AGP: CP/228

FAO SPECIFICATIONS FOR PLANT PROTECTION PRODUCTS

ENDOSULFAN

(I,4,5,6,7,7-hexachloro-8,9,10-trinorborn-5-en-2,3-ylene bismethylene) sulfite

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS Rome, 1989 Group on Pesticide Specifications

FAO Panel of Experts on Pesticide Specifications, Registration Requirements and Application Standards

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DISCLAIMER¹

FAO 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.

FAO disclaims any and all liability for any injury, death, loss, damage or other prejudice of any kind that may 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, FAO 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.

FAO 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, FAO does not in any way warrant or represent that any pesticide claimed to comply with a FAO specification actually does so.

¹ This disclaimer applies to all specifications published by FAO.

INTRODUCTION TO FAO SPECIFICATIONS DEVELOPED UNDER THE OLD PROCEDURE

Between 1975 and 2000, FAO published booklets of specifications for technical materials and related formulations of plant protection products. Revisions of, and additions to, already published specifications will be issued when necessary. However, all changes and revisions of FAO specifications are now subject to the new procedure described in the *Manual on the development and use of FAO and WHO Specifications for Plant Protection Products*, FAO Plant Production and Protection Paper No. 173, Rome 2002 (*Revised First Edition available only on the FAO home page of the Internet at: http://www.fao.org/ag/agp/agpp/pesticid/*)

FAO specifications developed under the old procedure are based on the requirements defined in the Fourth Edition of the *Manual on the development and use of FAO specifications for plant protection products*, Plant Production and Protection Paper No. 128, Rome 1995.

This manual contained detailed definitions and other essential background information on basic procedures and technical principles adopted by the group on Pesticide Specifications of the FAO Panel of Experts on Pesticide Specifications, Registration Requirements, Application Standards and Prior Informed Consent, such as:

1. Categories of Specifications (Section 3.1 of the Manual)

<u>FAO Tentative Specifications (Code 'S/T', formerly 'TS')</u> are those which have been recommended by FAO as preliminary specifications and which are based on minimum requirements. The methods of analysis cited are normally supplied by the manufacturer or may already have been published or be the subject of collaborative work.

<u>FAO Provisional Specifications [Code 'S/P', formerly ('S')]</u> are those for which more evidence of the necessary parameters is available and where some collaborative study of the methods of analysis has been carried out.

FAO (full) Specifications (Code 'S/F', formerly 'S').

Specifications that have all necessary requirements together with CIPAC (full) methods, or other collaboratively studied (proven) methods.^{2, 3}

Wherever possible, standards for apparatus and common names for pesticides are those approved by the International Organization for Standardization (ISO).

- 2. Expression of active ingredient content (Section 4.2.5 of the Manual)
 - for solids, liquid technical materials, volatile liquids (of maximum boiling point 50°C) and viscous liquids (with minimum kinematic viscosity of 1 x 10^3 m²/s at 20°C) the FAO Specification shall be based on expression of the content as g/kg;

- for all other liquids the active ingredient content of the product shall be declared in terms of g/kg *or* g/l at 20°C. If the customer requires both g/kg *and* g/l at 20°C, then in case of dispute the analytical results shall be calculated as g/kg.
- 3. Tolerance on content (Section 4.2.7 of the Manual)

A declared content of active ingredient must be included in all specifications, and one of the problems immediately arising is the level of tolerance acceptable about the nominal figure. The tolerance is influenced by (a) the reproducibility of the method of analysis, (b) the sampling error and (c) the manufacturing variance.

Allowable variations in analytical results (i.e. tolerances in content of active ingredient) with respect to specific pesticide consignments are intended to cover reasonable variations in the contents of active ingredients. For examples of such tolerances, see the table in Section 4.2.7 of the Manual.

4. Containers/packaging

FAO guidelines are in preparation.

Containers shall comply with pertinent national and international transport and safety regulations.

Technical materials, dustable powders and granules

Containers shall be suitable, clean, dry and as specified, and shall not adversely affect, or be affected by, the contents, but shall adequately protect them against external conditions.

Wettable powders

The product shall be packed in suitable, clean, dry containers as specified in the order. The container shall provide all necessary protection against compaction, atmospheric moisture, loss by vaporization and/or contamination to ensure that the product suffers no deterioration under normal transit and storage conditions.

