than 985 g/kg) and, when determined, the average measured content shall not be lower than the declared minimum content.

3 Relevant impurities (Note 1)

Note1 There are no relevant impurities to be controlled in products of the manufacturers identified in evaluation reports 333/2004, 333/2005 and 333/2006.2. However, becisthemic acid chloride [(1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxoyl chloride], sometimes spelt bicisthemic acid chloride, can occur as a result of certain manufacturing processes. If this impurity could occur at ≥1 g/kg (of deltamethrin) in the products of other manufacturers, it would be designated as a relevant impurity and a clause would be required to limit its concentration.

* Specifications may be revised and/or additional evaluations may be undertaken. Ensure the use of current versions by checking at: <http://www.who.int/quality/en/>.

WHO SPECIFICATIONS FOR PUBLIC HEALTH PESTICIDES

DELTAMETHRIN DUSTABLE POWDER

WHO specification 333/DP (April 2005*)

This specification, which is PART ONE of this publication, is based on an evaluation of data submitted by the manufacturers whose names are listed in the evaluation reports (333/2004, 333/2005, 333/2006.2). It should be applicable to relevant products of this manufacturer, and those of any other formulators who use only TC from the evaluated sources. The specification is not an endorsement of those products, nor a guarantee that they comply with the specification. The specification may not be appropriate for the products of other manufacturers who use TC from other sources. The evaluation reports (333/2004, 333/2005, 333/2006.2), as PART TWO, form an integral part of this publication.

1 Description

The material shall consist of a homogeneous mixture of technical deltamethrin, complying with the requirements of WHO specification 333/TC (April 2005), together with carriers and any other necessary formulants. It shall be in the form of a fine, free-flowing powder, free from visible extraneous matter and hard lumps.

2 Active ingredient

2.1 Identity tests (333/DP/M/2, CIPAC Handbook L, p.45, 2006)

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

2.2 Deltamethrin content (333/DP/M/3, CIPAC Handbook L, p.45, 2006)

The deltamethrin content shall be declared (g/kg) and, when determined, the average measured content shall not differ from that declared by more than the following tolerance:

Declared content, g/kg	Tolerance
up to 25	± 25% of the declared content
Note: the upper limit is included in the range	

3 Relevant impurities (Note 1)

4 Physical properties

4.1 pH range (MT 75.3, CIPAC Handbook J, p.131, 2000)

The pH of an aqueous dispersion shall be 4.5 to 7.5.

* Specifications may be revised and/or additional evaluations may be undertaken. Ensure the use of current versions by checking at: <http://www.who.int/quality/en/>.

4.2 **Dry sieve test** (MT 59.1, CIPAC Handbook F, p.177, 1995)

Maximum: 2% retained on a 75 µm test sieve.

5 **Storage stability**

5.1 **Stability at elevated temperature** (MT 46.3, CIPAC Handbook J, p.128, 2000)

After storage at $54 \pm 2^\circ\text{C}$ for 14 days, the determined average active ingredient content must not be lower than 95% relative to the determined mean content found before storage (Note 2) and the formulation shall continue to comply with the clauses for:

- pH range (4.1);
- dry sieve test (4.2).

Note 1 There are no relevant impurities to be controlled in products of the manufacturers identified in evaluation reports 333/2004, 333/2005 and 333/2006.2. However, becisthemic acid chloride [(1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxoyl chloride], sometimes spelt bicisthemic acid chloride, can occur as a result of certain manufacturing processes. If this impurity could occur at ≥ 1 g/kg (of deltamethrin) in the products of other manufacturers, it would be designated as a relevant impurity and a clause would be required to limit its concentration.

Note 2 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.

DELTAMETHRIN WETTABLE POWDER

WHO specification 333/WP (September 2005*)

This specification, which is PART ONE of this publication, is based on an evaluation of data submitted by the manufacturers whose names are listed in the evaluation reports (333/2004, 333/2005, 333/2006.2). It should be applicable to relevant products of this manufacturer, and those of any other formulators who use only TC from the evaluated sources. The specification is not an endorsement of those products, nor a guarantee that they comply with the specification. The specification may not be appropriate for the products of other manufacturers who use TC from other sources. The evaluation reports (333/2004, 333/2005, 333/2006.2), as PART TWO, form an integral part of this publication.

1 Description

The material shall consist of a homogeneous mixture of technical deltamethrin, complying with the requirements of WHO specification 333/TC (April 2005), together with filler(s) and any other necessary formulators. It shall be in the form of a fine powder free from visible extraneous matter and hard lumps.

2 Active ingredient

2.1 Identity tests (333/WP/M/2, CIPAC Handbook L, p.45, 2006)

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

2.2 Deltamethrin content (333/WP/M/3, CIPAC Handbook L, p.45, 2006)

The deltamethrin content shall be declared (g/kg) and, when determined, the average measured content shall not differ from that declared by more than the following tolerances:

Declared content, g/kg	Tolerance
up to 25	± 25% of the declared content
above 25 up to 100	± 10% of the declared content
Note: the upper limit is included in each range	

3 Relevant impurities (Note 1)

4 Physical properties

4.1 pH range (MT 75.3, CIPAC Handbook J, p.131, 2000)

The pH of an aqueous dispersion shall be 4.5 to 7.5.

* Specifications may be revised and/or additional evaluations may be undertaken. Ensure the use of current versions by checking at: <http://www.who.int/quality/en/>.

4.2 **Wet sieve test** (MT 59.3, CIPAC Handbook F, p.179, 1995)

Maximum: 2% retained on a 75 µm test sieve.

4.3 **Suspensibility** (MT 15.1, CIPAC Handbook F, p.145, 1995) (Notes 2 & 3)

A minimum of 60% of the deltamethrin content found under 2.2 shall be in suspension after 30 min in CIPAC Standard Water D at $30 \pm 2^\circ\text{C}$ (Note 4).

4.4 **Persistent foam** (MT 47.2, CIPAC Handbook F, p.152, 1995) (Note 5)

Maximum: 60 ml after 1 min.

4.5 **Wettability** (MT 53.3, CIPAC Handbook F, p.164, 1995)

The formulation shall be completely wetted in 2 min without swirling.

5 Storage stability

5.1 **Stability at elevated temperature** (MT 46.3, CIPAC Handbook J, p.128, 2000)

After storage at $54 \pm 2^\circ\text{C}$ for 14 days, the determined average active ingredient content must not be lower than 95% relative to the determined mean content found before storage (Note 6) and the formulation shall continue to comply with the clauses for:

- pH range (4.1);
- wet sieve test (4.2);
- suspensibility (4.3);
- wettability (4.5).

Note 1 There are no relevant impurities to be controlled in products of the manufacturers identified in evaluation reports 333/2004, 333/2005 and 333/2006.2. However, becisthemic acid chloride [(1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxoyl chloride], sometimes spelt bicisthemic acid chloride, can occur as a result of certain manufacturing processes. If this impurity could occur at ≥ 1 g/kg (of deltamethrin) in the products of other manufacturers, it would be designated as a relevant impurity and a clause would be required to limit its concentration.

Note 2 The formulation should be tested at the highest and lowest rates of use recommended by the supplier, provided this does not exceed the conditions given in method MT 15.1.

Note 3 This test will normally only be carried out after the heat stability test, 5.1.

Note 4 Chemical assay is the only fully reliable method to measure the mass of active ingredient still in suspension. However, the simpler gravimetric method, MT 168, may be used on a routine basis provided that it has been shown to give equal results to those of chemical assay. In case of dispute, chemical assay shall be the "referee method".

Note 5 The mass of sample to be used in the test should be at the highest rate of use recommended by the supplier.

Note 6 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.

WHO SPECIFICATIONS FOR PUBLIC HEALTH PESTICIDES

DELTAMETHRIN AQUEOUS SUSPENSION CONCENTRATE

WHO specification 333/SC (April 2005*)

This specification, which is PART ONE of this publication, is based on an evaluation of data submitted by the manufacturers whose names are listed in the evaluation reports (333/2004, 333/2005, 333/2006.2). It should be applicable to relevant products of this manufacturer, and those of any other formulators who use only TC from the evaluated sources. The specification is not an endorsement of those products, nor a guarantee that they comply with the specification. The specification may not be appropriate for the products of other manufacturers who use TC from other sources. The evaluation reports (333/2004, 333/2005, 333/2006.2), as PART TWO, form an integral part of this publication.

1 Description

The material shall consist of a suspension of fine particles of technical deltamethrin, complying with the requirements of WHO specification 333/TC (April 2005), in an aqueous phase together with suitable formulants. After gentle agitation the material shall be homogeneous (Note 1) and suitable for further dilution in water.

2 Active ingredient

2.1 Identity tests (333/SC/M/2, CIPAC Handbook L, p.45, 2006)

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

2.2 Deltamethrin content (333/SC/M/3, CIPAC Handbook L, p.45, 2006)

The deltamethrin content shall be declared (10 g/kg or g/l at $20 \pm 2^\circ\text{C}$, Note 2) and, when determined, the average measured content shall not differ from that declared by more than $\pm 15\%$.

3 Relevant impurities (Note 3)

4 Physical properties

4.1 pH range (MT 75.3, CIPAC Handbook J, p.131, 2000)

The pH of an aqueous dispersion shall be 4.5 to 7.5.

4.2 Pourability (MT 148, CIPAC Handbook F, p.348, 1995)

Maximum "residue": 5%.

* Specifications may be revised and/or additional evaluations may be undertaken. Ensure the use of current versions by checking at: <http://www.who.int/quality/en/>.

4.3 Spontaneity of dispersion (MT 160, CIPAC Handbook F, p.391, 1995)
(Note 4)

A minimum of 90% of the deltamethrin content found under 2.2 shall be in suspension after 5 min in CIPAC Standard Water D at $30 \pm 2^\circ\text{C}$.

4.4 Suspensibility (MT 161, CIPAC Handbook F, p.394, 1995) (Note 4)

A minimum of 90% of the deltamethrin content found under 2.2 shall be in suspension after 30 min in CIPAC Standard Water D at $30 \pm 2^\circ\text{C}$.

4.5 Wet sieve test (MT 185, CIPAC Handbook K, p.148, 2003)

Maximum: 2% retained on a 75 μm test sieve.

4.6 Persistent foam (MT 47.2, CIPAC Handbook F, p.152, 1995) (Note 5)

Maximum: 50 ml after 1 min.

5 Storage stability

5.1 Stability at 0°C (MT 39.3, CIPAC Handbook J, p.126, 2000)

After storage at $0 \pm 2^\circ\text{C}$ for 7 days, the formulation shall continue to comply with the clauses for:

- suspensibility (4.4);
- wet sieve test (4.5).

5.2 Stability at elevated temperature (MT 46.3, CIPAC Handbook J, p.128, 2000)

After storage at $54 \pm 2^\circ\text{C}$ for 14 days, the determined average active ingredient content must not be lower than 95% relative to the determined mean content found before storage (Note 6) and the formulation shall continue to comply with the clauses for:

- pH range (4.1);
- pourability (4.2);
- spontaneity of dispersion (4.3);
- suspensibility (4.4);
- wet sieve test (4.5).

Note 1 Before sampling to verify the formulation quality, inspect the commercial container carefully. On standing, suspension concentrates usually develop a concentration gradient from the top to the bottom of the container. This may even result in the appearance of a clear liquid on the top and/or of sediment on the bottom. Therefore, before sampling, homogenize the formulation 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). Large containers must be opened and stirred adequately. After this procedure, the container should not contain a sticky layer of non-dispersed matter at the bottom. 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. All the physical and chemical tests must be carried out on a laboratory sample taken after the recommended homogenization procedure.

Note 2 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 3 There are no relevant impurities to be controlled in products of the manufacturers identified in evaluation reports 333/2004, 333/2005 and 333/2006.2. However, becisthemic acid chloride [(1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxoyl chloride], sometimes spelt bicisthemic acid chloride, can occur as a result of certain manufacturing processes. If this impurity could occur at ≥ 1 g/kg (of deltamethrin) in the products of other manufacturers, it would be designated as a relevant impurity and a clause would be required to limit its concentration.

Note 4 Chemical assay is the only fully reliable method to measure the mass of active ingredient still in suspension. However, the simpler gravimetric method, MT 168, may be used on a routine basis provided that it has been shown to give equal results to those of chemical assay. In case of dispute, chemical assay shall be the "referee method".

Note 5 The mass of sample to be used in the test should be at the highest rate of use recommended by the supplier.

Note 6 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.

WHO SPECIFICATIONS FOR PUBLIC HEALTH PESTICIDES

DELTAMETHRIN EMULSIFIABLE CONCENTRATE

WHO specification 333/EC (April 2005*)

This specification, which is PART ONE of this publication, is based on an evaluation of data submitted by the manufacturers whose names are listed in the evaluation reports (333/2004, 333/2005, 333/2006.2). It should be applicable to relevant products of this manufacturer, and those of any other formulators who use only TC from the evaluated sources. The specification is not an endorsement of those products, nor a guarantee that they comply with the specification. The specification may not be appropriate for the products of other manufacturers who use TC from other sources. The evaluation reports (333/2004, 333/2005, 333/2006.2), as PART TWO, form an integral part of this publication.

1 Description

The material shall consist of technical deltamethrin, complying with the requirements of WHO specification 333/TC (April 2005), dissolved in suitable solvents, together with any other necessary formulants. It shall be in the form of a stable homogeneous liquid, free from visible suspended matter and sediment, to be applied as an emulsion after dilution in water.

