AGP : CP / 39

FAO SPECIFICATIONS
FOR PLANT PROTECTION PRODUCTS

PYRETHRUM
AND
PIPERONYL BUTOXIDE

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS
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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/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)

   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 1×10^3 m^2/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.

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[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/ag/agp/agpp/pesticid/).

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.

PYRETHRUM EXTRACT
(See note 1)

FAO Specification Code 32/1a/S/4:

.1 DESCRIPTION

The material shall consist of the clear extracts at 20ºC (see note 2) obtained from pyrethrum flowers (Chrysanthemum cinerariaefolium); suitable for compounding insecticide formulations. Hydro carbon solvents used for dilution, such as refine kerosene, and anti-oxidants are permitted.

.2 ACTIVE INGREDIENT

.2.1 Total Pyrethrins (see CIPAC I; p.599, section 1.3, method 32/1a/M/1.3)

The total pyrethrins content (minimum: 20% w/w) shall be declared and, when determined, the content obtained shall not differ from that declared by more than ± 5% of the declared content.

.2.2 Pyrethrins I and II (Ibid)

The minimum contents of pyrethrins I and II shall be declared.

.3 IMPURITIES

.3.1 Water (Ibid, p.606, section 1.5)

Maximum: 0.5%.

.3.2 Material Insoluble in Dichlorodifluoromethane (Ibid, p.606, section 1.6))

Maximum: 1.5% (see note 3).

.4. CONTAINERS

The extract shall be packed in metal cans or drums with an interior lining of tin, or epoxy, or baked phenolic resin that will not adversely affect the stability of the extract.

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

PYRETHRUM DUSTS

FAO Specification Code 32/2/S/4:

.1 DESCRIPTION

The product shall consist of a homogeneous mixture containing pyrethrum extract as the only active ingredient, together with carriers and any necessary formulants. Synergists may also be included. The product shall be a coarse, free flowing powder, free from visible extraneous matter and hard aggregates.

The pyrethrum extract used in the product shall comply with the specification for "Pyrethrum Extract" and "Piperonyl Butoxide, if used (see p.1 and p.11, respectively).

.2 ACTIVE INGREDIENT

.2.1 Pyrethrins (see CIPAC I; p.608, section 1.2, method 32/2/M/1.2)

The pyrethrins content shall be declared and, when determined, the percentage obtained shall not differ from that declared by more than + 10 to -5% of the declared content.

.2.2 Pyrethrins I and II (Ibid)

The minimum contents of both pyrethrins I and II shall be declared.

.2.3 Synergists (Ibid, p.608, section 1.3 –see note 4)

If the formulation contains piperonyl butoxide or other synergists (see note 5) content shall be declared and, when determined, the percentage obtained shall not differ from that declared by more ± 5% of the declared content.

.3 IMPURITIES

.3.1 Water (Ibid, p.609, section 1.5)

Maximum: 1.0% (see note 6).

.4 PHYSICAL PROPERTIES

.4.1 pH (Ibid, p.608, section 1.4)

Minimum: 6.0.

Maximum: 7.0.

.4.2 Dry Sieve Test (Ibid, p.609, section 1.6)

Not less that 99% shall pass through a 150 µm test sieve.

.5 STORAGE STABILITY

.5.1 Heat Stability (Ibid, p.609, section 1.7)

After storage at 40 ± 2ºC for 28 days, the product shall continue to comply with .2.1, .2.2, .2.3 and .4.1 (see note 7).

.6 CONTAINERS

They shall be suitable, clean, dry, and as specified in the order. They shall not affect, or be affected by, the product, but shall adequately protect it from external influences.

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

PYRETHRUM SOLUTIONS
Syngergized by Piperonyl Butoxide

FAO Specification Code 32/4/S/4:

.1 DESCRIPTION

The product shall consist of a clear petroleum hydrocarbon solution synergized by technical piperonyl butoxide (see note 8).

It shall be formulated from pyrethrum extract and piperonyl butoxide complying with the specifications for "Pyrethrum Extract" and "Piperonyl Butoxide Technical"(see p.4 and p.10, respectively).

.2 ACTIVE INGREDIENTS

.2.1 Pyrethrins (see CIPAC I; p.609, section 1.3, method 32/4/M/1.3)

The content of pyrethrins (% w/w and/or g/l at 20ºC) shall be declared and, when determined, the content obtained shall not differ from that declared by more than ± 5% of the declared content.

.2.2 Pyrethrins I and II (Ibid)

The minimum contents of both pyrethrins I and II shall be declared.