The product shall be protected by an adequate moisture barrier. This may be a suitable bag of polyethylene or alternative means of giving equal or better protection.

Solutions and emulsifiable concentrates

Containers shall be lined, where necessary, with a suitable material, or the interior surfaces shall be treated to prevent corrosion and/or deterioration of the contents.

Additional information should be given in all specifications where particular pesticides present problems in packaging.

5. Biological information

Phytotoxicity

No test can be specified to cover the possible phytotoxicity of a formulation to all crops. When a crop is not mentioned in the instructions for use, purchasers should check with the supplier that the material is suitable, always provided that such a use is not restricted or legally forbidden.

Wetting of crops

The dilute spray should satisfactorily wet the leaves of the specified crops when used in accordance with the instructions. Test method MT 53.2, CIPAC F, p.162, may be useful.

¹ Should national pesticide specifications developed from these approved FAO specifications deviate from them, the National Authority responsible for making such changes is requested to inform the FAO Plant Protection Service of the nature of, and the reasons for, the modifications.

² Methods of analysis and miscellaneous techniques referred to in these specifications have been developed and adopted by CIPAC (Collaborative International Pesticides Analytical Council Ltd.). See CIPAC Handbooks 1 (1970), 1A (1980), 1B (1983), 1C (1985), D (1988), E (1993), F (1995), G (1995), CIPAC Proceedings 1980 and 1981, obtainable from Black Bear Press Limited, King's Hedges Road, Cambridge CB4 2PQ, England. The page numbers of specific methods are given in parentheses in the specifications. Copies of methods not yet published can be obtained from the FAO Plant Protection Service.

³ Information on standard waters for laboratory evaluation of pesticidal formulations will be found in CIPAC Monograph 1, Standard Waters and an FAO Survey on Naturally Occurring Waters (1972), Black Bear Press Limited, King's Hedges Road, Cambridge CB4 2PQ, England.

SUBMISSION OF DRAFT SPECIFICATIONS TO FAO

Any organization, commercial firm or interested individual is encouraged to submit relevant specifications, or proposals for revision of existing specifications, for pesticide products for consideration and possible adoption by FAO. Correspondence should be addressed to the Pesticide Management Group, Plant Production and Protection Division, FAO, Viale delle Terme di Caracalla, 00153 Rome, Italy.

General guidelines on preparing draft specifications are given in the *Manual on the development and use of FAO and WHO Specifications for Plant Protection Products*, FAO Plant Production and Protection Paper No. 173, Rome 2002 (Revised First Edition available only on the FAO home page of the Internet at: <u>http://www.fao.org/ag/agp/agpp/pesticid/</u>).

Specifications which are considered suitable for further processing are assigned priorities and circulated to appropriate organizations and specialists to comment. Comments, together with other relevant information, are then reviewed in detail by the Group on Specifications of the FAO Panel of Experts on Pesticide Specifications, Registration Requirements, Application Standards and Prior Informed Consent. The drafts are converted into <u>FAO Provisional Specifications</u>, or full <u>FAO Specifications</u>.

INFORMATION

COMMON NAME: Endosulfan (ISO)

EMPIRICAL FORMULA: C9H6Cl6O3S

RMM: 406.9

CAS REGISTRY NUMBER: 115-29-7

CIPAC CODE NUMBER: 89

CHEMICAL NAME:

- (I,4,5,6,7,7-hexachloro-8,9,10-trinorborn-5-en-2,3-ylene bis methylene) sulfite (IUPAC)
- 6,7,8,9,10,10-hexachloro-I,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin 3-oxide (CA)

ENDOSULFAN TECHNICAL

FAO Specification 89/TC/S (1989)

.1 DESCRIPTION

The material shall consist of endosulfan together with related manufacturing impurities and shall be cream or brown coloured granules, flakes or powders, free from visible extraneous matter and added modifying agents.

.2 ACTIVE INGREDIENT

.2.1 Identify tests (CIPAC IC, 89/TC/M2/2, p.2110)

Where the identity of the active ingredient is in doubt, then it shall comply with at least one additional test.

.2.2 Endosulfan (-/M2/3, p.2110 (Referee method) or CIPAC I, 89/1/(M)1/1. 2, p.361)

The endosulfan content shall be declared (not less than 940 g/kg) and when determined, the content obtained shall not differ from that declared by more than +/-20 g.