2 Active ingredient

2.1 Identity tests (333/EC/M/2, CIPAC Handbook L, p.45, 2006)

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

2.2 Deltamethrin content (333/EC/M/3, CIPAC Handbook L, p.45, 2006)

The deltamethrin content shall be declared (g/kg or g/l at $20 \pm 2^\circ\text{C}$, Note 1) and, when determined, the average measured content shall not differ from that declared by more than the following tolerances:

Declared content, g/kg or g/l at $20 \pm 2^\circ\text{C}$	Tolerance
up to 25	$\pm 15\%$ of the declared content
above 25 up to 100	$\pm 10\%$ of the declared content
above 100 up to 250	$\pm 6\%$ of the declared content
Note: the upper limit is included in each range	

3 Relevant impurities (Note 2)

* Specifications may be revised and/or additional evaluations may be undertaken. Ensure the use of current versions by checking at: <http://www.who.int/quality/en/>.

4 Physical properties

4.1 pH range (MT 75.3, CIPAC Handbook J, p.131, 2000)

The pH of an aqueous dispersion shall be 4.5 to 7.5.

4.2 Emulsion stability and re-emulsification (MT 36.1.1, CIPAC Handbook F, p.108, 1995) (Note 3)

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

Time after dilution	Limits of stability
0 h	Initial emulsification complete
0.5 h	Cream: none
2.0 h	"Cream", maximum: 1 ml "Free oil": none
24 h	Re-emulsification complete
24.5 h	"Cream": none "Free oil": none.

Note: tests at 24 h are required only where the results at 2 h are in doubt.

4.3 Persistent foam (MT 47.2, CIPAC Handbook F, p.152, 1995) (Note 4)

Maximum: 50 ml after 1 min.

5 Storage stability

5.1 Stability at 0°C (MT 39.3, CIPAC Handbook J, p.126, 2000)

After storage at $0 \pm 2^\circ\text{C}$ for 7 days, the volume of solid and/or liquid which separates shall not be more than 0.3 ml.

5.2 Stability at elevated temperature (MT 46.3, CIPAC Handbook J, p.128, 2000)

After storage at $54 \pm 2^\circ\text{C}$ for 14 days, the determined average active ingredient content must not be lower than 95% relative to the determined average content found before storage (Note 5) and the formulation shall continue to comply with the clauses for:

- pH range (4.1);
- emulsion stability and re-emulsification (4.2).

Note 1 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 2 There are no relevant impurities to be controlled in products of the manufacturers identified in evaluation reports 333/2004, 333/2005 and 333/2006.2. However, becisthemic acid chloride [(1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxoyl chloride], sometimes spelt bicisthemic acid chloride, can occur as a result of certain manufacturing processes. If this impurity could occur at ≥ 1 g/kg (of deltamethrin) in the products of other manufacturers, it

would be designated as a relevant impurity and a clause would be required to limit its concentration.

Note 3 This test will normally only be carried out after the heat stability test: 5.2.

Note 4 The mass of sample to be used in the test should be at the highest rate of use recommended by the supplier.

Note 5 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.

DELTAMETHRIN ULTRA LOW VOLUME LIQUID

WHO specification 333/UL (September 2005*)

This specification, which is PART ONE of this publication, is based on an evaluation of data submitted by the manufacturers whose names are listed in the evaluation reports (333/2004, 333/2005, 333/2006.2). It should be applicable to relevant products of this manufacturer, and those of any other formulators who use only TC from the evaluated sources. The specification is not an endorsement of those products, nor a guarantee that they comply with the specification. The specification may not be appropriate for the products of other manufacturers who use TC from other sources. The evaluation reports (333/2004, 333/2005, 333/2006.2), as PART TWO, form an integral part of this publication.

1 Description

The material shall consist of technical deltamethrin, complying with the requirements of WHO specification 333/TC (April 2005), together with any necessary formulants (Note 1). It shall be in the form of a stable homogeneous liquid, free from visible suspended matter and sediment.

2 Active ingredient

2.1 Identity tests (333/UL/M/2, CIPAC Handbook L, p.45, 2006)

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

2.2 Deltamethrin content (333/UL/M/3, CIPAC Handbook L, p.45, 2006)

The deltamethrin content shall be declared (g/kg or g/l at $20 \pm 2^\circ\text{C}$, Note 2) and, when determined, the average measured content shall not differ from that declared by more than the following tolerances:

Declared content, g/kg or g/l at $20 \pm 2^\circ\text{C}$	Tolerance
up to 25	$\pm 15\%$ of the declared content
above 25 up to 100	$\pm 10\%$ of the declared content

Note: the upper limit is included in each range

3 Relevant impurities (Note 3)

4 Physical properties (Notes 4 & 5)

4.1 pH range (MT 75.3, CIPAC Handbook J, p.131, 2000)

The pH of an aqueous dispersion shall be 4.5 to 7.5.

* Specifications may be revised and/or additional evaluations may be undertaken. Ensure the use of current versions by checking at: <http://www.who.int/quality/en/>.

5 Storage stability

5.1 Stability at 0°C (MT 39.3, CIPAC Handbook J, p.126, 2000)

After storage at $0 \pm 2^\circ\text{C}$ for 7 days, no separation of solid and/or oily material shall occur.

5.2 Stability at elevated temperature (MT 46.3, CIPAC Handbook J, p.128, 2000)

After storage at $54 \pm 2^\circ\text{C}$ for 14 days, the determined average active ingredient content must not be lower than 95% relative to the determined average content found before storage (Note 6) and the formulation shall continue to comply with the clause for:

- pH range (4.1).

Note 1 Although it is not included in this specification, the flash point of the product shall comply with national and/or international transport regulations for flammable materials.

Note 2 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 3 There are no relevant impurities to be controlled in products of the manufacturers identified in evaluation reports 333/2004, 333/2005 and 333/2006.2. However, becisthemic acid chloride [(1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxoyl chloride], sometimes spelt bicisthemic acid chloride, can occur as a result of certain manufacturing processes. If this impurity could occur at ≥ 1 g/kg (of deltamethrin) in the products of other manufacturers, it would be designated as a relevant impurity and a clause would be required to limit its concentration.

Note 4 Viscosity can be critically important for successful application of a UL formulation but the requirements are dependent upon both the formulation and the application technique or equipment. For this reason, no clause is provided for kinematic viscosity.

Note 5 Loss of droplet mass by volatilisation can be critical for UL formulations because, if the losses are significant, the proportion of the spray which drifts from the target, and the distance over which the drift occurs, is likely to increase. The volatilization and additional drift that occur in practice are dependent on the initial droplet size spectrum and the height through which droplets fall, the air temperature and wind speed. In addition, a degree of volatilization which may be unacceptable for one type of application may be of little or no consequence in another case. At present, no method is available to allow measurement of loss by volatilization to be related to the potential increase in drift and therefore no clause is provided for volatility.

Note 6 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.

WHO SPECIFICATIONS FOR PUBLIC HEALTH PESTICIDES

DELTAMETHRIN WATER DISPERSIBLE GRANULES

WHO specification 333/WG (April 2005*)

This specification, which is PART ONE of this publication, is based on an evaluation of data submitted by the manufacturers whose names are listed in the evaluation reports (333/2004, 333/2005, 333/2006.2). It should be applicable to relevant products of this manufacturer, and those of any other formulators who use only TC from the evaluated sources. The specification is not an endorsement of those products, nor a guarantee that they comply with the specification. The specification may not be appropriate for the products of other manufacturers who use TC from other sources. The evaluation reports (333/2004, 333/2005, 333/2006.2), as PART TWO, form an integral part of this publication.

1 Description

The material shall consist of a homogeneous mixture of technical deltamethrin, complying with the requirements of the WHO specification 333/TC (April 2005), together with carriers and any other necessary formulants. It shall be in the form of granules for application after disintegration and dispersion in water. The formulation shall be dry, free-flowing, essentially non-dusty, and free from visible extraneous matter and hard lumps.

2 Active ingredient

2.1 Identity tests (333/WG/M/2, CIPAC Handbook L, p.45, 2006, Note 1)

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

2.2 Deltamethrin content (333/WG/M/3, CIPAC Handbook L, p.45, 2006, Note 1)

The deltamethrin content shall be declared (g/kg) and, when determined, the average measured content shall not differ from that declared by more than the following tolerances:

Declared content, g/kg	Tolerance
up to 25	± 25% of the declared content
above 25 up to 100	± 10% of the declared content
above 100 up to 250	± 6% of the declared content

Note: the upper limit is included in each range

3 Relevant impurities (Note 2)

* Specifications may be revised and/or additional evaluations may be undertaken. Ensure the use of current versions by checking at: <http://www.who.int/quality/en/>.

4 Physical properties

- 4.1 **Acidity** (MT 31, CIPAC Handbook F, p.96, 1995)
Maximum acidity: 5 g/kg calculated as H₂SO₄.
- 4.2 **Wettability** (MT 53.3, CIPAC Handbook F, p.165, 1995)
The formulation shall be completely wetted in 1 min without swirling.
- 4.3 **Wet sieve test** (MT 167, CIPAC Handbook F, p.416, 1995)
Maximum: 1% retained on a 75 µm test sieve.
- 4.4 **Degree of dispersion** (MT 174, CIPAC Handbook F, p.435, 1995)
Dispersibility: minimum 90% after 2 min stirring.
- 4.5 **Suspensibility** (MT 168, CIPAC Handbook F, p.417, 1995) (Note 3)
A minimum of 70% shall be in suspension after 30 minutes in CIPAC Standard Water D at 30 ± 2°C.
- 4.6 **Persistent foam** (MT 47.2, CIPAC Handbook F, p.152, 1995)
Maximum: 40 ml after 1 min.
- 4.7 **Dustiness** (MT 171, CIPAC Handbook F, p.425, 1995) (Note 4)
Essentially non-dusty.
- 4.8 **Flowability** (MT 172, CIPAC Handbook F, p.430, 1995)
At least 99% of the formulation shall pass through a 5 mm test sieve after 20 drops of the sieve.

5 Storage stability

- 5.1 **Stability at elevated temperature** (MT 46.3, CIPAC Handbook J, p.128, 2000)
After storage at 54 ± 2°C for 14 days, the determined average active ingredient content must not be lower than 95% relative to the determined average content found before storage (Note 5) and the formulation shall continue to comply with the clauses for:
- acidity (4.1);
 - wet sieve test (4.3);
 - degree of dispersion (4.4);
 - suspensibility (4.5);
 - dustiness (4.7).

Note 1 The collaboratively tested method for analysis of wettable powders (WP) is applicable to water dispersible granules (WG). It is, however, recommended that samples be ground to a powder prior to analysis.

Note 2 There are no relevant impurities to be controlled in products of the manufacturers identified in evaluation reports 333/2004, 333/2005 and 333/2006.2. However, becisthemic acid chloride [(1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxoyl chloride], sometimes spelt bicisthemic acid chloride, can occur as a result of certain manufacturing processes. If this impurity could occur at ≥1 g/kg (of deltamethrin) in the products of other manufacturers, it

would be designated as a relevant impurity and a clause would be required to limit its concentration.

Note 3 Chemical assay is the only fully reliable method to measure the mass of active ingredient still in suspension. However, the simpler gravimetric method, MT 168, may be used on a routine basis provided that it has been shown to give equal results to those of chemical assay. In case of dispute, chemical assay shall be the "referee method".

Note 4 Measurement of dustiness must be carried out on the sample "as received" and, where practicable, the sample should be taken from a newly opened container, because changes in the water content of samples may influence dustiness significantly. The optical method, MT 171, usually shows good correlation with the gravimetric method and can, therefore, be used as an alternative where the equipment is available. Where the correlation is in doubt, it must be checked with the formulation to be tested. In case of dispute the gravimetric method shall be used.

Note 5 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.

WHO SPECIFICATIONS FOR PUBLIC HEALTH PESTICIDES

DELTAMETHRIN EMULSION, OIL IN WATER

WHO specification 333/EW (April 2006*)

This specification, which is PART ONE of this publication, is based on an evaluation of data submitted by the manufacturers whose names are listed in the evaluation reports (333/2004, 333/2005, 333/2006.2). It should be applicable to relevant products of this manufacturer, and those of any other formulators who use only TC from the evaluated sources. The specification is not an endorsement of those products, nor a guarantee that they comply with the specification. The specification may not be appropriate for the products of other manufacturers who use TC from other sources. The evaluation reports (333/2004, 333/2005, 333/2006.2), as PART TWO, form an integral part of this publication.

1 Description

The formulation shall consist of an emulsion of technical deltamethrin, complying with the requirements of WHO specification 333/TC (April 2005), in an aqueous phase together with suitable formulants. After gentle agitation, the formulation shall be homogeneous (Note 1) and suitable for dilution in water.

2 Active ingredient

2.1 Identity tests (333/EW/M/2, CIPAC Handbook L, p.45, 2006)

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

2.2 Deltamethrin content (333/EW/M/3, CIPAC Handbook L, p.45, 2006)

The deltamethrin content shall be declared (20 g/kg or g/l at $20 \pm 2^\circ\text{C}$, Note 2) and, when determined, the mean measured content shall not differ from that declared by more than $\pm 15\%$.

3 Relevant impurities (Note 3)

4 Physical properties

4.1 pH range (MT 75.3, CIPAC Handbook J, p.131, 2000)

The pH of an aqueous dispersion shall be 4.5 to 7.5.

4.2 Pourability (MT 148, CIPAC Handbook F, p.348, 1995)

Maximum "residue": 5%

* Specifications may be revised and/or additional evaluations may be undertaken. Ensure the use of current versions by checking at: <http://www.who.int/quality/en/>.

4.3 **Emulsion stability and re-emulsification** (MT 36.1, CIPAC Handbook F, p. 108, 1995) (Note 4)

The formulation, when diluted at $30 \pm 2^\circ\text{C}$ (Note 5) with CIPAC standard waters A and D, shall comply with the following:

Time after dilution	Limits of stability
0 h	Initial emulsification complete
0.5 h	Cream: none
2.0 h	"Cream": none "Free oil": none
24 h	Re-emulsification complete
24.5 h	"Cream": none "Free oil": none.

