



Food and Agriculture Organization
of the United Nations

FAO Specifications for Plant Protection Products

PROFENOFOS (AGP:CP/359)

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED
NATIONS Rome, 1998

CONTENTS

DISCLAIMER.....	3
INTRODUCTION.....	4
SUBMISSION OF DRAFT SPECIFICATIONS TO FAO	7
PROFENOFOS.....	8
Emulsifiable concentrates.....	11
Ultra low volume liquids	14

DISCLAIMER ¹

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/pest-and-pesticide-management/en/>)

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)

FAO Tentative Specifications (Code 'S/T', formerly 'TS') 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.

FAO Provisional Specifications [Code 'S/P', formerly ('S')] 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 $1 \times 10^3 \text{ m}^2/\text{s}$ 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: <http://www.fao.org/pest-and-pesticide-management/en/>).

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 FAO Provisional Specifications, or full FAO Specifications.

AGP:CP/359, 1997

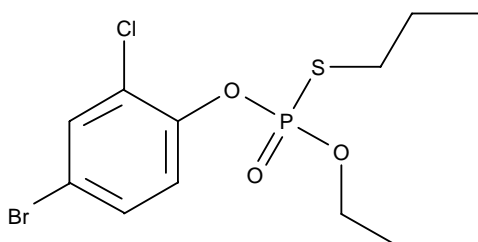
PROFENOFOS

O-4-bromo-2-chlorophenyl O-ethyl S-propyl phosphorothioate

INFORMATION

COMMON NAME: profenofos (ISO)

STRUCTURAL FORMULA:



EMPIRICAL FORMULA: C₁₁H₁₅BrClO₃PS

RMM: 373.6

CAS REGISTRY NUMBER: 41198-08-7

CIPAC CODE NUMBER: 461

CHEMICAL NAMES:

O-4-bromo-2-chlorophenyl O-ethyl S-propyl phosphorothioate (IUPAC).

O-(4-bromo-2-chlorophenyl) O-ethyl S-n-propyl phosphorothioate (CA).

PROFENOFOS TECHNICAL

FAO Specification 461/TC/S/F (1997)

1. DESCRIPTION

The material shall consist of profenofos, together with related manufacturing impurities and shall be in the form of a clear to slightly turbid, yellowish liquid, free from visible extraneous matter and added modifying agents.

2. ACTIVE INGREDIENT

2.1. Identity test (461/TC/M/2, CIPAC H, p. 222)

An identity test is required if the identity of the active ingredient is in doubt (e.g. IR spectrum).

2.2. Profenofos (461/TC/M/3, CIPAC H, p. 222)

The profenofos content shall be declared (not less than 890 g/kg) and, when determined, the content obtained shall not differ from that declared by more than ± 25 g/kg.

3. IMPURITIES

3.1. 4-Bromo-2-chlorophenol (Note 1)

Maximum: 1.0 %.

3.2. Water (MT 30.1, CIPAC F, p.91)

Maximum: 0.2 %.

Note 1 The analytical method is available from the Pesticide Management Group of the FAO Plant Protection Service or can be [downloaded here](#).

PROFENOFOS EMULSIFIABLE CONCENTRATES

FAO Specification 461/EC/S/F (1997)

1. DESCRIPTION

The material shall consist of technical profenofos, complying with the requirements of FAO specification 461/TC/S/F (1997), dissolved in suitable solvents together 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 test (461/EC/M/2, CIPAC H, p. 222)

An identity test is required if the identity of the active ingredient is in doubt (e.g. IR spectrum).

2.2. Profenofos (461/EC/M/3, CIPAC H, p. 222)

The profenofos content shall be declared (g/kg or g/l at $20 \pm 0.5^\circ\text{C}$. Note 2) and, when determined, the content obtained shall not differ from that declared by more than the following amounts:

<u>Declared content</u>	<u>Permitted tolerance</u>
Above 100 up to 250 g/kg or g/l	$\pm 6\%$ of the declared content
Above 250 up to 500 g/kg or g/l	$\pm 5\%$ of the declared content
Above 500 g/kg or g/l	± 25 g/kg or g/l

3. PHYSICAL PROPERTIES

3.1. pH range (MT 75.2, CIPAC F, p.206)

pH range: 3.0 to 7.0.