.2.3 Isomer content (-/M2/3, p. 2110 (Referee method) or CIPAC I, 89/1/(M)1/1.2, p. 361)

alpha-isomer : 64 to 67% beta-isomer : 29 to 32%

of the total endosulfan content.

.2.4 Freezing Point (MT 1, CIPAC I, p. 822)

Minimum : 80°C

.3 IMPURITIES

.3.1 Endosulfan-ether (note 1)

Maximum : 10 g/kg

.3.2 Endosulfan-alcohol (note 1)

Maximum : 20 g/kg

.3.3 Endosulfan sulfate (note 1)

Maximum : 2 g/kg

.3.4 Loss on drying (MT 17.1, CIPAC I, p. 871)

Maximum : 10 g/kg

.3.5 Material insoluble in acetone (MT 27, CIPAC I, p. 894)

Maximum : 10 g/kg

.4 PHYSICAL PROPERTIES

.4.1 Acidity (MT 31.2.1., CIPAC I, p. 905)

Maximum : 1 g/kg, calculated as H2SO4

Note 1 No standardized method, but the Referee method (CIPAC 1C, 89/TC/M2/3, p. 2110) may be used.

ENDOSULFAN DUSTABLE POWDERS

FAO Specification 89/DP/S (1989)

.1 **DESCRIPTION**

The material shall consist of a homogeneous mixture of technical endosulfan (complying with the requirements of FAO specification 89/TC/S (1989)), together with carriers and any 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 (CIPAC IC, 89/DP1M2/2, p. 2113)

Where the identity of the active ingredient is in doubt, then the isolated active ingredient shall comply with at least one additional test.

.2.2 Endosulfan (CIPAC IC, 89/Dp/M2/3, p. 2113 (Referee method) or CIPAC I, 89/2/(M)/1.2, p. 367)

The endosulfan content shall be declared (g/kg) and when determined, the content obtained shall not differ from that declared by more than +/-10%.

.3 IMPURITIES

.3.1 Water (MT 30.1, CIPAC I, p. 897)

Maximum : 20 g/kg

.4 PHYSICAL PROPERTIES

.4.1 Acidity or alkalinity (MT 31.1.2 CIPAC I, p. 903 or 31.2.2. p. 905)

Maximum acidity : 1 g/kg, calculated as H2SO4

Maximum alkalinity : 2 g/kg, calculated as NaOH.

.4.2 Dry sieve test (CIPAC I, 89/2/(M)/1.4, p. 368)

4.2.1. Crop protection (Use on plants)

Maximum : 5% retained on a 75 µm test sieve

Not more than $(0.005 \times X)\%$ of the amount of sample used for the determination shall be present as endosulfan in the residue on the sieve, where X is the endosulfan content (g/kg) found under .2.2 (note 1).

.4.2.2 Crop protection (ground application), stored products and similar

uses.

Maximum: 2% retained on a 150 µm test sieve

Not more than $(0.002 \times X)\%$ of the amount of sample used for the determination shall be present as endosulfan in the residue on the sieve, where X is the endosulfan content (g/kg) found under .2.2 (note 1).

.4.3 Flowability (MT 44, CIPAC IA, p. 1567)

If required, maximum flow number: 14

In the absence of proven methodology, this clause is for information only.

.5 STORAGE STABILITY

.5.1 Stability at 54°C (MT 46.1.4. CIPAC I, p. 953)

After storage at 54 +/- 2° C for 14 days, the product shall continue to comply with .2.2, .4.1 and .4.2.

i.e. <u>(0.005 x 50) x 20</u> g 100

nor 0.02 g for .4.2.2,

i.e. <u>(0.002 x 50) x 20 g</u> 100

Note 1 If the product has a found content of 50 g/kg of endosulfan and 20 g of sample is used in the test, then the amount of endosulfan in the residue should not exceed 0.05 g for .4.2.1

ENDOSULFAN WETTABLE POWDERS

FAO Specification 89/WP/S (1989)

.1 **DESCRIPTION**

The material shall consist of a homogeneous mixture of technical endosulfan (complying with the requirements of FAO Specification 89/TC/S (1989)), together with fillers and any other necessary formulants. 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 (CIPAC IC, 89/WP/M2/2, p. 2114)

Where the identity of the active ingredient is in doubt, then the isolated active ingredient shall comply with at least one additional test.