Note: tests at 24 h are required only where the results at 2 h are in doubt.

4.4 **Persistent foam** (MT 47.2, CIPAC Handbook F, p. 152, 1995) (Note 6)

Maximum: 20 ml after 1 min.

5 **Storage stability**

5.1 **Stability at 0°C** (MT 39.3, CIPAC Handbook J, p.126, 2000)

After storage at $0 \pm 2^\circ\text{C}$ for 7 days, no separation of particulate or oily matter shall be visible after gentle agitation.

5.2 **Stability at elevated temperature** (MT 46.3, CIPAC Handbook J, p.128, 2000)

After storage $54 \pm 2^\circ\text{C}$ for 14 days the determined average active ingredient content must not be lower than 95%, relative to the determined average content found before storage (Note 7), and the formulation shall continue to comply with the clauses for:

- pH range (4.1),
- emulsion stability and re-emulsification (4.3).

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

Before sampling to verify the formulation quality, the commercial container must be inspected carefully. On standing, emulsions may develop a concentration gradient which could even result in the appearance of a clear liquid on the top (sedimentation of the emulsion) or on the bottom (creaming up of the emulsion). Therefore, before sampling, the formulation must be homogenised 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). Large containers must be opened and stirred adequately.

- Note 2 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 3 There are no relevant impurities to be controlled in products of the manufacturers identified in evaluation reports 333/2004, 333/2005 and 333/2006.2. However, becisthemic acid chloride [(1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxoyl chloride], sometimes spelt bicisthemic acid chloride, can occur as a result of certain manufacturing processes. If this impurity could occur at ≥1 g/kg (of deltamethrin) in the products of other manufacturers, it would be designated as a relevant impurity and a clause would be required to limit its concentration.
- Note 4 This test will normally be carried out only after the test of stability at elevated temperature (5.2).
- Note 5 The formulation should be tested at the highest and lowest rates of use recommended by the supplier.
- Note 6 The test should be carried out at the highest application concentration.
- Note 7 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.

WHO SPECIFICATIONS FOR PUBLIC HEALTH PESTICIDES

DELTAMETHRIN LONG-LASTING (COATED) INSECTICIDAL NET

WHO specification 333/LN (August 2006*)

This specification, which is PART ONE of this publication, is based on an evaluation of data submitted by the manufacturer whose name is listed in the evaluation report (333/2006.1). It should be applicable to relevant products of this manufacturer but it is not an endorsement of those products, nor a guarantee that they comply with the specification. The specification may not be appropriate for the products of other manufacturers, irrespective of the source of TC. The evaluation report 333/2006.1, given in PART TWO, forms an integral part of this publication.

1 Description

The material shall consist of netting (Note 1), formed from knitted poly-filament polyester fibres, treated with technical deltamethrin complying with WHO specification 333/TC (April 2005) together with any necessary stabilizers, plasticizers and other formulants. The product shall appear clean, free from visible extraneous matter (Note 2), free from damage (such as splitting or tearing) and visible manufacturing defects (such as poorly made seams or a weave that is either not uniform or too loose to remain uniform in use). It shall be suitable for use as an insecticidal net with long-lasting activity (Notes 3 & 4).

2 Active ingredient

2.1 Identity tests (CIPAC 333/LN/M/2) (Notes 5 & 6)

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

2.2 Total deltamethrin content (CIPAC 333/LN/M/3) (Notes 5, 6, 7 & 8)

The deltamethrin content shall be declared (0.9 g/kg in 150 denier yarn, 1.4 g/kg in 100 denier yarn, 1.8 g/kg in 75 denier yarn) and, when determined, the average content measured shall not differ from that declared by more than $\pm 25\%$.

2.3 Deltamethrin release characteristics (CIPAC Handbook, Note 9)

The measured average retention index of deltamethrin shall be not less than 80% (Note 10).

3 Relevant impurities (Note 11)

* Specifications may be revised and/or additional evaluations may be undertaken. Ensure the use of current versions by checking at: <http://www.who.int/quality/en/>.

4 Physical properties

4.1 Netting mesh size

When counted by the method given in Note 12, the netting shall have a minimum of 24 complete holes/cm² and an average in the range 24-26 complete holes/cm².

4.2 Dimensional stability of netting to washing (Note 13)

Dimensional stability (warp and weft): not exceeding $\pm 10\%$ of the initial dimensions.

4.3 Bursting strength (Note 14)

The minimum bursting strength of the fabric shall be declared (not less than 250, 350 or 400 kPa, respectively, for fabric made from 75, 100 or 150 denier yarn) and, when determined, the average shall be not less than that declared.

If seams are present, their average bursting strength shall be not less than the measured average for the fabric.

5 Storage stability

5.1 Stability at elevated temperature (MT 46.3, CIPAC Handbook J, p.128, 2000)

After storage at $40 \pm 2^\circ\text{C}$ for 8 weeks (Note 15), the determined average active ingredient content must not be lower than 95% of the average content found before storage (Note 16) and the netting shall continue to comply with the clauses for:

- retention index (2.3);
- dimensional stability (4.2);
- bursting strength (4.3).

Note 1 The specification applies to netting, in bulk, and to made up bed nets, which may be rectangular or circular in design. The nets or netting may be white or uniformly coloured, for example, yellow, pink, khaki or light brown, blue or dark blue, green or dark green.

Note 2 Occasional short lengths of loose thread present in made up nets are not considered to be extraneous matter.

Note 3 A long-lasting insecticidal net is expected to retain insecticidal activity during its life span and through a number of washes. The clause for deltamethrin release characteristics (2.3) is based on a model washing regime and compliance with the limit does not guarantee that activity will be retained through any particular number of washes performed according to local practice.

Note 4 Flammability of the product is not part of the specification but it should be measured by the manufacturer, according to 16CFR Part 1610, and the result presented on the package.

The linear density (denier or decitex) of the fibres cannot be measured in the manufactured net but it should be identified on the packaging.

Note 5 For complete identification and good quantification, deltamethrin (i.e. the [S,1R,3R]-isomer) must be separated from the [R,1R,3R]-isomer (otherwise known as the R-isomer), which is not part of the active ingredient and not a relevant impurity. The isomers may be separated by non-chiral techniques.

Note 6 Methods for the identification and determination of deltamethrin content in various formulations are available in CIPAC Handbook L, p.45, 2006. Extension of the method to

LN was adopted by CIPAC in 2006, but the extension is not yet published in a Handbook. Prior to publication of the Handbook, copies of the method for analysis of LN may be obtained through the CIPAC website, <http://www.cipac.org>.

- Note 7** Each net is expected to conform to the specification and, for the purposes of testing for compliance, material from two or more nets must not be mixed. Samples for testing should be taken in such a way as to be representative of the net or netting. Unless stated otherwise for the test, samples should be assembled from pieces taken from nets as shown in Figure 1. Samples from bulk netting should be assembled from similar pieces taken along a suitable diagonal across the netting. The sizes of pieces should be appropriate to the tests required and sufficient for both pre- and post-storage stability tests. Samples for chemical analysis may be cut into 1-2cm squares and thoroughly mixed, to provide representative test portions (this is not appropriate for tests of some physical properties).
- Note 8** The declared values for deltamethrin content in g/kg are equivalent to 55 mg/m² in each case. The deltamethrin content may be declared as both g/kg and mg/m² but, in cases of dispute, g/kg values shall be used. If the active ingredient content is also specified as mg/m² of netting, the actual content on this basis is calculated from the measured values for active ingredient content in g/kg and mass of net/m². Mass of net/m² should be determined according to ISO 3801 (1977).
- Note 9** The test method for determination of deltamethrin retention index was adopted by CIPAC in 2006 but it is not yet published in a Handbook. Prior to publication of the Handbook, copies of the method may be obtained through the CIPAC website, <http://www.cipac.org>.
- Note 10** The minimum of 80% retention index applies to the average obtained from triplicate tests performed on samples removed from the same net or batch of netting.
- Note 11** There are no relevant impurities to be controlled in products of the manufacturer identified in evaluation reports 333/2004, 333/2005 and 333/2006.2. However, becisthemic acid chloride [(1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxyl chloride], sometimes spelt bicisthemic acid chloride, can occur as a result of certain manufacturing processes. If this impurity could occur at ≥1 g/kg (of deltamethrin) in the products of other manufacturers, it would be designated as a relevant impurity and a clause would be required to limit its concentration.
- Note 12** Mesh size is determined by counting the number of holes in a square of the fabric. Counting may be done directly on the fabric or indirectly by scanning/photocopying the fabric. Indirect methods may ease counting and provide a permanent record. Before counting, the fabric should be conditioned according to ISO 139 (1973) (4 h, 20°C, 65% relative humidity).
- Use a template to define the square of netting, taking care not to stretch or distort the fabric. The template should be a rigid sheet, 1-2 mm thick, in or on which an accurately calibrated (±1% in each dimension) square (e.g. 2 x 2 or 5 x 5 cm) has been cut or marked. If a template is not available and a ruler must be used, great care is required to ensure that the area counted is square. If practicable, at least one edge of the square to be counted should be aligned with a row of complete holes in the fabric. Count 5 replicate squares in pieces selected according to figure 1, calculate the average and note the lowest value.
- Note 13** Dimensional stability is determined as follows. Method of preparation: ISO 3759 (1995, under revision in 2006). Method of washing: ISO 6330 (2001). Method of calculation: ISO 5077 (1984). Size of test portions: 500 x 500 mm; mark off 350 x 350 mm within the test portion. Test a total of 4 replicate portions, duplicates washed in each of 2 separate loads. Type of washing machine: ISO type A (front loading). Washing programme: ISO type 6A (1 main wash, 3 rinses, 40°C). Fill the washer with dummy load (with fabric as per ISO standard) up to the standard of 2 to 4 kg. Each test portion should be washed in a bag, to avoid stretching of the fabric. Drying: flat drying.
- Note 14** ISO 13938 part 1 (1999) and ISO 13938 part 2 (1999), with conditioning of the fabric as specified in the ISO standard. Results should be based tests of 7.3 cm² areas of fabric. Five replicate tests should be conducted on samples taken according to Figure 1 and the average calculated.

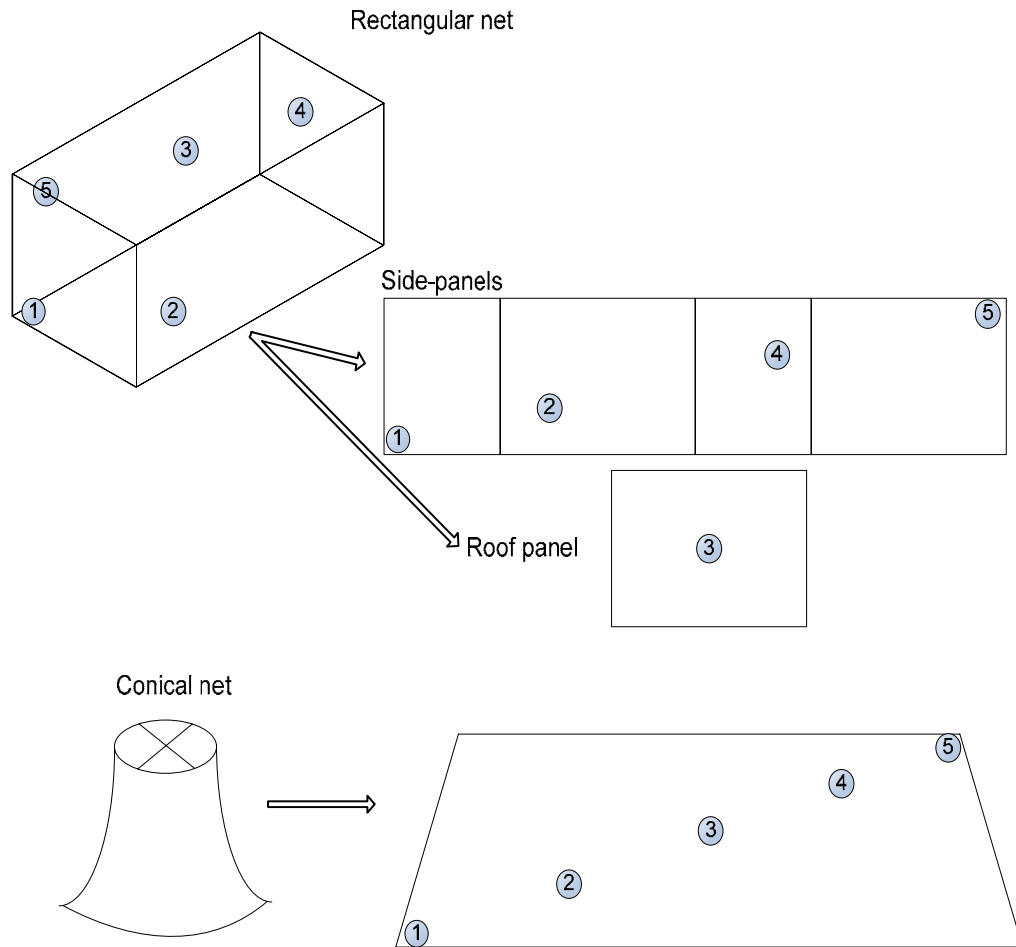
The method for testing seam bursting strength is identical to that used to test the fabric, except that 5 replicate tests should be performed with a seam centred on the test head. Up to 5 seams may be tested, to provide the total of 5 measurements.

Note 15 The LN has long-term stability at temperatures up to and about 40°C. Conversion of deltamethrin to the R-isomer may occur at higher temperatures, particularly above 50°C, and the LN should be kept away from direct sunlight and heat sources. The test should be conducted on samples without the presence of packaging material.

Note 16 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.

