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FAO SPECIFICATIONS FOR PLANT PROTECTION PRODUCTS

2,4-D

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

FAO specifications are developed with the basic objective of ensuring, as far as possible, that pesticides complying with them are satisfactory for the purpose for which they are intended. However, the Group on Pesticide Specifications of the FAO Panel of Experts on Pesticide Specifications, Registration Requirements and Application Standards wishes to emphasize that, owing to the complexity of the problem involved, questions such as the suitability of pesticides for the control of a particular pest must be decided at national or provincial level. These specifications should not be assumed to be an endorsement, by either the Group of Experts or FAO, of the use of a particular compound for a given purpose.

Accordingly, neither the Food and Agriculture Organization of the United Nations (FAO) nor the members of the Group on Pesticide Specifications of the FAO Panel of Experts on Pesticide Specifications, Registration Requirements and Application Standards warrant that pesticides complying with these specifications are suitable for control of any given pest or for use in any particular area.

Furthermore, the preparation and use of pesticides complying with these specifications are not exempt from any safety regulation or other legal or administrative provision applicable thereto. Neither FAO nor any member of the FAO Group of Experts shall be liable for any injury, loss, damage or prejudice of any kind that may be suffered as a result of the preparation or use of pesticides complying with these specifications.

Additionally, the Group of Experts wishes to warn users of specifications that improper field mixing and/or application of pesticides can result in either a lowering or complete loss of their efficacy. This holds true even in cases where such pesticides comply with the specifications indicated.

Accordingly, the Group of Experts and/or FAO can accept no responsibility for the consequences of improper field mixing and/or application.

INTRODUCTION

From time to time, FAO publishes 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, but during the interval between editions, revisions may be printed in the FAO Plant Protection Bulletin.

The specifications contained herein have been carefully reviewed and agreed by the Group on Pesticide Specifications of the FAO Panel of Experts on Pesticide Specifications, Registration Requirements and Application Standards after consultations with official government scientists, the pesticides industry through GIFAP (Groupement International des Associations Nationales de Fabricants de Produits Agrochimiques or, in English, International Group of National Associations of Manufacturers of Agrochemical Products) and, where appropriate, with individual manufacturers [1].

FAO has edited a *Manual on the development and use of FAO Specifications for Plant Protection Products*, FAO Plant Production and Protection Paper No. 85, Rome 1987 (available in English, French and Spanish from the FAO Plant Protection Service).

This manual contains 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 and Application Standards, such as:

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

   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].

   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 Tentative specifications (Code "S/T", formerly "ts") are those which have been recommended by FAO as preliminary specifications 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.

   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 $50^{o}C$) and viscous liquids (with minimum kinematic viscosity of 1×10^{-3} m^2/s at $20^{o}C$) 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 $20^{o}C$. If the buyer requires both g/kg and g/l at $20^{o}C$, 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 content of active ingredient. 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 product/material, but shall adequately protect it 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, oxidation, 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 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 1, p.965 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) and D (1988), 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. A copy of a method 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, 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 Pesticides Control Officer, Plant Production and Protection Division, FAO, Via delle Terme di Caracalla, 00100 Rome, Italy.

General guidelines on preparing draft specifications are given in Plant Production and Protection Paper 85, *Manual on the Development and Use of FAO Specifications for Plant Protection Products*, FAO, Rome, 1995 (English only).

Specifications which are considered suitable for further processing are assigned priorities and circulated to appropriate organizations and specialists for 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 and Application Standards. The drafts are converted into FAO Provisional Specifications, or full FAO Specifications.

2,4-D

(2,4-dichlorophenoxy)acetic acid

INFORMATION

COMMON NAME: 2,4-D (ISO)

STRUCTURAL FORMULA:

EMPIRICAL FORMULA: $C_8H_6Cl_2O_3$

RMM: 221.0

CAS REGISTRY NUMBER: 94-75-7

CIPAC CODE NUMBER: 1

CHEMICAL NAMES:

(2,4-dichlorophenoxy)acetic acid (IUPAC and CA)

2,4-D TECHNICAL

FAO Specification 1/TC/S/F (1992)

.1 DESCRIPTION

The material shall consist of 2,4-D together with related manufacturing impurities and shall be white to brown crystals, granules, flakes, powder or lumps with not more than slight odour. If shall be free from visible extraneous matter and added modifying agents.

.2 ACTIVE INGREDIENT

.2.1 Identity tests (1/TC/M3/2, CIPAC 1C, p.2060)

An identity test is required if the identity of the active ingredient is in doubt.

.2.2 2,4-D (1/TC/M3/5.1, GLC method, CIPAC 1C, p.2061, or 1/TC/M3/5.2, HPLC as referee method, p.2062)

The 2,4-D content of the material shall be declared (not less than 960 g/kg) and, when determined, the content obtained shall not differ from that declared by more than ± 15 g/kg.