2.2 Endosulfan (CIPAC IC, 89/WP/M.2/3, p. 2114 (Referee method) or CIPAC I, 89/3/(M)/1.2, p. 369)

The endosulfan content shall be declared (g/kg) and when determined, the content obtained shall not differ from that declared by more than the following amounts :

Declared content	Permitted tolerance
Up to 500 g/kg	+/- 5% of the declared content
Above 500 g/kg	+/- 25 g

.3 IMPURITIES

.3.1 Water (MT 17.4.1, CIPAC I, p. 874)

Maximum : 20 g/kg

.4 PHYSICAL PROPERTIES

.4.1 Acidity or alkalinity (MT 31.1.2, CIPAC I, p. 903 or 31.2.2, p. 905)

Maximum acidity : 2 g/kg calculated as H2SO4

Maximum alkalinity : 2 g/kg calculated as NaOH

.4.2 Wet sieve test (MT 59.3, CIPAC I, p. 981)

Maximum : 2% retained on a 75 µm sieve

.4.3 Suspensibility (CIPAC I, 89/3/(M)/1.5, p. 369) (notes 1 and 2)

A minimum of 50% of the endosulfan content found under .2.2 shall be in suspension after 30 min in CIPAC Standard Water C.

Alternatively, if the buyer requires other CIPAC Standard Waters to be used, then this shall be specified when ordering.

.4.4 Persistent foam (MT 47, CIPAC I, p. 954) (note 3)

Maximum : 25 ml after 1 min.

.4.5 Wetting of the product (MT 53.3.1, CIPAC I, p. 967)

It shall be completely wetted in 2 minutes without swirling.

.5 STORAGE STABILITY

.5.1 Stability at 54°C (MT 46.1.1, CIPAC I, p. 951)

After storage at 54 +/- 2° C for 14 days, the product shall continue to comply with .2.2, .4.1 and .4.2.

Note 1 The product should be tested at the highest and lowest rates of use recommended by the supplier, provided this does not exceed the conditions given in the method.

Note 2 This test will normally only be carried out after the heat stability test .5.1.

Note 3 The amount of sample to be used in the test should be specified.

ENDOSULFAN SOLUTIONS

FAO Specification 89/OL/S (1989)

.1 **DESCRIPTION**

The material shall consist of a solution of technical endosulfan (complying with the requirements of FAO specification 89/TC/S (1989)) together with any necessary formulants. It shall be free from visible suspended matter and sediment.

.2 ACTIVE INGREDIENT

.2.1 Identity tests (note 1)

Where the identity of the active ingredient is in doubt, then the isolated active ingredient shall comply with at least one additional test.

.2.2 Endosulfan (note 2)

The endosulfan content shall be declared (g/kg or g/l at 20 C, Note 3) and when determined, the content obtained shall not differ from that declared by more than the following amounts:

Declared content

Permitted tolerance

up to 50 g/kg or g/l above 50 g/kg or g/l +/- 10% of the declared content +/- 5 g

.3 IMPURITIES

.3.1 Water (MT 30.1, CIPAC I, p. 897)

Maximum : 5 g/kg

.4 PHYSICAL PROPERTIES

.4.1 Acidity or alkalinity (note 4)

Maximum acidity : 0.5 g/kg calculated as H2S04

Maximum alkalinity : 0.5 g/kg calculated as NaOH

.4.2 Flash point (MT 12, CIPAC I, p. 846)

If required, the flash point of the product shall not be lower than the minimum declared flash point. A closed cup method shall be used and the method stated. (note 5)

.4.3 Miscibility with hydrocarbon oil (MT 23, CIPAC I, p. 891)

If required, the product shall be miscible with the appropriate hydrocarbon oil. (note

6)

.5 STORAGE STABILITY

5.1 Stability at 0 C (MT 39.1, CIPAC I, p. 930)

After storage at 0 +/- 1° C for 7 days, the volume of solid and/or Liquid which separates shall be not more than 0.3 ml.

5.2 Stability at 54 C (MT 46.1.3, CIPAC I, p. 952)

After storage at 54 +/- 2° C for 14 days, the product shall continue to comply with .2.2 and .4.1.