Figure 1 General method for sampling rectangular and conical nets

The sample should consist of 5 pieces, taken approximately according to the pattern shown in the diagram below. Sufficient material should be taken for all tests and thus the pattern indicated is intended only for general guidance. For most tests, pieces should be taken at least 15cm from seams or selvages, avoiding folded and creased areas, so as to represent the netting material as fully as possible. To test the bursting strength of seams (clause 3.3), up to 5 seams may be tested, avoiding intersections, to provide a total of 5 measurements.



PART TWO

EVALUATION REPORTS

DELTAMETHRIN

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WHO SPECIFICATIONS FOR PUBLIC HEALTH PESTICIDES

DELTAMETHRIN

FAO/WHO EVALUATION REPORT 333/2006.2

Recommendations

The Meeting recommended the following.

- (i) The existing WHO specifications for deltamethrin TC, DP, SC, EC WG, WP, UL and EW should be extended to encompass the products of Heranba Industries Ltd*.
- (ii) The existing FAO specifications for deltamethrin TC, DP, WP, SC, EC, UL, WG, EG and EW should be extended to encompass the products of Heranba Industries Ltd.

Appraisal

The Meeting considered data and information submitted by Heranba Industries Ltd (Mumbai, India) in support of extension of the existing FAO and WHO specifications.

The Meeting was provided by Heranba with commercially confidential information on the manufacturing process, the manufacturing specification and 5-batch analysis data for all detectable impurities. The batch data were derived from analyses conducted in two different laboratories on the same 5 batches. Data from the 1st laboratory addressed the content of active ingredient and all impurities with manufacturing specifications ≥ 1 g/kg. Data from the 2nd laboratory primarily addressed the content of deltamethrin. Although there was an apparent slight bias between the two series of data for deltamethrin content (988, 983 g/kg averages), the differences (-3.5 to -5.9 g/kg) were within the reproducibility ($R = 17$ g/kg at 998 g/kg) expected from the current CIPAC method (CIPAC L). The manufacturer proposed that the deltamethrin data from the 2nd laboratory should be utilized in the equivalence determination. Although these values were slightly lower than those from the first laboratory, mass balances were $\geq 99.4\%$ with no unknowns detected. The confidential information was confirmed as being similar to that submitted for registration in Taiwan.

The manufacturing specification indicated non-equivalence with the reference profile of impurities, with respect to bicyclic acid anhydride [(1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxylic anhydride]. A similar issue had been considered by the 2005 JMPS (FAO/WHO evaluation report 333/2005, below) and WHO had advised that the impurity should not be considered relevant. Bicyclic acid chloride [(1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxyl chloride] was also present in the Heranba profile but, as the levels were < 1 g/kg, it remained a non-relevant impurity, on the same basis as that decided by the 2005 JMPS. The studies on acute toxicology, submitted by Heranba, were

* Note: extension of the existing WHO specification for deltamethrin LN was neither proposed by the company nor considered by the Meeting, as part of evaluation 333/2006.2.

considered by WHO/PCS (PCS 2006) to indicate equivalence of toxicology (FAO/WHO 2006) with the data supporting the reference profile. The Meeting therefore concluded that Heranba deltamethrin TC should be considered equivalent to that on which the existing specifications are based (FAO/WHO evaluation report 333/2004, below).

Heranba confirmed that the deltamethrin TC and formulations produced by the company comply with existing FAO and WHO specifications. The company also stated that existing CIPAC methods for identification and determination of deltamethrin were appropriate for the analysis of the company's TC and formulations.

Heranba proposed a specification for deltamethrin WT. A specification for WT had been proposed in 2004 by Bayer CropScience (see evaluation report 333/2004) but was not adopted, due to the lack of a suitably validated analytical method and unresolved problems regarding physical characteristics and the corresponding test methods. These issues were finally resolved by Bayer CropScience when an analytical method for deltamethrin in WT was adopted by CIPAC in 2006. A revised specification for WT is scheduled for JMPS evaluation in 2007 and a decision on the 2006 Heranba proposal for extension of the WT specification was therefore deferred.

**SUPPORTING INFORMATION
FOR
EVALUATION REPORT 333/2006.2**

Physico-chemical properties of deltamethrin

Table 1. Physico-chemical properties of pure deltamethrin

Parameter	Value(s) and conditions	Purity %	Method	Reference
Vapour pressure	1.24 x 10 ⁻⁵ mPa at 25°C	98	OECD 104	HIL/D12/02
Melting point	100°C	98	CIPAC MT 2	HIL/D12/03
Temperature of decomposition	>270°C	98	CIPAC MT 2	HIL/D12/03
Solubility in water	<0.1 mg/l at 25°C, pH not stated	98	CIPAC MT 157.2	HIL/D12/04

Table 2. Chemical composition and properties of technical deltamethrin (TC)

Manufacturing process, maximum limits for impurities ≥1 g/kg, 5 batch analysis data	Confidential information supplied and held on file by FAO and WHO. Mass balances were 99.81-99.84%, with no unknowns.
Declared minimum deltamethrin content	985 g/kg
Relevant impurities ≥ 1 g/kg and maximum limits for them	None
Relevant impurities < 1 g/kg and maximum limits for them	None
Stabilisers or other additives and maximum limits for them	None
Melting temperature of the TC	98-101°C

Formulations

Heranba deltamethrin formulations are registered and sold in Malaysia, Taiwan and Thailand.

Methods of analysis and testing

Heranba confirmed that the existing CIPAC methods for the determination of active ingredient content and for testing physical properties are satisfactory for use with their products.

ANNEX 1

HAZARD SUMMARY PROVIDED BY THE PROPOSER

Note: Heranba Industries Ltd provided written confirmation that the toxicological data included in the following summary were derived from deltamethrin having impurity profiles similar to those referred to in Table 2, above.

Table A. Toxicology profile of Heranba deltamethrin technical material, based on acute toxicity, irritation and sensitization

Species	Test	Duration and conditions	Result	Reference
Rat (m,f)	Oral	OECD 401 Purity 98.5%	LD ₅₀ (m) = 324 (305-343) mg/kg bw LD ₅₀ (f) = 341 (309-372) mg/kg bw	8400
Rat (m,f)	Dermal	OECD 402 Purity 98.5%	LD ₅₀ >2000 mg/kg bw	8402
Rat (m,f)	Inhalation	OECD 403 Purity 98.5%	LC ₅₀ = 3.1 (2.3-4.2) mg a.i./l	8517
Rabbit (m,f)	Skin irritation	OECD 404 Purity 98.5%	Non-irritant	8403
Rabbit (m,f)	Eye irritation	OECD 405 Purity 98.5%	Not irritant	8404
Guinea pig (m)	Skin sensitization	OECD 406 Purity 98.5%	Non-sensitizer	8484

Table B. Mutagenicity profile of Heranba deltamethrin technical material, based on *in vitro* and *in vivo* studies

Species	Test	Duration and conditions	Result	Reference
<i>Salmonella typhimurium</i>	Ames test, reverse mutation assay (<i>in vitro</i>)	OECD 471; ±S9 metabolic activation; 0.5, 5, 50 and 5000 µg/plate mixed in DMSO. Deltamethrin purity 98.5%	Non-mutagenic	8525
<i>Saccharomyces cerevisiae</i>	Gene mutation	OECD 480; ±S9 metabolic activation; up to 5000 µg/plate. Deltamethrin purity 98.5%	Non-mutagenic	8528
<i>Mus musculus</i> (Swiss albino)	Mouse bone marrow cytogenetic assay (chromosomal aberration)	OECD 475; 10, 5, 2.5 mg/kg b.w. triethylenemelamine (TEM) dose at 1.0 mg/kg b.w. via split-dose i.p. injection. Deltamethrin purity 98.5%	Non-mutagenic	8527

Table C. Ecotoxicology profile of Heranba deltamethrin technical material

Species	Test	Duration and conditions	Result	Reference
Water flea, <i>Daphnia magna</i>	Acute immobilization test	24 h. OECD 202, purity 98.5%	EC ₅₀ = 0.45 µg/l	8436
Freshwater fish, <i>Poecilia reticulata</i>	Acute toxicity	24-96 h. OECD 203, purity 98.5%	LC ₅₀ (24 h) >1.6 µg/l LC ₅₀ (48 h) >1.6 µg/l LC ₅₀ (72 h) = 1.17 µg/l LC ₅₀ (96 h) 0.56 µg/l	8433
Japanese quail, <i>Coturnix coturnix japonica</i>	Acute dietary toxicity	8 days, OECD 205, purity 98.5%	LC ₅₀ = 545.51 ppm	8435

ANNEX 2. REFERENCES

Heranba document number	Year and title of report or publication details
8400	2001. Acute oral toxicity study in Wistar rats.
8402	2001. Acute dermal toxicity study in Wistar rats.
8403	2001. Study on Primary skin irritation in Himalayan albino Rabbits.
8404	2001. Study on irritation to mucous membrane* in Himalayan albino Rabbits.
8433	2001. Acute toxicity to freshwater fish.
8435	2001. Acute dietary toxicity study in Japanese quail.
8436	2001. Acute immobilization test with deltamethrin technical in <i>Daphnia magna</i> .
8484	2001. Skin sensitization potential in guinea pig.
8517	2001. Acute inhalation study in Wistar rats.
8525	2001. Mutagenicity evaluation by Ames <i>Salmonella typhimurium</i> -Reverse Mutation assay.
8527	2001. Mutagenicity evaluation by mouse bone marrow cytogenetic assay Chromosomal aberration.
8528	2001. Mutagenicity evaluation: Induction of gene mutation in Yeast – <i>Saccharomyces cerevisiae</i> .
CIPAC L	Collaborative International Pesticides Analytical Council (CIPAC). Deltamethrin. Handbook L, p. 45. Harpenden, U.K., 2006.
FAO/WHO 2006	Manual on development and use of FAO and WHO specifications for Pesticides. March 2006 revision, published in the internet at http://www.fao.org/ag/agp/agpp/pesticid/ and http://www.who.int/quality/en/ .
HIL/D12/02	2002. Study on vapor pressure of deltamethrin technical.
HIL/D12/03	2003. Study on melting point of deltamethrin technical.
HIL/D12/04	2003. Study on solubility in water of deltamethrin technical.
PCS 2006	2006. JMPS specification for Heranba Industries deltamethrin. Assessment prepared for PCS and submitted to WHOPES on 8 September, 2006.

* Refers to the eye.

WHO SPECIFICATIONS FOR PUBLIC HEALTH PESTICIDES

DELTAMETHRIN

FAO/WHO EVALUATION REPORT 333/2006.1

Recommendations

The Meeting recommended that:

- (i) the specification for deltamethrin LN (coated type) proposed by Vestergaard-Frandsen, as amended, should be adopted by WHO;
- (ii) future proposals for extension of this specification to apparently equivalent LN formulations should be supported by evidence to show whether or not the test method and limit for active ingredient release characteristics are appropriate for the additional products.

Appraisal

Supporting data and draft specifications for deltamethrin LN, provided by Vestergaard-Frandsen, were considered by the JMPS for development of a new WHO specification in 2004, 2005 and 2006. Appropriate clauses, limits and methods of testing for certain parameters of this new type of formulation were also developed by the company over this period.

Deltamethrin-treated long-lasting insecticidal nets (LN) were tested/evaluated by WHOPES and an interim recommendation for their use in malaria prevention and control was issued (WHO 2004). A provisional specification guideline for LN formulations was accepted by the 2004 JMPS and published by WHO. The guideline for LN was subsequently amended, taking into account the recommendations of a WHO consultation on mosquito nets (WHO 2005) and published in the revised manual (FAO/WHO 2006). However, the apparent diversity of LN technologies currently requires specification clauses and limits to be tailored to individual products. The LN under consideration is a warp-knitted fabric in which the active ingredient resides mainly within a coating applied to yarn filaments. The yarn is a poly-filament type, because with this type of LN it is important to maximize surface area, rather than yarn volume, per unit area of fabric.

The manufacturer confirmed that the active ingredient incorporated into the LN was from a source compliant with the existing WHO specification for deltamethrin TC.

Description clause

The Meeting and manufacturer agreed that the specification should be applied to white or coloured fabrics made from 75, 100 or 150 denier yarn and that it should apply equally to made up, ready-to-use nets (rectangular and circular) or to netting in bulk.

Active ingredient content clause

With the aim of achieving similar levels of efficacy, the target dose of deltamethrin for all weights of fabric is similar on an area basis (55 mg/m^2) but it differs when expressed as g/kg. The mg/m^2 value is calculated from measurements of g/kg and

fabric density in g/m². The Meeting agreed that the declared and tolerance values should be based on g/kg, with the corresponding mg/m² value being defined in a Note to the specification.

The Meeting observed that random variations in the distribution of deltamethrin may influence the variation in measured values. The manufacturer initially provided between- and within-batch data based on analysis of single 100 cm² pieces but variations in deltamethrin distribution made it difficult to interpret individual results. Subsequently, larger samples were prepared from material taken from multiple positions, cut into approximately 2 x 2 cm pieces, mixed and sub-sampled for analysis. The manufacturer then showed that analysis of portions equivalent to 100 cm² provided a precision similar to that of portions which were 3 or 5 times larger but otherwise produced similarly. This procedure provided good precision (RSD <2% in one experiment).

The analytical method for determination of the deltamethrin in LN was validated as an extension of the existing CIPAC method for deltamethrin and adopted by CIPAC in 2006.

Relevant impurities clause

There are no relevant impurities identified in the existing WHO specification for deltamethrin TC. During the production of deltamethrin LN, a small proportion of conversion to the *R*-isomer can occur and this must be controlled by the manufacturer to avoid potentially significant losses of active ingredient. The isomerization can also occur if the LN is exposed to excessive heat during storage or use. The Meeting agreed that the *R*-isomer should remain designated as a non-relevant impurity and hence remain excluded from the specification.