3.2. Emulsion stability and re-emulsification (MT 173, CIPAC F, p. 431)

The product, when diluted at $30 \pm 2^\circ\text{C}$ (Note 3) with CIPAC standard waters A and D, shall comply with the following:

<u>Time after dilution</u>	<u>Limits of stability</u>
----------------------------	----------------------------

0h	Initial emulsification complete
0.5h	Minimum 70 %
2.0h	Minimum 50 %
24h	Re-emulsification complete (Note 4)
24.5h	Minimum 50 % (Note 4)

Alternatively, if the buyer requires other CIPAC standard waters or temperature to be used, then this shall be specified when ordering.

3.3. Flash point (MT 12 .2, CIPAC F, p.37)

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. STORAGE STABILITY

4.1. Stability at 0°C (MT 39.1, CIPAC F, p.128)

After storage at $0 \pm 1^\circ\text{C}$ for 7 days, no solid or liquid shall separate.

4.2. Stability at 54 °C (MT 46.1.3 , CIPAC F, p.148. Note 6)

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 under 2.2. and the product shall continue to comply with 3.1. and 3.2.

Note 1 Methods available from the Pesticide Information Officer, FAO Plant Production and Protection Division.

Note 2 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 3 The product should be tested at the highest and lowest rates of use recommended by the supplier. The volume of water per ha is variable.

Note 4 This test needs only be carried out in case of doubt as to the emulsion stability result of the 2 hours test.

Note 5 Attention is drawn to the appropriate national and international regulations on handling and transport of flammable materials.

Note 6 Samples of the product taken before and after the storage stability test should be analyzed together after the test to reduce the analytical error.

PROFENOFOS ULTRA LOW VOLUME LIQUIDS

FAO Specification 461/UL/S/F (1997)

1. DESCRIPTION

The material shall consist of technical profenofos, complying with the requirements of FAO specification 461/TC/S/F (1997) together with any other necessary formulants. It shall be in the form of a stable liquid, free from visible suspended matter and sediment. The solution is ready for use through ULV equipment.

2. ACTIVE INGREDIENT

2.1. Identity test (461/UL/M/2, CIPAC H, p. 222)

An identity test is required if the identity of the active ingredient is in doubt (e.g. IR spectrum).

2.2. Profenofos (CIPAC 461/UL/M/3, CIPAC H, p. 222)

The profenofos content shall be declared (g/kg or g/l at $20 \pm 0.5^{\circ}\text{C}$. Note 2) and, when determined, the content obtained shall not differ from that declared by more than the following amounts:

Declared content

Above 100 up to 250 g/kg or g/l

Above 250 up to 500 g/kg or g/l

Permitted tolerance

$\pm 6\%$ of the declared content

$\pm 5\%$ of the declared content

3. PHYSICAL PROPERTIES

3.1. pH range (MT 75.2, CIPAC F, p.205)

pH range: 3.0 to 7.0.

3.2. Flash point (MT 12, CIPAC F, p.31)

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 3).

3.3. Kinematic viscosity range (MT 22, CIPAC F, p.75)

The viscosity range shall be specified and the viscosity measurement shall be carried out with a U-tube viscometer (Note 4).

4. STORAGE STABILITY

4.1. Stability at 0°C (MT 39.1, CIPAC F, p.128)

After storage at $0 \pm 1^\circ\text{C}$ for 7 days, no solid or liquid shall separate.

4.2. Stability at 54°C (MT 46.1.3, CIPAC F, p.150. Note 5)

After storage at $54 \pm 2^\circ\text{C}$ for 14 days, the determined average active ingredient content must not be lower than 96 % relative to the determined average content found under 2.2. and the product shall continue to comply with 3.1.

Note 1 The analytical method is available from the Pesticide Management Group of the FAO Plant Protection Service or can be [downloaded here](#).

Note 2 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 3 Attention is drawn to the appropriate national and international regulations on handling and transport of flammable materials.

Note 4 Viscosity per se is not a quality criteria of the product. However, if the formulation is applied through ground equipment, in which the flow rate depends on viscosity, a viscosity range shall be specified, e.g. 5 to 10 mm²/s or 10 to 16 mm²/s at $30 \pm 2^\circ\text{C}$. (1 mm²/s = 1 cSt).

