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FAO SPECIFICATIONS FOR PLANT PROTECTION PRODUCTS

DINOTERB

2-tert-butyl-4,6-dinitrophenol

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

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

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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/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 x 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.

<u>Technical materials, dustable powders and granules</u>

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.

<u>Wettable powders</u>

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, Via delle Terme di Caracalla, 00153, Rome, Italy.

General guidelines in 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.

INFORMATION

COMMON NAME:	Dinoterb (ISO)
EMPIRICAL FORMULA:	$C_{10}H_{12}N_2O_5$
RMM:	240.2
CAS REGISTRY NUMBER:	1420-07-1
CIPAC CODE NUMBER:	238

CHEMICAL NAME:

2-tert-butyl-4,6-dinitrophenol (IUPAC)
2-(1,1-dimethylethyl)-4,6-dinitrophenol (CA)

DINOTERB TECHNICAL

FAO Specification 238/TC/S (1990)

.1 DESCRIPTION

The material shall consist of dinoterb together with related manufacturing impurities and shall be a yellow to orange crystalline solid free from visible extraneous matter and added modifying agents.

The material may contain up to about 50 g/kg of water (Note 1).

.2 ACTIVE INGREDIENT

.2.1 Identity tests (See methods in Appendix 1)

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

.2.2 Dinoterb (CIPAC 1B, 238/TC/M/3, p.1797)

The dinoterb content shall be declared on a dry weight basis (not less than 990 g/kg) and when determined, the content obtained shall not differ from that declared by more than +/- 20 g.

.3 IMPURITIES

.3.1 Water (MT 30.1, CIPAC 1, p.897)

The average content of water in the batch supplied shall be declared.

.3.2 Free Mineral Acidity (see method in Appendix 2)

Maximum: 5 g/kg of the dry weight, expressed as H_2SO_4

.3.3 Inorganic nitrites (see method in Appendix 3)

Maximum : 2 mg/kg of the dry weight, expressed as sodium nitrite.

Note 1 For safety considerations in the manufacturing process dinoterb is generally supplied as a damp material.

Note 2 This includes any kind of salts of nitrous acid.

DINOTERB SALT-SOLUBLE CONCENTRATES

FAO Specification 238/SL/S (1990)

.1 DESCRIPTION

The material shall consist of a solution of technical dinoterb, complying with the requirements of FAO specification 238/TC/S (1990) in the form of salts, together with any necessary formulants. It shall be free from visible suspended matter and sediment.

.2 ACTIVE INGREDIENT

.2.1 Identity test (see methods in Appendix 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 Dinoterb (CIPAC 1B, 238.1/SL/M/3, p.1800)

The dinoterb 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 (g/kg or g/l)	Permitted tolerance
Up to 250	+/- 6% of the declared content
Above 250 up to 500	+/- 5% of the declared content
Above 500	+/- 25 g

.3 PHYSICAL PROPERTIES

.3.1 pH range (MT 75, CIPAC LA, p.1589)

If required, the pH range shall be declared.

.3.2 Flash point (MT 12, CIPAC 1A, p.846) (Note 2)

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.

.3.3 Miscibility with water (MT 41, CIPAC 1, p.933)

The product shall give a clear to slightly turbid solution after dilution and standing for 18h at 20°C with CIPAC Standard Water C. No coarse sediment shall be observable. (Note 3).

.4 STORAGE STABILITY

.4.1 Stability at O C (MT 39, CIPAC 1, p.930)

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

.4.2 Stability at 54 C (MT 46.1.3, CIPAC 1, p.930)

After storage at 54 +/- 2°C for 14 days, the product shall continue to comply with 2.2, 3.1 and 3.3.

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

Note 3 The concentration should not be higher than the highest concentration recommended in the instructions for use.

APPENDIX 1

IDENTITY TESTS

.1 Technical material

.1.1 Use the GLC method 238/TC/M/3 (CIPAC 1B, p.1797). The relative retention time of dinoterb with respect to the internal standard for the sample should not deviate by more than 1% from that for the calibration solution.

.1.2 As additional test in case of doubt, determine the melting point range by applying the method MT 2, CIPAC 1A, p.1552, but after drying the damp material in an oven at 65 C for 15 h. Melting point range: 125-127 C.