Deltamethrin retention index clause

An adequate amount of active ingredient must be present on the surface of the LN, for efficacy, but the majority must reside within the coating of the LN, to avoid excessive losses during washing and to provide a reservoir from which the surface is replenished with active ingredient. The depth and quality of coating therefore strongly influence the retention of deltamethrin when the LN is subjected to repeated washing. The manufacturer provided data showing that approximately constant proportions of the remaining deltamethrin are removed from the polymer by successive washings with aqueous detergent. Broadly, the coating behaves rather like a solid solution of deltamethrin, with diffusion within the coating occurring much more slowly than the rate at which the deltamethrin can be removed from its surface, emulsified by aqueous detergent. Thus, following rapid depletion of the surface deltamethrin by washing, a short "curing" period at room temperature (or 2 h at 40°C) is required, to re-establish an equilibrium level of the active ingredient on the surface of polymer.

Depletion of total active ingredient content by washing (retention index) is most easily accomplished by analysis of separate washed and unwashed pieces of the same fabric. However, this may be expected to introduce additional sampling (i.e. sub-sampling) error into the measurement because, unlike most other formulation types, LN products cannot be mixed thoroughly prior to testing. As the diffusion behaviour within the polymer appears to be simple, it may be thought that the influence of the additional sub-sampling error could be minimized by a test involving repetitive washing but the available data did not support this approach. The

manufacturer's data from single- and multi-wash tests showed little difference in the dispersion of results for retention index per wash, indicating that other variables may be more important than sub-sampling error.

A single-wash test for determination of retention index was therefore developed by the manufacturer and validated by collaborative study, through CIPAC in 2006. The method was subsequently modified to calculate retention index as the average of triplicate determinations, to enhance repeatability and hence reproducibility of results.

Physical properties clauses

The clauses for physical properties mostly specify ISO methods, with the exception of netting mesh size, which does not require standardization, and deltamethrin retention index, which relates to the active ingredient. Where the ISO standard provides alternative methods, the option to be used is specified in a Note to the specification.

The Meeting accepted the manufacturer's explanation that the test for bursting strength obviated the need for a separate clause for tearing strength.

The Meeting noted that the $\pm 10\%$ tolerance on dimensional stability to washing was higher than the standard 5% given in the LN guideline (FAO/WHO 2006). The manufacturer explained that the manufacturing process is such that dimensional stability and deltamethrin retention index are inversely related. The Meeting therefore agreed that the 10% tolerance was an acceptable compromise.

Storage stability clause

Although deltamethrin in bulk is stable over a very wide temperature range and has very low volatility, in the LN it behaves differently. The manufacturer stated that deltamethrin in the LN has better stability at 40-47°C than at 54°C and its concentration remains almost unchanged after 2 years at 40°C. At 54°C for 14 days, about 15% of the deltamethrin was lost, mainly due to conversion to the *R*-isomer. Above 80°C, deltamethrin is rapidly and completely lost from the LN, apparently due to volatilization. At 40°C for 8 weeks, the loss of deltamethrin was minimal ($\leq 0.5\%$ conversion to *R*-isomer) and the physical properties of the fabric were maintained. The Meeting accepted the need to specify use of the alternative test regime.

**SUPPORTING INFORMATION
FOR
EVALUATION REPORT 333/2006.1**

Uses

The LN is intended for personal protection and disease vector control.

The formulation and its characteristics

The deltamethrin LN formulation characterized by the proposed specification is registered for use in Indonesia, Ethiopia, India, Kenya, Nigeria and Nicaragua and registration is in progress in Mexico, USA, Sri Lanka, Mali, Benin, Ghana, Brazil, Columbia, Malaysia, Peru, Columbia, Tanzania and Vietnam. The product is a fabric, made of warp-knitted multi-filament polyester fibres bearing a bonded layer of polymer containing deltamethrin.

The netting is available in three fabric densities, 30, 40 and 60 g/m² corresponding to the use of 75, 100 and 150 denier yarns, and may be white or coloured.

The netting is normally sold made up into ready-to-use bed nets, which are distributed in plastic bags, to minimize contamination with filth or water. The specification applies to rectangular and circular bed nets, as well as to the netting in bulk (which may be incorporated into other products).

ANNEX 1: REFERENCES

Reference	Full reference
FAO/WHO 2006	Manual on development and use of FAO and WHO specifications for pesticides, March 2006 revision of the 1 st edition. FAO, Rome, March 2006; WHO, Geneva, March 2006 (internet publications).
WHO 2004	Report of the Seventh WHOPES Working Group Meeting, WHO/HQ, Geneva, 2-4 December 2003. Geneva, World Health Organization, document WHO/CDS/WHOPES/2004.8.
WHO 2005	Technical consultation on specifications and quality control of netting materials for mosquito nets (untreated and treated). 29 November–02 December 2005. WHO Headquarters, Geneva, Switzerland.

WHO SPECIFICATIONS FOR PUBLIC HEALTH PESTICIDES

DELTAMETHRIN

FAO/WHO EVALUATION REPORT 333/2005

Recommendations

The Meeting recommended the following.

- (i) The existing (April 2005) WHO specifications for deltamethrin TC, DP, SC, EC WG, and existing (September 2005) WHO specifications for deltamethrin WP, UL, together with the previously unpublished specification for deltamethrin EW (all originally based upon data submitted by Bayer CropScience) should be amended to include a footnote regarding the potentially relevant impurity, becisthemic acid chloride, and extended to encompass the products of Tagros (India) and Argos (South Africa). WHOPEs evaluation of the EW having been completed satisfactorily, this specification should now be adopted by WHO.
- (ii) The existing (May 2005) FAO specifications for deltamethrin TC, DP, SC, EC and WG, together with the previously unpublished specifications for deltamethrin EW, EG, WP and UL (all based upon data submitted by Bayer CropScience) should be amended to include a footnote regarding the potentially relevant impurity, becisthemic acid chloride¹, and extended to encompass the products of Tagros (India) and Argos (South Africa). Analytical methods for determination of deltamethrin in EW, EG, WP and UL having been adopted by CIPAC, these specifications should now be adopted by FAO.

Appraisal

The Meeting considered data and information submitted by Tagros (India) and Argos (South Africa), for extension of: (i) existing FAO specifications for deltamethrin TC, DP, SC, EC and WG (May 2005); (ii) existing WHO specifications for TC, DP, SC, EC, WG (April 2005); (iii) existing WHO specifications for WP and UL (September 2005); (iv) pending FAO specifications for EG, EW, UL and WP; and (v) a pending WHO specification for EW.

Argos provided written confirmation that their products (currently WP only) contain only deltamethrin sourced from Tagros and therefore the Meeting agreed that decisions and recommendations relating to Tagros would apply also to Argos.

The Meeting was provided by Tagros with commercially confidential information on the manufacturing process and batch analysis data on all detectable impurities. Mass balances were very high (99.91–100.10%), with no unknowns detected. The UK Health & Safety Executive (HSE) confirmed that there were no significant differences between the data submitted to FAO/WHO and those submitted for registration in the UK.

In evaluating the equivalence of Tagros and Bayer TCs, the Meeting considered two potentially relevant impurities, becisthemic acid anhydride [(1*R*,3*R*)-3-(2,2-

¹ The name “becisthemic” is sometimes spelt “bicisthemic” but the common spelling is used here.

dibromovinyl)-2,2-dimethylcyclopropane carboxylic anhydride] and becisthemic acid chloride [(1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxyl chloride]. Neither impurity occurred in the Bayer TC ≥ 1 g/kg but, initially, the Tagros manufacturing limits were 10 g/kg and 2 g/kg, respectively. The acute toxicology data submitted by Tagros suggested that these higher limits might be reflected in the classification of the company's TC as a mild irritant of eyes. The data for the acid anhydride were complicated by the fact that the analytical method did not distinguish between the anhydride and becisthemic acid and the analytical method used for determination of becisthemic acid chloride was based on titration and appeared to lack specificity.

WHO/PCS secretariat assessed the results of eye irritation studies conducted (to GLP and according to OECD guidelines 404 and 405) by Tagros and concluded that, as defined by GHS (GHS 2003), the Tagros TC should not be classified as an eye irritant (PCS 2005a). WHO/PCS also concluded that, on the basis of toxicology, the Tagros TC should be considered equivalent to that of Bayer and the Meeting agreed.

No information was available on the toxicity of becisthemic anhydride but WHO/PCS concluded that it should not be considered a relevant impurity (PCS 2005a). Limited experimental data had indicated that a structural analogue of it, namely chrysanthemic anhydride, is sensitizing to the skin and, applying the precautionary principle, JMPS had previously concluded that the analogue should be considered a relevant impurity in *d*-allethrin (JMPS 2002). However, when assessing the relevance of a chlorine analogue of becisthemic anhydride (namely 2,2-dimethyl-3-(2,2'-dichlorovinyl) cyclopropane carboxylic acid anhydride), it was considered that there was no justification for declaring it a relevant impurity. That is, the structure-activity relationship was not considered to be strong enough to extend the weak data for sensitization from the non-halogenated chrysanthemic anhydride to the dichloro analogue of becisthemic acid anhydride (PCS 2005b). In keeping with this rationale, PCS considered that the structural relationship between chrysanthemic anhydride and becisthemic anhydride does not justify the classification of becisthemic anhydride as sensitizing. The conclusion was supported by the fact that Tagros deltamethrin TC was not sensitizing to the skin of Guinea pigs (studies performed according to the OECD guideline and GLP). The Meeting therefore agreed that becisthemic anhydride is not a relevant impurity in deltamethrin TC.

As in the case of the anhydride, no information was available on the toxicity of becisthemic acid chloride but WHO/PCS secretariat concluded that it should be considered a relevant impurity (PCS 2005a), if it occurred at ≥ 1 g/kg in the TC. WHO/PCS advised that, based on its chemical structure, becisthemic acid chloride is likely to be irritating (several acid chlorides are corrosive). WHO/PCS noted that, in the GHS classification, substances for which structure-activity or structure-response assessments indicate that they may be corrosive/irritant, should be classified as corrosive/irritant, in the absence of human or experimental data (GHS 2003). WHO/PCS thus recommended that becisthemic acid chloride should be considered as a relevant impurity. GHS guidelines (GHS 2003) indicate that when classifying a mixture, the data used should be derived primarily from studies on the mixture itself. As Tagros deltamethrin, apparently containing the becisthemic acid chloride at ≤ 2 g/kg, (also and becisthemic acid and anhydride at a sum concentration of ≤ 10 g/kg), was not irritating, WHO/PCS concluded that this limit could be accepted as the specification limit for becisthemic acid chloride. WHO/PCS noted that the conclusion is the same if the GHS second tier approach, classification by the concentration of

the impurity, is applied: a mixture is classified as corrosive if the sum concentration of corrosive components is $\geq 5\%$ and as an irritant if it is ≥ 1 but $< 5\%$.

Tagros then replaced the company's non-specific titrimetric procedure with an HPLC method, which was more specific for the determination of becisthemic acid chloride and which had a limit of quantification of 0.1 g/kg (Tagros 2006a, 2006b). In consequence, the manufacturer reported that levels of this impurity did not occur at up to 2 g/kg, as previously stated, but were actually < 1 g/kg. The Tagros manufacturing specification for this impurity was consequently lowered to < 1 g/kg. Although the stability of the impurity during analysis was not reported, the HPLC method did not seem likely to under-estimate the concentration of becisthemic acid chloride and the change in manufacturing specification from 2 to < 1 g/kg effectively changed the designation of this impurity to non-relevant. Consequently, there was no requirement for a specification clause to control its concentration. The new data had not been considered by a national registration authority.

The Meeting agreed that a cautionary footnote should be added to specifications, to indicate that becisthemic acid chloride could be a relevant impurity in the products of other manufacturers, if it occurred at ≥ 1 g/kg deltamethrin.

Overall, Tagros deltamethrin complied with the existing specification for TC but the becisthemic acid anhydride (not becisthemic acid chloride) indicated non-equivalence of the impurity profiles. However, the PCS assessment of this impurity and the available hazard data for Tagros deltamethrin TC showed no evidence to suggest that the Tagros TC presents greater or additional hazards compared with Bayer deltamethrin TC. Thus the Meeting concluded that the two TCs should be considered equivalent.

Tagros and Argos stated their products otherwise complied with the existing (September 2005) FAO and/or WHO specifications for TC, WP, EC and SC. The Meeting noted that development of the specifications for WT, proposed by Bayer and Tagros, cannot be addressed until suitable methods become available to test tablet hardness and friability.

CIPAC methods are available for identification and determination of deltamethrin in the TC and formulations, with the exception of WT for which the CIPAC method status is tentative. A more acceptable method is therefore required to support the proposed specification for WT.

The Meeting noted that WHOPES trials of deltamethrin EW had been successfully completed (WHO 2006).