.2.3 Piperonyl butoxide (see note 4)

The piperonyl butoxide content (% w/w and/or g/l at 20ºC) shall be declared and, when determined, the content obtained shall not differ from that declared by more than ± 5% of the declared content.

.3 PHYSICAL PROPERTIES

.3.1 Flash Point (Ibid, p.609, section 1.5)

The flash point of the finished product shall not be lower than the minimum declared flash point. The procedure used shall be stated, e.g., Abel Method.

.3.2 Odour

The solvent used shall not impart objectionable odours to the finished product.

.4 STORAGE STABILITY

.4.1 Heat Stability (Ibid, p.952, MT 46.1.3 – see note 9)

After storage at 40 ± 2ºC for 28 days, the product shall continue to comply with .2.1 and .2.2 (see note 7).

.5 CONTAINERS

They shall be suitable, clean, dry and as specified in the order. They shall not affect, or be affected by, the product, but shall adequately protect it from external influences.

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

PIPERONYL BUTOXIDE TECHNICAL

Draft Specification Code 33/1/S/4:

.1 DESCRIPTION

Technical piperonyl butoxide (see note 10) consists, essentially, of clear amber liquid, free from extraneous materials or added modifying agents.

.2 ACTIVE INGREDIENT (CIPAC Method 33/1/M/1.3 – see note 11)

The piperonyl butoxide content shall be declared (minimum: 90% w/w) and, when determined, the content obtained shall not differ from that declared by more than ± 2 percentage units.

.3 PHYSICAL PROPERTIES

.3.1 Specific gravity (CIPAC I, MT 3.2. p. 832)

Minimum: 1.045.
Maximum: 1.080.

.3.2 Refractive index (CIPAC Method 33/1/M/1.4 – see note 12)

Minimum: 1.4920.
Maximum: 1.5100 at 20ºC

.4 CONTAINERS

The piperonyl butoxide shall be supplied in metal cans or drums (see note 13) having an interior lining that will not adversely affect the stability or colour of the product.
The containers shall comply with pertinent national and international transport and safety regulations.

NOTES

1. This is a technical material

2. Prolonged storage at temperature above 40ºC may lead to decomposition of the pyrethrins.

3. Omit for other than use in aerosols.

4. Method not in CIPAC I. However, method “Piperonyl butoxide – Official Final Action” in “AOAC” methods of analysis” 11th ed., 1970, p. 116, is suitable and may be used.

5. If synergists other than piperonyl butoxide are included, the relevant methods of analysis must be supplied by the manufacturer.

6. Higher water content may cause decomposition of the pyrethrins.

7. It shall be prominently stated on the label that the product should not be stored at temperatures above 40ºC.

8. A typical ratio is one part by weight of pyrethrins with six parts of piperonly butoxide.

9. The solution shall be stored in sealed glass ampoules.

10. Piperonyl butoxide is the trivial name for 5-[2-(2-butoxyethoxy) ethoxymethyl]-6-proply-1,3-benzodioxole, or 3,4-methylene dioxy-6-proply-benzyl (butyl) diethylene glycol ether, or a-[2-(butoxyethoxy)ethoxy] -4,5 methylene dioxy-2-propyltoluene.

11. Method of analysis not included in CIPAC I. However, methods given in "AOAC Methods of Analysis" (11th ed., 1970, p. 116) are suitable, except that the standard should be purified as follows:

    Piperonyl butoxide Reference Standard

    The proposed method (AOAC 11th ed.) requires a reference standard for the colorimetric estimation; it is stated that the reference standard may be prepared by low-pressure fractional distillation of technical piperonyl butoxide without giving details. Samples may now be obtained from The National Physical Laboratory (NPL), Teddington, Middlesex, England.

    Purity of Reference Standard

    Whereas the AOAC (11th ed.) made the statement that the reference should be taken as 100% purity, it has been found that the purity is 98 ± 1%. The National Physical Laboratory, has checked purity by thin layer chromatography and gas-liquid chromatography.

    Preparation of Reference Standard

    Place technical piperonyl butoxide (60ml) in a 100ml Claisen flask fitted with a short Vigreux column (3-4"). * Connect to receivers and a suitable vacuum pump capable of maintaining a vacuum of 0.1 -1.0 Torr, and distil. Reject the first 20 ml of distillate and collect the next 25ml of distillate. The indicated temperature will be in the range 165 - 195ºC.

* A convenient distillation head and condenser is supplied by Bantamware, ref. K287450.

12. Method not included in CIPAC I.

13. Unlined mild steel drums should not be used as they cause discolouration.