.3 IMPURITIES

.3.1 Water (MT 30.1 CIPAC 1, p.897 as referee method or MT 30)

Maximum: 15 g/kg.

.3.2 Free phenols (MT 69.1, CIPAC 1, p.998)[1] (Note 1)

Maximum: 3 g/kg, calculated as 2,4-dichclorophenol.

.3.3 Sulphated ash (MT 29.1, CIPAC 1A, p.1562) (Note 2)

Maximum: 5 g/kg.

[1] Alternative method available from the Plant Protection Officer, FAO Plant Production and Protection Division

.3.4 Triethanolamine insolubles[2]

A triethanolamine solution of the material shall leave not more than 1 g/kg residue on a 105 µm test sieve and the sieved solution shall be clear or opalescent and shall contain not more than a trace of sediment.

Note 1 The content of free phenols is limited to avoid possible taint of neighbouring crops and foodstuffs. Moreover, free phenols are a potential source for generating chlorinated micro-contaminants.

Note 2 The mass of sample to be used should be 10 g.

[2] Method available from the Plant Protection Officer, FAO Plant Production and Protection Division

2,4-D SODIUM SALT TECHNICAL

FAO Specification 1.1Na/TC/S/F (1992)

.1 DESCRIPTION

The material shall consist of 2,4-D sodium salt monohydrate together with related manufacturing impurities and shall be a white to brown crystalline powder with not more than a slight odour. It shall be free from visible extraneous matter and added modifying agents.

.2 ACTIVE INGREDIENT

.2.1 Identity tests (1.1Na/TC/M2/2, CIPAC 1C, p.2063)

An identity test is required if the identity of the active ingredient is in doubt.

.2.2 2,4-D (1.1Na/TC/M2/4.1, GLC method, CIPAC 1C, p.2065, or 1.1Na/TC/M2/4.2, HPLC as referee method, CIPAC 1C, p.2065)

The 2,4-D content of the material shall be declared (not less than 815 g/kg) and, when determined, the content obtained shall not differ from that declared by more than ± 2.5% of the declared content.

.3 IMPURITIES

.3.1 Water (Free water and water of hydration) (MT 30.1, CIPAC 1, p.897 as referee method or MT 30.2)

Maximum: 90 g/kg.

.3.2 Free phenols (MT 69.1, CIPAC 1, p.998)[3] (Note 1)

Maximum: 3 g/kg, calculated as 2,4-dichlorophenol, of the 2,4-D content found under .2.2.

[3] Alternative method available from the Plant Protection Officer, FAO Plant Production and Protection Division

.3.3 Water insolubles (MT 10.3 B, CIPAC 1, p.844)

An aqueous solution of the material shall pass completely through a 250 µm test sieve, and not more than 1 g/kg shall remain on a 150 µm test sieve. The sieved solution shall be clear or opalescent and shall contain not more than a trace of sediment.

.4 PHYSICAL PROPERTIES

.4.1 Rate of solution (MT 60, CIPAC 1, p.983)

All the material, other than the insoluble material content found under .3.3, shall dissolve in 5 min in distilled water and the solution, after standing for 18 h, shall have not more than a trace of additional sediment.

Note 1 The content of free phenols is limited to avoid possible taint of neighbouring crops and foodstuffs. Moreover, free phenols are a potential source for generating chlorinated micro-contaminants.

2,4-D TECHNICAL ESTERS

FAO Specification 1.3/TC/S/F (1992)

.1 DESCRIPTION

The material shall consist of 2,4-D technical ester(s), together with related manufacturing impurities. It shall be free from visible water and suspended matter.

.2 ACTIVE INGREDIENT

.2.1 Ester(s)

The names of the 2,4-D ester(s) present shall be declared, and in the case of mixed esters the approximate content of each shall be stated.

.2.2 Identity tests (1.3/TC/m2/2, CIPAC 1C, p.2068)

An identity test is required if the identity of the active ingredient is in doubt.

.2.3 2,4-D (1.3/TC/m2/4.1, GLC method, CIPAC 1C, p.2069, or 1.3/TC/m2/4.2, HPLC as referee method, CIPAC 1C, p.2071)

.2.3.1 Purity of ester

The ester purity shall be determined by HPLC by reference to analytical standard 2,4-D ester. The purity of the ester shall not be below 920 g/kg calculated on technical grade ester.
The nature of the alcohol shall be clearly declared.
Mixed esters shall be determined by direct HPLC analysis or acid equivalent analyzed after the acid has been cleaved.

.2.3.2 2,4-D content

The 2,4-D content shall be declared in g/kg of 2,4-D acid equivalent and shall not be lower than the quantity calculated using the formula:

$$\frac{\text{M. Wt. of 2,4-D} = 221}{\text{[M. Wt. of 2,4-D ester]}} \times 920 \text{ g/kg}$$

.3 **IMPURITIES**

.3.1 Free phenols (MT 69.1, CIPAC 1, p.998)[4] (Note 1)

Maximum: 3 g/kg, calculated as 2,4-dichlorophenol, of the 2,4-D content found under .2.3.