**SUPPORTING INFORMATION
FOR
EVALUATION REPORT 333/2005**

Physico-chemical properties of deltamethrin

Table 1. Physico-chemical properties of pure deltamethrin (Tagros)

Parameter	Value(s) and conditions	Purity %	Method	References
Vapour pressure	1.24 X 10 ⁻⁸ Pa at 20°C	98.0	EEC A4	10803
Melting point	98-101°C	98.0	EEC A1, A2	10761
Temperature of decomposition	>300°C	98.0	EEC A1, A2	10761
Solubility in water, at 20°C	0.16 x 10 ⁻⁶ g/l at 20°C	98.0	OECD 105	10760
Octanol/water partition coefficient, at 23°C	log P K _{ow} = 4.61 at 25 ± 1°C	98.0	EEC A8	10806
Hydrolysis characteristics, at 25°C	Negligible hydrolysis at 50°C, pH 4.0 & 7.0 Half-life = 1.75 d at 40°C, pH 9.0 Half-life = 1.85 d at 30°C, pH 9.0	98.0	OECD 111	12430

Table 2. Chemical composition and properties of technical deltamethrin (TC)

Manufacturing process, maximum limits for impurities ≥ 1 g/kg, 5 batch analysis data	Confidential information supplied and held on file by FAO and WHO. Mass balances were 99.91-100.10%, with no unknowns.
Declared minimum deltamethrin content	985 g/kg
Relevant impurities ≥ 1 g/kg and maximum limits for them	None.
Relevant impurities < 1 g/kg and maximum limits for them	None
Stabilisers or other additives and maximum limits for them	None
Melting temperature of the TC	98-101°C

Formulations

Tagros deltamethrin TC is registered and sold in Taiwan, China, Korea, Australia South Africa, Ecuador and Spain; the EC formulation in Taiwan, Saudi Arabia, Azerbaijan, Kyrgystan, Cambodia, Turkmenistan, Sri Lanka and Romania; the SC formulation in Taiwan; the WP formulation in Sri Lanka, Azerbaijan and Sudan; and the ULV formulation in Lebanon.. Argos formulations are registered and sold in South Africa.

Methods of analysis and testing

Tagros and Argos confirmed that the existing CIPAC methods for the determination of active ingredient content and for testing physical properties are satisfactory for use with their products.

ANNEX 1

HAZARD SUMMARY PROVIDED BY THE PROPOSER

Note: Tagros provided written confirmation that the toxicological and ecotoxicological data included in the following summary were derived from deltamethrin having impurity profiles similar to those referred to in Table 2, above.

Table A. Toxicology profile of Tagros deltamethrin technical material, based on acute toxicity, irritation and sensitization

Species	Test	Duration and conditions	Result	References
Rat, Wistar, m	Acute oral	14 d obs., dose: 0, 500, 750 & 1000 mg/kg bw, vehicle vegetable oil, OECD 401, 99.2% purity	(i) LD ₅₀ = 612 mg/kg bw (ii) LD ₅₀ = 782 mg/kg bw	7719
Mouse Swiss albino, m & f	Acute oral	14 d obs., dose 0, 50, 100 & 150 mg/kg bw, vehicle vegetable oil, OECD 401, 99.2% purity	LD ₅₀ = 86 mg/kg bw (m) LD ₅₀ = 130 mg/kg bw (f)	7720
Rat, Wistar, m & f	Acute dermal	14 d obs., dose 0 & 2000 mg/kg bw OECD 402, 99.2% purity	LD ₅₀ >2000 mg/kg bw (m,f)	7721
Rat, Wistar, m & f	Acute inhalation	14 d obs., dose 0, 1.63, 3.11 & 4.39 mg/l, OECD 403, 99.2% purity	LC ₅₀ = 3.16 mg/l	8205
Rabbit	Skin irritation	Observation 1, 24, 48 & 72 h after treatment, dose: 500 mg (4 hours), OECD 404, 99.2% purity	non-irritant	7722
Rabbit	Eye irritation	Observation: 1, 24, 48 & 72 h after treatment, dose: 69 mg, OECD 405, 99.2% purity	mild irritant	7723
Guinea pig, Hartley	Skin sensitization	Observation: 28 d, dose 25 mg, OECD 406, 99.2% purity	non-sensitizer	7724

Deltamethrin has moderate to high acute toxicity when administered orally to the rat or mouse. Dullness, lethargy, mild tremor, salivation, diarrhoea and paralysis were observed in both male and female rats 2 h after treatment and persisted up to 24 h. In the rat, deltamethrin is less toxic by the dermal route but is highly toxic by inhalation. Deltamethrin is a mild-irritant to eye and non-irritant to skin of rabbits. It is not a skin sensitizer in the Guinea pig.

Table B. Toxicology profile of Tagros deltamethrin technical material, based on repeated administration (sub-acute to chronic)

Species	Test	Duration and conditions	Result	References
Rat	90 day oral	90 d, dose 0, 25, 50 & 75 mg/kg bw/d, OECD 408, purity 98%	NOAEL = 50 mg/kg bw/day	10380

Table C. Mutagenicity profile of Tagros deltamethrin technical material, based on *in vitro* and *in vivo* studies

Species	Test	Duration and conditions	Result	References
<i>Salmonella typhimurium</i> (TA 98, TA100, TA1535, TA1537)	Bacterial reverse mutation assay	Gaitonde Committee guideline 6.3.0 (1); dosage 0.5–5000 µg/plate, purity 98.65%	Negative	11270
TK6 human lymphoblastoid cells	Mammalian cell gene mutation test	Dosage 0.005, 0.01, 0.04 & 0.06 mM with S-9 mix; 0.005, 0.01, 0.02 & 0.04 mM without S-9 mix; OECD 476, purity 98.65%	Negative	10401
Human lymphocytes	DNA damage and repair	OECD 482; dosage 250, 500, & 1000 µg/plate, purity 98.65%	Negative	8181
Chinese hamster ovary (CHO) cells	Chromosomal aberration assay	Dosage 6, 7, & 8 µM with S-9 mix, 2, 4 & 6 µM without S-9 mix, OECD 473, purity 98.65%	Negative	10399
Mouse, Swiss Albino (m,f)	Mouse micronucleus assay	Gaitonde Committee guideline 6.3.0.2 (A), dosage 0,7.5,15,30 mg/kg, once or twice orally – 24h apart, purity 98.65%	Negative	11271
Mouse, Swiss Albino bone-marrow cells	Chromosomal aberration	Gaitonde Committee Guideline 6.3.0 (1), dosage: 0, 7.5, 15, 30 mg/kg b.w., once orally, purity 98.65%	Negative	11272
Mouse Swiss Albino (m, germinal cells)	Dominant lethal mutation	Gaitonde Committee Guideline 6.3.0.2 (A), dosage 0, 1.25, 2.5, 5 mg/kg b.w, once orally, purity 98.65%	Negative	11273

Table D. Ecotoxicology profile of Tagros deltamethrin technical material

Species	Test	Duration and conditions	Result	References
<i>Daphnia magna</i> (water flea)	Acute immobilization	Dosage: 1, 2, 4, 8 and 16 µg/l, 24 h, OECD 202, 99.2% purity	EC ₅₀ = 4.14 µg/l	7739
<i>Poecilia immobiliza</i> (freshwater fish)	Acute toxicity	Dosage: 0, 0.75, 1.13, 1.68, 2.53 and 3.79 µg/l, 96 h, OECD 203, 99.2% purity	LC ₅₀ = 1.74 µg/l	7737
<i>Chlorella vulgaris</i> (green algae)	Growth	72 h, OECD 201, 98.65% purity	EC ₅₀ = 22.77 µg/l	11442
<i>Lampito mauritii</i> (earthworm)	Acute toxicity	Dosage 150–1000 mg/kg dry soil, 14 d, OECD No.207, 99.2% purity	LC ₅₀ >1000 mg/kg dry soil	8321
<i>Apis cerana indica</i> (honey bee)	Acute contact toxicity	Dosage 0.05–1.20 ppm, 24 h, EPPO 170, 99.2% purity	LC ₅₀ = 0.52 ppm	8320
<i>Coturnix coturnix japonica</i> (Japanese quail)	Dietary toxicity	Dosage 1000, 2000, 3000, 4000 or 5000 ppm, 5 d, OECD 205, 99.2% purity	LC ₅₀ >5000 ppm	7738

Under laboratory conditions, deltamethrin is highly toxic for fish, aquatic arthropods, and honeybees. However, under field conditions, lasting adverse effects are not likely to occur under recommended conditions of use.

ANNEX 2. REFERENCES

Tagros document number or other reference	Year and title of report or publication details
7719	2000. Acute oral toxicity study with deltamethrin technical in Wistar rat.
7720	2000. Acute oral toxicity study with deltamethrin technical in Swiss albino mice.
7721	2000. Acute Dermal toxicity study with deltamethrin technical in Wistar rat.
7722	2000. A study on primary skin irritation of deltamethrin technical in Himalayan albino rabbits.
7723	2000. A study on irritation on mucous membrane of deltamethrin technical in Himalayan albino rabbits.
7724	2000. Skin sensitization potential of deltamethrin technical in Guinea pigs.
7737	2000. Acute toxicity of deltamethrin technical to freshwater fish, <i>Poecilia immobiliza</i> .
7738	2000. Dietary toxicity study with deltamethrin technical in Japanese quails.
7739	2000. Acute immobilization test with deltamethrin technical in <i>Daphnia magna</i> .
8181	2000. Mutagenicity evaluation of deltamethrin technical in human lymphocytes (DNA damage and repair – unscheduled synthesis).
8205	2000. Acute inhalation toxicity study with deltamethrin technical in Wistar rats.
8320	2001. Toxicity of deltamethrin technical to honey bee, <i>Apis indica</i> .
8321	2001. Toxicity of deltamethrin technical to earthworm, <i>Lampito mauritii</i> .
10380	2002. Sub-acute oral toxicity study with deltamethrin technical in Wistar rats.
10399	2002. Mutagenicity evaluation of deltamethrin technical – <i>in vitro</i> chromosomal aberration assay.
10401	2002. Mutagenicity evaluation of deltamethrin technical – <i>in vitro</i> mammalian cell gene mutation test.
10760	2002. Study on solubility of deltamethrin technical in water.
10761	2002. Study report on melting point, boiling point and relative density of deltamethrin technical.
10803	2002. Study report on vapour pressure of deltamethrin technical.
10806	2002. Study on partition co-efficient (<i>n</i> -octanol/water) of deltamethrin technical.
11270	2002. Mutagenicity evaluation of deltamethrin technical by Ames <i>Salmonella typhimurium</i> – reverse mutation assay.
11271	2002. Mutagenicity evaluation of deltamethrin technical – <i>in vivo</i> mouse micronucleus assay.
11272	2002. Mutagenicity evaluation of deltamethrin technical – <i>in vivo</i> by mouse bone marrow cytogenetic assay (chromosomal aberration)
11273	2002. Mutagenicity evaluation of deltamethrin technical – <i>in vivo</i> by dominant lethal test in mouse.
11442	2002. Effect of deltamethrin technical on the growth of green alga (<i>Chlorella vulgaris</i>).
12430	2002. Study on hydrolysis (abiotic) of deltamethrin technical.
GHS 2003	The Globally Harmonized System of Classification and Labelling of Chemicals (GHS) at: http://www.unece.org/trans/danger/publi/ghs/ghs_rev00/English/GHS-PART-3e.pdf , accessed on 9 September, 2005.
JMPS 2002	WHO Specifications and evaluations for public health pesticides. <i>d</i> -Allethrin. accessed at http://www.who.int/whopes/quality/en/dAllethrin_spec_eval_March_04.pdf on 12 September, 2005.
PCS 2005a	14 September 2005. Relevance of impurities in deltamethrin, and equivalence of two deltamethrin products.
PCS 2005b	11 January 2005. Dermal irritation and sensitization of permethrin.
Tagros 2006a	Method of analysis for the determination of bicisthemic acid content corresponding to deltamethrin content in deltamethrin technical. Tagros report on study No. 05063. E-mail sent to M. Zaim (WHO) and G Vaagt (FAO) on 24 February 2006.

Tagros document number or other reference	Year and title of report or publication details
Tagros 2006b	ReRe: Deltamethrin specifications E-mail sent to M. Zaim (WHO) and G Vaagt (FAO) on 01 March 2006.
WHO 2006	Report of the Ninth WHOPES Working Group Meeting. Geneva, World Health Organization, 2006 (WHO/CDS/NTD/WHOPES/2006.2).

WHO SPECIFICATIONS FOR PUBLIC HEALTH PESTICIDES

DELTAMETHRIN FAO/WHO EVALUATION REPORT 333/2004

Explanation

The data for deltamethrin were evaluated for a review of existing FAO specifications for TC, WP, EC, DP, UL (AGP: CP/243-1991) and WHO specifications for TC (WHO/SIT/24/R2-1999), WP (WHO/SIF/42/R2-1999), EC (WHO/SIF/43/R2-1999), DP (WHO/SIF/44/R2-1999), UL (WHO/SIF/46/R1-1999), SC (WHO/SIF/64/R2-1999), interim WHO specifications for WT (WHO/IS/00.1-1999) and to support proposed new FAO specifications for EG, EW, WG and a new WHO specification for EW.

Deltamethrin is a single stereoisomer pyrethroid and is not under patent.

Deltamethrin was first evaluated by the FAO/WHO JMPR in 1980. Subsequent JMPR reviews were undertaken in 1984-1988, 1990 and 1992. Residues resulting from veterinary uses of deltamethrin were evaluated by the FAO/WHO Expert Committee on Food Additives (JECFA) in 1999. It was evaluated/reviewed by the European Commission (List 1) under Directive 91/414/EEC and is included in Annex 1, meaning that formulations may be registered in EU countries.

The draft specifications and the supporting data were provided by Bayer Crop Science in 2003, for evaluation in 2004.

Uses

Deltamethrin is a synthetic insecticide belonging to the pyrethroid family. It is widely registered and used in agriculture and in public and animal health as a broad spectrum insecticide against noxious insects belonging to groups such as Orthoptera, Thysanoptera, Heteroptera, Homoptera, Diptera, Coleoptera, Lepidoptera and Hymenoptera, and against ticks and mites. Main uses in agriculture are in top fruits, grapes, a wide range of vegetables, oilseed rape, cotton, cereals and maize. It is also recommended for use against locusts, indoor insects and pests of stored grain and timber. Deltamethrin is also widely used as an acaricide/insecticide for the control of ticks, mites and insect pests of livestock. Deltamethrin is non-systemic with contact and stomach action.