.2 Soluble concentrates

.2.1 Use the GLC method 238.1/SL/M/3 (CIPAC 1B, p.1800). The relative retention time of dinoterb with respect to the internal standard for the sample should not deviate by more than 1% from that for the calibration solution.

.2.2 As additional test in case of doubt, determine the melting point range by applying the method MT 2, CIPAC 1A, p.1552, after extracting the active ingredient as follows: "acidify 1 ml of the sample by adding 15 ml of 1N H_2SO_4 solution in a separating funnel. Extract with 15 ml of diethylether. Evaporate the solvent and dry in an oven at 65 C for 15 h."

The melting point of the isolated material should be 126-127°C and shall not be depressed by admixture of an equal amount of pure dinoterb.

APPENDIX 2

DETERMINATION OF FREE MINERAL ACIDITY

OUTLINE OF METHOD

The acidity is determined by titration with standard alkali. The end point is determined electrometrically.

REAGENTS

Hydrochloric acid: O.1 N solution
Methanol
Sodium hydroxide : O.1 N standardized solution

APPARATUS

pH meter
10 ml burette
250 ml measuring cylinder
5 ml pipette

PROCEDURE

Weigh, accurately, about 1 g (w g) of sample and dissolve in 101 ml of methanol. Add 20 ml of water and 5 ml of 0.1 N hydrochloric acid. Homogenize. Titrate electrometrically with 0.1 N sodium hydroxide to the first end point with a pH value lying between 3 and 4 (t ml).

Carry out a blank determination on methanol (100 ml), distilled water (20 ml) and 0.1 N hydrochloric acid (5 ml) with 0.1 N sodium hydroxide (b ml).

Acidity calculated as H_2SO_4 = $\frac{49.04\ N\ (t - b)\ g/kg}{w}$

Where: N = normality of the sodium hydroxide.

APPENDIX 3

DETERMINATION OF INORGANIC NITRITES

OUTLINE OF METHOD

Dissolve the sample in acetic acid. Add Griess reagent and measure the absorption of the formed complex at 550 nm.

REAGENTS

Acetic acid
Hydrochloric acid 6 mol/1 in water
N-(1-Naphthyl) ethylenediamine, dihydrochloride
Sodium nitrite
Sulfanilic acid

Solution A: dissolve 100 mg of N-(1-naphthyl) ethylenediamine dihydrochloride in a 30% v/v acetic acid solution in 100 ml of water.

Solution B: dissolve 1 g of sulfanilic acid in a 30% v/v acetic acid solution in 100 ml of water.

Solution C (Griess reagent): mix 100 ml of solution and 100 ml of the solution B. The solution C must be used the same day as for the preparation.

APPARATUS

Pipettes
50, 100 and 1000 ml volumetric flasks
Quartz cuvettes, 5 cm
Spectrophotometer set at 550 nm
Thermostated bath

PROCEDURE

a) Calibration

Dissolve 0.050 g of sodium nitrite in water and dilute to 1000 ml in a volumetric flask. Dilute 1 ml of this stock solution to 100 ml with water in a volumetric flask (working solution). Transfer 0, 0.1, 0.2, 0.3, 0.5, 0.7 and 1 ml of the nitrite working solution to 50 ml volumetric flasks, add 5 ml of acetic acid, mix the contents, add 2.5 ml of freshly prepared solution C, then 2.5 ml of hydrochloric acid, 6 mol/1, and mix again. Heat at 50 C for 30 minutes. Allow to cool at room temperature, dilute to 50 ml with acetic acid and mix. Measure the absorbance in a 5 cm cell using distilled water in the reference cell. Substract the blank test (0 ml of the nitrite working solution) from the reading obtained on the nitrite solutions. Hence prepare a calibration graph plotting ml of the nitrite working solution against absorbance.

b) Determination

Weigh, to the nearest mg, about 500 mg of sample (wg), dissolve in and dilute to 50 ml in a volumetric flask. Pipette 5 ml of this solution into a 50 ml volumetric flask. Add 2.5 ml of freshly prepared solution C, then 2.5 ml of hydrochloric acid, 6 mol/1,
and mix. Measure the absorbance and deduct the blank test from the value obtained with the sample. Read off the number of ml of the nitrite working solution (x ml) equivalent to the absorbance found.

Sodium nitrite content = $\frac{5\ x}{W}$ mg/kg