NOTE 1	Use the method 89/EC/M2/2 CIPAC IC, p. 2114.
NOTE 2	Use the method 89/EC/M2/3 CIPAC IC, p. 2114 (Referee Method) or 89/5/(M)/1.2, CIPAC I, p. 371, as starting point for further development.
NOTE 3	If the buyer requires both g/Kg and g/l at 20°C, then in case of dispute, the analytical result shall be calculated as g/kg.
NOTE 4	The method MT 31.1.3 CIPAC I, p. 904 or 31.2.3 p. 905 have not been tested on this type of formulation but may be used as a starting point for further development.
NOTE 5	Attention is drawn to the appropriate national and international regulations on handling and transport of flammable materials.
NOTE 6	The concentration should not be higher than the highest concentration recommended in the instructions for use.

ENDOSULFAN EMULSIFIABLE CONCENTRATES

FAO Specification 89/EC/S (1989)

.1 **DESCRIPTION**

The material shall consist of technical endosulfan (complying with the requirements of FAO specification 89/TC/S (1989)) dissolved in suitable solvents and with any other necessary formulants. It shall be in the form of a stable liquid, free from visible suspended matter and sediment.

.2 ACTIVE INGREDIENT

.2.1 Identity tests (CIPAC IC, 89/EC/M2/2, p. 2114)

Where the identity of the active ingredient is in doubt, then the isolated active ingredient shall comply with at least one additional test.

.2.2 Endosulfan (CIPAC IC, 89/EC/M2/3, p. 2114 (Referee method) or CIPAC I, 89/5/(M)/1.2, p. 371)

The endosulfan content shall be declared (g/kg or g/l at 20 C, Note 1) and when determined, the content obtained shall not differ from that declared by more than the following amounts:

Declared content	Permitted tolerance
up to 500 g/kg or g/l	+/- 5% of the declared content
above 500 g/kg or g/l	+/- 25 g

.3 IMPURITIES

.3.1 Water (MT 30.1, CIPAC I, p. 897)

Maximum : 0.5 g/kg

.4 PHYSICAL PROPERTIES

.4.1 Acidity or alkalinity (MT 31.1.3, CIPAC I p. 904 or 31.2.3 p. 905)

Maximum acidity : 0.5 g/kg H2SO4

Maximum alkalinity : 0.5 g/kg calculated as NaOH

.4.2 Emulsion stability and re-emulsification (MT 36.1.1., CIPAC I, p. 910)

After the heat stability test (5.2), the product, when diluted (note 2) at 30 C (note 3) with CIPAC Standard Waters A and C, shall comply with the following:

Time after dilution	Limits of stability
0 h	Initial emulsification complete
0.5 h	"Cream", maximum : 2 ml
2.0 h	"Cream", maximum : 4 ml
	"Free oil", nil
24 h (note 4)	Re-emulsification complete
24.5 h (note 4)	"Cream", maximum : 4 ml
	"Free oil", maximum : 0.5 ml

In special cases, a test using CIPAC Standard Waters A and C before the heat stability test may be necessary.

Alternatively, if the buyer requires other CIPAC Standard Waters to be used, then this shall be specified when ordering.

.4.3 Flash point (MT 12, CIPAC I, p. 846)

If required, the flash point of the product shall not be lower than the minimum declared flash point. A closed cup method shall be used and the method stated. (note 5)

.5 STORAGE STABILITY

.5.1 Stability at 0°C (MT 39.1, CIPAC I, p. 930)

After storage at 0 +/-1°C for 7 days, the volume of solid and/or liquid which separates shall be not more than 0.3 ml.

.5.2 Stability at 54°C (MT 46.1.3, CIPAC I, p. 952)

After storage at 54 +/- 2° C for 14 days, the product shall continue to comply with .2.2 and 4.1.

- NOTE 1 If the buyer requires both g/kg and g/l at 20°C, then in case of dispute, the analytical result shall be calculated as g/kg.
- NOTE 2 Do not top up the spray tank with fresh concentrate.

- NOTE 3 Unless another temperature is specified
- NOTE 4 These tests need only be carried out in case of doubt as to the emulsion stability result of the 2 hour test.
- NOTE 5 Attention is drawn to the appropriate national and international regulations on handling and transport of flammable materials.