Identity

ISO common names

Deltamethrin (BSI, draft E-ISO), deltaméthrine ((f) draft F-ISO)

Synonyms

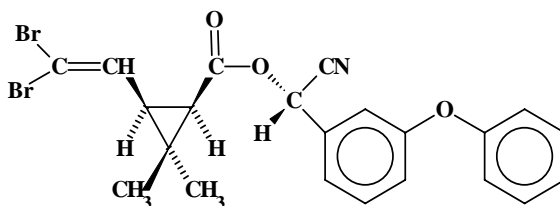
Decamethrin (rejected common name)

Chemical names

IUPAC (S)- α -cyano-3-phenoxybenzyl (1R,3R)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxylate

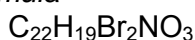
CA [1*R*-[1*α*(*S**),3*α*]-cyano(3-phenoxyphenyl)methyl 3-(2,2-dibromoethenyl)-2,2-dimethylcyclopropanecarboxylate

Structural formula



deltamethrin is one of the eight possible stereoisomers having the structure shown.

Empirical formula



Relative molecular mass

505.2 g/mol

CAS Registry number

52918-63-5

CIPAC number

333

Identity tests

HPLC retention time; TLC; IR, NMR and mass spectra.

Physical and chemical properties

Table 1. Physicochemical properties of pure deltamethrin

Characteristic	Value	Purity, %	Method	Reference
Vapour pressure	1.25 x 10 ⁻⁸ Pa at 25°C 4.13 x 10 ⁻⁸ Pa at 35°C 1.98 x 10 ⁻⁷ Pa at 45°C	99.7	US EPA 63-9, equivalent to 92/69/EC A6 or OECD 104, gas sat. method	A47916
Melting point	100-102°C	99.7	OECD 102, US EPA 63-9	A70753
Boiling point	decomposes below boiling point	99.7	OECD 103 & 92/69/EC A2	A38362
Decomposition temperature	270°C	99.7	OECD 103 & 92/69/EC A2	A38362
Solubility in water	2 x 10 ⁻⁷ g/l, 25°C; not pH-dependent but determined at pH 7.49-7.85. <5 x 10 ⁻⁶ g/l, 20°C by column elution method, pH 6.2	99.6	US EPA 63-8 (A45109) 92/69/EC A6 & OECD 105	C009221
Solubility in organic solvents at 20°C	acetone: 300-600 g/l dimethylsulfoxide: 200-300 g/l acetonitrile: 60-75 g/l at 1,2 dichloroethane: >600 g/l ethyl acetate: 200-300 g/l xylene: 150-200 g/l methanol: 8.15 g/l <i>n</i> -heptane 2.47 g/l	98.6	Similar to CIPAC MT181	C009220

Table 1. Physicochemical properties of pure deltamethrin

Characteristic	Value	Purity, %	Method	Reference
Octanol:water partition coefficient	$P_{OW} = 40200$ at 25°C $\log P (Kow) = 4.6$ P_{OW} is not pH dependent	99.3	USEPA 63-11	A47915
Hydrolysis	Negligible hydrolysis at 25°C at pH 5 Negligible hydrolysis at 25°C at pH 7 Half-life = 2.5 days at 25°C at pH 9	98.0	US EPA 161-1	A45079
Photolysis	Deltamethrin is directly photo-transformed, $DT_{50} = 48$ days, and indirectly photo-transformed, $DT_{50} = 4$ days. A 6000 W xenon arc light system was used, reportedly of intensity corresponding to one half of the intensity of the sun (wavelength 310-740 nm, $25 \pm 1^\circ C$). <i>Trans</i> -deltamethrin was found as a photolysis product but $\leq 2.0\%$.	99.3	US EPA 161-2	A41919
Dissociation characteristics	Does not dissociate.	-	-	-

Table 2. Chemical composition and properties of deltamethrin technical material (TC)

Manufacturing process, maximum limits for impurities ≥ 1 g/kg, 5 batch analysis data.	Confidential information supplied and held on file by FAO and WHO. Mass balances were 98.6–99.5%. No unidentified impurities were reported.
Declared minimum deltamethrin content:	985 g/kg
Relevant impurities ≥ 1 g/kg and maximum limits for them:	None
Relevant impurities < 1 g/kg and maximum limits for them:	None
Stabilizers or other additives and maximum limits for them:	None
Melting or boiling temperature range	100-102°C

Hazard summary

Notes.

- (i) The proposers provided written confirmation that the toxicological and ecotoxicological data included in the summary below were derived from deltamethrin having impurity profiles similar to those referred to in the table above.
- (ii) The conclusions expressed in the summary below are those of the proposers, unless otherwise specified.

Table 3. Toxicology profile of the deltamethrin technical material, based on acute toxicity, irritation and sensitization

Species	Test	Duration and conditions	Result
Rat, Sprague-Dawley	Oral	Deltamethrin (purity 98%) in corn oil administered by gavage as a single dose to 5 groups of 5 male and five female non-fasted rats. Observation period 14 d.	LD ₅₀ = 87 mg/kg bw/day
Rat (Wistar ICO:WI (IOPS AF/Han))	Dermal	Deltamethrin (purity 98.6%) in corn oil, topically administered to intact shaved skin of 5 males and 5 females. Administered dose 2000 mg/kg bw. Observation period 14 d.	LD ₅₀ >2000 mg/kg bw
Rat, Sprague-Dawley	Inhalation	Groups of 7 males and 7 females exposed whole body for a single 6-h period to dust particulate aerosol atmospheres of deltamethrin (purity not specified) at concentrations of 0 (air only) 0.049, 0.430, 0.540 and 0.720 mg/l air.	LC ₅₀ = 0.6 mg/l
Rabbit, New Zealand White	Skin irritation	24-h exposure (abraded and intact skin). Skin evaluated at 48 h post-treatment. Deltamethrin purity 98%.	Non irritating
Rabbit, New Zealand White	Eye irritation	Eyes evaluated at 1, 24, 72 h & 7 d in accordance with Draize method. Deltamethrin purity 99.2%.	Non irritating
Guinea pig, albino	Skin sensitization	M & K, Buehler. Deltamethrin purity 99.2%.	Non sensitizing

Table 4. Toxicology profile of deltamethrin technical material based on repeated administration (sub-acute to chronic)

Species	Test	Duration and conditions	Result
Mouse, male/female, CD& CrI	90 day dietary sub-chronic study	OECD Guideline 408. Dosage: 0, 6, 62, 600 and 1300 mg/kg bw/day for males and 0, 8, 77, 740 and 1400 mg/kg bw/day for females. Deltamethrin (purity 99.7%) administered in diet.	NOAEL = 62 mg/kg bw/day based on clonic contractions and clinical signs of poor condition at 600 mg/kg bw/day.
Rat, Male/female, CD	14 day inhalation toxicity study	Study performed prior to GLP regulations. Dosage: 3, 10 and 56 mg/m ³ . Deltamethrin administered by inhalation, whole body exposure for 6 h/day, 5 days/week for 2 weeks and 4 days on the 3rd week (total 14 exposures).	NOAEC = 9.6 mg/m ³ corresponding to 2.6 mg/kg bw per exposure based on neurological signs.
Rat, Male/female, SD	21 day dermal toxicity study	OECD Guideline 410. Dosage: 0, 100, 300, 1000 mg/kg bw/day. Deltamethrin (purity, 99.6%) mixed with PEG 400 administered dermally for 6 h on 21 consecutive days.	NOAEL = 1000 mg/kg bw/day for systemic toxicity.

Table 4. Toxicology profile of deltamethrin technical material based on repeated administration (sub-acute to chronic)

Species	Test	Duration and conditions	Result
Rat, male/ female, Crl/CD	90 day dietary sub-chronic study	Study performed prior to GLP regulations. Dosage: 0, 0.1, 1.0, 2.5 and 10 mg/kg/day. Deltamethrin administered by oral gavage as solution in PEG 200	NOEL = 1.0 mg/kg/day LOEL = 2.5 mg/kg/day, depression of body weight.
Dog, male/female, beagle	90 day dietary sub-chronic study	OECD Guideline 409. Dosage: 0, 2, 10 and 50 mg/kg bw/day. Deltamethrin (purity 98.9%) administered orally in gelatine capsules.	NOAEL = 10 mg/kg bw/day based on neurological disturbances and reduced food intake and body-weight gain at 50 mg/kg bw/day.
Dog, male/ female, beagle	1-year dietary study	OECD Guideline 452. EPA/FIFRA Guideline 83-1. Dosages: control 4 M + 4 F; 1 mg/kg/day 4 M + 4 F; 10 mg/kg/day 4 M + 4 F; 50 mg/kg/day 4 M + 4 F. Deltamethrin (purity 98.9%) administered orally in gelatine capsules.	NOAEL = 1 mg/kg/day.
Rat, male/ female , Charles River CD strain	Two year oral toxicity and carcinogenicity study in rats	Study performed prior to GLP regulations. Dosages :0, 2, 20 and 50 ppm. Deltamethrin (purity 98%) concentration adjusted for each dosage level to give 2.5 g corn oil/kg basal diet for each group. Control rats received 2.5 g corn oil/kg basal diet.	NOEL = 25 ppm, i.e. 1 mg/kg/day. 50 ppm (LEL) produced consistent decrease in body weight gain in the males. No oncogenic effects were noted at up to and including 50 ppm (HDT).
Mouse, Male/ female, Charles River CD - 1.	Two-year oral toxicity and carcinogenicity study in mice	Study performed prior to GLP regulations. Dosages:0, 1, 5, 25 and 100 ppm. Deltamethrin (purity 98%) concentration adjusted for each dosage to give 5 g corn oil/kg basal diet for each dosage. Control mice received 5 g corn oil/kg of basal diet.	No adverse effects observed at highest dose tested. Therefore NOEL = 100 ppm, i.e. 12 mg/kg/day in male and 15 mg/kg/day in female. No oncogenic effects observed up to and including 100 ppm (HDT)
Rat, male/ female , Charles River, Crl:CD® BR VAF/Plus®	Two- generation reproduction	EPA - FIFRA Guidelines 83-4; OECD Guidelines section 4, No.416. 30 M + 30 F per treatment group (5 groups receiving, 0, 5, 20, 80 and 320 ppm deltamethrin in daily diet, deltamethrin purity 99.7%.	Parental toxicity NOEL = 80 ppm = 4.2 to 12.4 mg/kg bw/day. Offspring toxicity NOEL = 80 ppm = 10.6 to 26.4 mg/kg bw/day. Reproduction toxicity NOEL = 320 ppm = 18.3 to 43.8 mg/kg bw/day.

Table 5. Mutagenicity profile of deltamethrin technical material based on *in vitro* and *in vivo* tests

Species	Test	Conditions	Result
<i>Salmonella typhimurum</i> , strains TA98, TA100, TA1535, TA1537 and <i>E. Coli</i> WP2uvrA	Bacterial reverse mutation assay (Ames assay), <i>in vitro</i>	± S9 metabolic activation. Concentrations up to 5000 µg/plate. Deltamethrin purity 96%.	Negative
CHO/HGPRT	Mammalian cell gene mutation	Tested at 5 to 100 µg/ml without activation and 100 to 1500 µg/ml with activation.	Negative
Mouse (CD-1), males and females	Micronucleus formation assay, <i>in vivo</i>	Deltamethrin (purity 99.9%) dissolved in PEG 200 administered orally as a single dose to 5 to 8-week old mice at 500, 1000 and 2000 mg/kg bw. Each group was 5 animals/sex.	Negative
Chinese hamster ovary cells	Chromosome aberration assay	EPA/FIFRA 84-2 (b). Doses: 19, 38, 75, 150 µg/ml. TEM dose 0.5 µg/ml, CP dose 50 µg/ml. Metabolic activation S9. Deltamethrin purity 99.2%.	Negative
Rat primary hepatocyte	Unscheduled DNA synthesis	EPA/FIFRA 84-4. Doses: 4200, 1300, 420, 130, 42, 13, 4.2 µg/ml. Doses of DMBA 10 and 5 µg/ml. Deltamethrin purity 92%.	Negative
Male mouse strains	Dominant lethal assay	EPA 84-2, A.J. Bateman (1973) method.	Negative

Table 6. Ecotoxicology profile of deltamethrin technical material

Species	Test	Duration and conditions	Result
<i>Daphnia magna</i> (water flea)	Acute toxicity	48 h flow-through, observations at 24 and 48 h.	24 h EC ₅₀ >1.3 mg/l 48 h EC ₅₀ = 0.56 mg/l
<i>Oncorhynchus mykiss</i> (rainbow trout)	Acute toxicity		96 h LC ₅₀ = 0.26 µg/l
<i>Lepomis macrochirus</i> (bluegill sunfish)	Acute toxicity	FIFRA 72-1 96 h. Nominal concentrations: 0, 0.20, 0.41, 0.81, 1.6, 3.2 µg/l	96 h LC ₅₀ = 1.40 µg/l NOEC = 0.41 µg/l
<i>Selenastrum capricornutum</i> (green algae)	Effect on growth	OECD 201-1984. 96 h duration, 9.1 mg/l.	At the dose tested, no reduction in average growth occurred (deltamethrin partially degraded during test). E _b C ₅₀ uncertain but deltamethrin is not toxic to algae, because the initial and final test concentrations exceeded the water solubility of deltamethrin.
<i>Eisenia fetida</i> (earthworm)	Acute toxicity	OECD 207 14 days, artificial soil. Nominal concentrations: 167, 279, 465, 776 and 1290 ppm (dry soil).	LC ₅₀ >1290 mg/kg
<i>Apis mellifera</i> (honey bee)	Acute contact toxicity	Topical application, observed at 24 and 48 h, at 24°C.	LD ₅₀ (48 h) 1.5 ng/bee
<i>Apis mellifera</i> (honey bee)	Oral toxicity (normal feeding)	Dosed and observed for 24 and 48 h, at 24°C	LD ₅₀ 79 ng/bee

Table 6. Ecotoxicology profile of deltamethrin technical material

Species	Test	Duration and conditions	Result
<i>Anas platyrhynchos</i> (mallard duck)	Acute oral toxicity	FIFRA 71-1. Test levels: 215, 464, 1000, 2150, 4640 mg/kg.	LD ₅₀ >4640 mg/kg bw
<i>Colinus virginianus</i> (bobwhite quail)	Acute oral toxicity	FIFRA 71-1. Test levels 292, 486, 810, 1350, 2250 mg/kg.	LD ₅₀ >2250 mg/kg bw
<i>Anas platyrhynchos</i> (mallard duck)	Dietary toxicity	FIFRA 71-2. Test levels: 562, 1000, 1780, 3160, 5620 ppm	LC ₅₀ = 8039 ppm
<i>Colinus virginianus</i> (bobwhite quail)	Dietary toxicity	FIFRA 71-2. Test levels: 562, 1000, 1780, 3160, 5620 ppm	LC ₅₀ >5620 ppm

Deltamethrin was evaluated by the FAO/WHO JMPR in 1977, 1980, 1992, 1994, 1995 and 2000. On the basis of residues data from a wide range of crops, the JMPR concluded that intake of residues of deltamethrin is unlikely to present a public health concern (JMPR 1995). In 2000, the JMPR set an ADI of 0.01mg/kg bw and an acute RfD of 0.05 mg/kg bw.