Note 5 Samples of the product taken before and after the storage stability test should be analyzed together after the test to reduce the analytical error.

Determination of 4-Bromo-2-chlorophenol in Technical Grade Profenofos

Author: Dr. B. Schneider, Ciba Crop Protection, 4333 Münchwilen, Switzerland

This analytical method allows the quantitative determination of 4-bromo-2-chlorophenol in Profenofos technical material. Its determination in formulations might require modifications of this method depending on the nature of the auxiliaries present in the formulations. The method is based on gas chromatography and uses internal standard calibration for quantification.

1. Apparatus

The following details are examples of suitable equipment and conditions for its use.

Gaschromatograph:	Carlo Erba HRGC 5300 with flame ionisation detector
Integrator:	Spectra Physics Chromjet
Column:	Fused silica, Length: 15 m, Diameter: 0.32 mm Stationary phase: OV 1701 Film thickness: 0.25 µm Available from: J & W Scientific
Temperatures: Column:	Hold for 3 min at 50 °C, then set programme to bring the temperature to 160 ° at 10 °C / min., then heat to 280 °C at 4 °C / min and keep this temperature for 10 min.
Detector:	280 °C
Injection technique:	On-column
Carrier gas:	Hydrogen, 45 cm / min (determined with dichloromethane)
Make up gas:	Helium
Sensitivity:	range 1, attenuation 2 ¹⁰
Size of sample:	1 µl of reference solution / test solution prepared as described in paragraph 2
Duration of chromatography:	about 50 minutes

2. Preparation of Reference and Test Solutions

Internal standard:	Lindane = γ -Hexachlorocyclohexane Purity > 98 %
Reference substance:	4-Bromo-2-chlorophenol of known content
Internal standard stock solution:	Weigh 0.450 - 0.550 g of internal standard into a 100 ml volumetric flask. Make up to the mark with dichloromethane.
Reference solution:	Weigh 0.0500 - 0.0750 g of 4-bromo-2-chlorophenol into a 250 ml volumetric flask and make up to the mark with dichloromethane. Pipette 5.0 ml of this solution into a 100 ml volumetric flask and after addition of 5.0 ml internal standard stock solution dilute up to the mark with dichloromethane.
Test solution:	Weigh 90 to 100 mg of test substance into a 100 ml volumetric flask. Add 5.0 ml of internal standard stock solution and dilute up to the mark with dichloromethane.

3. Method

Inject reference solution until the internal standard / reference substance area quotients obtained for two consecutive chromatograms differ from one another by no more than 2%. From then on the following injection sequence is recommended:

... S₁ S₁ C S₂ S₂ C ...

C = reference solution

S = test solution (1, 2, ...n)

4. Retention Times (Typical Values)

Component	Retention time (minutes)	Relative retention
4-Bromo-2-chlorophenol	10.5	0.59
Internal standard (lindane)	17.8	1.00

5. Evaluation

Comparison of peak areas. The correction factor used in the calculation can be calculated from several single correction factors using a suitable averaging method.

5.1 Correction factor f

$$f = \frac{0.02 \cdot W_C \cdot F_{CI}}{F_C \cdot W_{CI}}$$

F_C = Peak area of 4-bromo-2-chlorophenol on the reference chromatogram

W_C = Weight of the reference substance of 4-bromo-2-chlorophenol, corrected for purity.

F_{CI} = Peak area of internal standard (lindane) on the reference chromatogram

W_{CI} = Weigh of the internal standard for the reference solution.

Note: W_{CI} is equal to the weigh of the internal standard in 5.0 ml of the stock solution when a stock solution of the internal standard is used.

5.2 Content

$$\% = \frac{F_S \cdot W_{SI} \cdot f \cdot 100}{W_S \cdot F_{SI}}$$

F_S = Peak area of 4-bromo-2-chlorophenol on the test chromatogram

W_S = Weight of the test sample

F_{SI} = Peak area of internal standard on the test chromatogram

W_{SI} = Weigh of the internal standard for the test solution.

Note: W_{SI} is equal to the weigh of the internal standard in 5.0 ml of the stock solution when a stock solution of the internal standard is used.

6. Example of a Gas Chromatogram of a Test Solution