The U.S. EPA considered that registered uses of deltamethrin will not cause unreasonable risk to humans.

The WHO hazard classification of deltamethrin is Class II, moderately hazardous.

Formulations

The main formulations type are DP, WP, EC, UL, SC and WT, with additional formulations now being produced (EG, EW, WG). These formulations are registered and sold in 90 countries throughout the world. Deltamethrin may be co-formulated with other pesticides, such as abamectin, azaconazole, buprofezin, chlorpyrifos, dichlorvos, difethialone, diflubenzuron, dimethoate, endosulfan, esbiothrin, fenitrothion, imidacloprid, pirimicarb, S-bioallethrin, tetramethrin, thiacloprid and triazophos.

Methods of analysis and testing

A full CIPAC method for determination of deltamethrin in TC, WP, EC, UL and DP was adopted in 1985 (CIPAC D). The method was based on normal-phase HPLC (silica column eluted with *iso*-octane/dioxan), with external standardization and UV detection at 254 nm. Identification of deltamethrin was by HPLC retention time (two techniques), with additional tests involving TLC, IR, NMR and MS.

A new analytical method for determination of deltamethrin in TC, DP, SC, EC and WG was adopted by CIPAC, with provisional status, in 2004. This method also involved HPLC but used a cyano-column and detection at 230 nm. The new method was also collaboratively studied for analysis of WT but the results were not within the normally acceptable range and CIPAC agreed that the method for WT should be given only tentative status. In 2005, the manufacturer is conducting a validation study for extension (according to CIPAC requirements) of the new method to the

analysis of WP, EG, EW and UL, and this will include a re-validation of the method for WT*. The original identity tests (CIPAC D) remain valid.

Test methods for determination of physico-chemical properties of the pure and technical active ingredient were EC, USEPA, OECD, CIPAC while those for the formulations were CIPAC, as indicated in the specifications. Methods for the determination of tablet hardness and disintegration time of WT are being validated under the auspices of CIPAC, for completion in 2005.

Physical properties

The physical properties, and the methods for testing and the limits proposed for them, of the DP, WP, SC, EC, UL, EG, EW, WT and WG formulations, comply with the requirements of the Manual (FAO/WHO 2002).

Containers and packaging

No special requirements for containers and packaging have been identified.

Expression of active ingredient

The active ingredient is expressed as deltamethrin, in g/kg in solid formulations, and in g/kg or g/l at $20 \pm 2\text{C}$ in liquid formulations.

Appraisal

The Meeting considered data on deltamethrin for the review of (i) existing FAO full specifications for TC, WP, EC, DP, UL; (ii) of existing WHO full specifications for TC, WP, EC, DP, UL, SC; (iii) of an existing WHO interim specification for WT; and for the development of proposed new FAO specifications for EG, EW, WG and a new WHO specification for EW. The data submitted by Bayer CropScience were in accordance with the requirements of the FAO/WHO Manual and supported the proposed specifications, which were largely in accordance with the requirements of the FAO/WHO manual.

Deltamethrin is a pyrethroid insecticide which is both very widely used and has a very wide range of applications in agriculture, animal health and public health. It is out of patent.

Deltamethrin consists of a single stereoisomer ((S)- α -cyano-3-phenoxybenzyl (1R,3R)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxylate), being one of the eight possible stereoisomers of the same structure.

Deltamethrin does not dissociate in water and it has extremely low water solubility and low volatility. The octanol/water partition coefficient is high, indicating fat solubility and partition into soil organic matter. Bioaccumulation does not occur because it is fairly rapidly metabolized and in soil it is neither mobile nor of long persistence.

* Extensions of the analytical method to EG, EW and WP were adopted by CIPAC in 2005 (CIPAC 2005a) but the status of the method for WT remained only tentative. The manufacturer explained that, for analytical purposes, the UL is analogous to the EC and CIPAC agreed that the method validation accepted in 2004 for analysis of EC could also encompass the UL (CIPAC 2005b).

Deltamethrin is stable to hydrolysis at pH 4-7 but is rather slowly hydrolyzed at pH 9. Direct and indirect photolysis can occur in solution and products, other, less insecticidally active, stereoisomers can be produced, along with other degradation products.

The WHO hazard classification is Class II, moderately hazardous and the FAO/WHO JMPR has allocated an ADI of 0.01 mg/kg bw and the acute RfD of 0.05 mg/kg bw. The JMPR concluded that intake of residues of deltamethrin is unlikely to present a public health concern and USEPA concluded that registered uses of deltamethrin will not cause unreasonable risk to humans. Deltamethrin is not a skin/eye irritant, nor a skin sensitizer, and there is no evidence of genotoxic, carcinogenic, mutagenic, teratogenic or reproductive effects. Like many other pyrethroids used as insecticides, deltamethrin is very highly toxic to fish, daphnids and non-target insects, such as honeybees.

The Meeting was provided with commercially confidential information on the manufacturing process and batch analysis data on all detectable impurities. None of the impurities was present at or above 1 g/kg. Mass balances were high (98.6–99.5%). The data were confirmed as identical to those evaluated for the registration of deltamethrin in Belgium.

The Meeting considered the relevance of impurities.

The existing FAO and WHO specifications included clauses to limit the concentrations of various impurities. The existing specifications for TC limited the concentration of “deltamethrin *R*-isomer” ((*R*)- α -cyano-3-phenoxybenzyl (1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxylate) to 10 g/kg; “deltamethrin acid chloride” ((1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxy chloride) to 2 g/kg; and the sum of “deltamethrin acid” ((1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxylic acid) and “deltamethrin acid anhydride” (the anhydride of (1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropane carboxylic acid) to 10 g/kg. The existing specifications for the formulations included clauses to limit “deltamethrin *R*-isomer” and water.

The manufacturer and the Meeting agreed that “deltamethrin acid” is not a relevant impurity and that it was not necessary to specify the maximum water content of formulations, partly because deltamethrin has extremely low affinity for water and partly because the water content is adequately controlled indirectly through clauses for physical properties. There was also agreement that, because the concentrations of “deltamethrin acid chloride” and “deltamethrin acid anhydride” do not approach 1 g/kg, that these should not be specified as relevant impurities.

The manufacturer proposed that “deltamethrin *R*-isomer” should be identified as a relevant manufacturing impurity. The manufacturer had validated an analytical method for the determination of this isomer. It has lower insecticidal activity than deltamethrin, it is also less hazardous to humans and the environment than deltamethrin and it has no other properties which would make relevant according to the criteria given in the FAO/WHO manual. The Meeting therefore agreed that it should not be identified as a relevant impurity. Its concentration will be controlled indirectly by the clause limiting the minimum content of the active ingredient, in the same way as any other non-relevant impurities, including those produced during storage.

A new analytical method for the determination of deltamethrin TC, SC, EC, DP and WG was adopted by CIPAC in 2004, with provisional status, which is sufficient to support the proposed specifications. However, the method did not give acceptable results for the analysis of WT and CIPAC allocated it a tentative status, which is not sufficient to support of the proposed specification for WT.

The manufacturer stated that validation of extensions of the new CIPAC method is in progress for the analysis of EG, EW, UL, WP and WT. These method extensions have to be adopted by CIPAC, with provisional status, to provide appropriate support for the specifications proposed for these formulations*.

Apart from the issue of relevant impurities, the proposed specifications were in accordance with the requirements of the FAO/WHO manual.

The existing specifications for deltamethrin formulations included various limits for pH range and acidity/alkalinity but the manufacturer rationalised these in the proposed specifications, specifying the same pH range (4-7.5) in all cases except WG. In the case of WG, the formulants produce a pH of 3.5-3.6 and the Meeting and manufacturer agreed that it is more appropriate to limit the measured concentration of acid than the pH range in this case.

In accordance with the guideline given in the manual, the existing WHO interim and proposed specifications for WT incorporated a clause for disintegration time. The manufacturer stated that the test method would be validated under the auspices of CIPAC in 2005 but a specification limit was not proposed. An acceptable clause and limit for degree of attrition (CIPAC MT 193 "Friability") was provided under the heading of "tablet integrity" but the manufacturer disputed the need to specify "no broken tablets". Both were specified in the existing interim WHO specification. The manufacturer proposed inclusion of a clause for tablet hardness and stated that the test method would be validated under the auspices of CIPAC in 2005 but specification limits were not proposed. Tablet hardness was not specified in the existing WHO interim specification. The Meeting agreed that the WT specification should then be reconsidered when the requirements and limits have been clarified.

Recommendations

The Meeting recommended the following.

- (i) Existing FAO specifications for deltamethrin TC, WP, EC, DP and UL should be withdrawn.
- (ii) Existing WHO full specifications for deltamethrin TC, WP, EC, DP, UL and SC, and the existing WHO interim specification for deltamethrin WT, should be withdrawn.
- (iii) The proposed specifications for deltamethrin TC, DP, SC, EC and WG, amended as described in the appraisal above, should be adopted by FAO and WHO.

* Extensions of the analytical method to EG, EW and WP were adopted by CIPAC in 2005 (CIPAC 2005a) but the status of the method for WT remained only tentative. The manufacturer explained that, for analytical purposes, the UL is analogous to the EC and CIPAC agreed that the method validation accepted in 2004 for analysis of EC could also encompass the UL (CIPAC 2005b).

(iv) The proposed specifications for deltamethrin EG and EW, amended as described in the appraisal above, should be adopted by FAO, subject to CIPAC adoption* of the analytical method extensions to these formulations.

(v) The proposed specifications for deltamethrin EW, amended as described in the appraisal above, should be adopted by WHO, subject to CIPAC adoption* of the analytical method extensions to these formulations and WHOPES testing/evaluation of the product for public health use.

(vi) The proposed specification for deltamethrin UL and WP, amended as described in the appraisal above, should be adopted by FAO and WHO, subject to CIPAC adoption* of the analytical method extensions to these formulations.

(vii) The proposed specification for deltamethrin WT should be reconsidered the JMPS when the physical test requirements, methods and proposed limits have been clarified and the test methods suitably validated; and that adoption of the specification should be subject to satisfactory validation of a method for analysis of this formulation type, by CIPAC.

(viii) Although specifications for DP, SC, EC, WG, EW, UL and WP used in agriculture and public health are identical, users must adhere to label recommendations and not use the products interchangeably.

References

Bayer Crop Science document No. or other reference	Year and title or publication details
A41919	1987. Determination of photodegradation of ¹⁴ C deltamethrin in aqueous solution.
CIPAC 2005a	333, Deltamethrin (two entries), in: Summary of the decisions taken at the 49 th CIPAC Meeting in Utrecht, the Netherlands, on Wednesday 8 th June, and Thursday 9 th June (a.m.) 2005. Document No. CIPAC/4492/P.
CIPAC 2005b	Item 4.5, Deltamethrin (4455), in: Minutes of the 49 th Annual meeting held at the Jaarbeurs Convention Centre in Utrecht, the Netherlands on Wednesday 8 th June, and Thursday 9 th June (a.m.) 2005. Document No. CIPAC/4491/P
CIPAC D	A. Martijn and W. Dobrat, eds., 1988. CIPAC Handbook volume D, pp. 57-66. Collaborative International Pesticides Analytical Council Ltd., Harpenden, UK.
FAO/WHO 2002	Manual on the development and use of FAO and WHO specifications for pesticides, 1 st edition. FAO plant production and protection paper, 173. FAO, Rome, 2002.
C009221	2000. Deltamethrin A.I. - solubility in water at 20°C.
C009220	2000. Deltamethrin A.I. - solubility in organic solvent at 20°C.
A38362	1988. Deltamethrin technical-decomposition point.
A45079	1990. Determination of aqueous hydrolysis rate and half life of deltamethrin.
A70753	1990. Deltamethrin A.I. – determination of melting point.
WHO 2002	The WHO recommended classification of pesticides by hazard and guidelines to classification 2000-2002. WHO, Geneva, 2002.
A47916	1991. Deltamethrin A.I. – determination of vapor pressure.

* Extensions of the analytical method to EG, EW and WP were adopted by CIPAC in 2005 (CIPAC 2005a) but the status of the method for WT remained only tentative. The manufacturer explained that, for analytical purposes, the UL is analogous to the EC and CIPAC agreed that the method validation accepted in 2004 for analysis of EC could also encompass the UL (CIPAC 2005b).

Bayer Crop Science document No. or other reference	Year and title or publication details
A47915	1991. Deltamethrin A.I. – determination of octanol water partition, coefficient.