.3.2 Free acidity (MT 66, CIPAC 1, p.995)

Maximum:
15 g/kg for free acidity in low-volatility esters or
30 g/kg for free acidity in high-volatility esters.

The free acidity is expressed as 2,4-D calculated from the total ester sample. Volatility is determined according to MT 13, CIPAC 1, p.858.

.3.3 Suspended solids (MT 40.2, CIPAC 1, p.932)

Maximum: 1 g/kg.

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

Maximum: 10 g/kg.

Note 1 The content of free phenols is limited to avoid possible taint of neighbouring crops and foodstuffs. Moreover, free phenols are a potential source for generating chlorinated micro-contaminants.

[4] Alternative method available from the Plant Protection Officer, FAO Plant Production and Protection Division

2,4-D ESTER EMULSIFIABLE CONCENTRATES

FAO Specification 1.3/EC/S/F (1992)

.1 DESCRIPTION

The material shall consist of an emulsifiable concentrate based on 2,4-D ester(s), complying with the requirements of FAO specification 1.3/TC/S/F (1992), as the active ingredient(s) together with any necessary formulants. It shall be in the form of a stable liquid, free from visible suspended matter and sediment.

.2 ACTIVE INGREDIENT

.2.1 Ester(s)

The name(s) of the 2,4-d ester(s) present shall be declared and the approximate content of each ester shall be stated.

.2.2 Identity tests (1.3/EC/m2/2, CIPAC 1C, p.2071)

An identity test is required if the identity of the active ingredient is in doubt.

.2.3 2,4-D (1.3/EC/m2/4.1, GLC method, CIPAC 1C, p.2071, or 1.3/EC/m2/4.2, HPLC as referee method, CIPAC 1C, p.2071)

The 2,4-dichlorophenoxyacetic acid equivalent content shall be declared (g/kg or g/l at 20×, 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	± 4% of the declared content
above 500 g/kg or g/l	± 20 g/kg or g/l

.3 IMPURITIES

.3.1 Free phenols (MT 69.1, CIPAC 1, p.998)[5] (Note 2)

Maximum: 3 g/kg, calculated as 2,4-dichlorophenol, of the 2,4-D acid equivalent content found under .2.3.

[5] Alternative method available from the Plant Protection Officer, FAO Plant Production and Protection Division

.3.2 Oil insoluble material (MT 35, CIPAC 1, p.910)

The product shall give a clear, or opalescent, homogeneous solution which shall leave not more than 1 g/kg residue on a 150 µm test sieve, and the sieved solution shall contain not more than a trace of sediment.

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

Maximum: 5 g/kg.

.4 PHYSICAL PROPERTIES

.4.1 Emulsion stability and re-emulsification (MT 36.1.1, CIPAC 1, p.910) (Note 3)

The product, when diluted at 30×👌 (Notes 4 and 5) 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 6)	Re-emulsification: complete
24.5 h (Note 6)	"cream", maximum: 4 ml
	"free oil", maximum: 0.5 ml

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

.4.2 Flash point (MT 12, CIPAC 1, p.846) (Note 7)

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.

.4.3 Volatility (MT 13, CIPAC 1, p.858)

It shall be stated whether the "volatility" of the product is high or low.

.5 STORAGE STABILITY

.5.1 Stability at 0×👌 (MT 39.1, CIPAC 1, p.930)

After storage at 0 ± 1×👌 for 7 days, the volume of solid and/or liquid which separates shall not be more than 3 ml/l.

.5.2 Stability at 54×👌 (MT 46.1.3, CIPAC 1, p.952)

After storage at 54 ± 2°C for 14 days, the determined average active ingredient content must not be lower than 97% relative to the determined average content found before storage (Note 8) and the product shall continue to comply with .3.2 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 results shall be calculated as g/kg.

Note 2 The content of free phenols is limited to avoid possible taint of neighbouring crops and foodstuffs. Moreover, free phenols are a potential source for generating chlorinated micro-contaminants.

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

Note 4 Unless another temperature is specified.

Note 5 The product should be tested at the highest and lowest rates of use recommended by the supplier.

Note 6 These tests need only be carried out in case of doubt as to the result of the 2-hour emulsion stability test.

Note 7 Attention is drawn to the appropriate national and/or international regulations on the handling and transport of flammable materials.

Note 8 Samples of the product taken before and after the storage stability test should be analysed together after the test in order to reduce the analytical error.

2,4-D SALT AQUEOUS SOLUTIONS

FAO Specification 1.1/SL/S/F (1992)

.1 DESCRIPTION

The material shall consist of technical 2,4-D complying with the requirements of FAO specification 1/TC/S/F (1992) or 1.1Na/TC/S/F (1992) as the active ingredient, formulated as a 2,4-D salt aqueous solution. It shall be free from visible suspended matter and sediment.

.2 ACTIVE INGREDIENT

.2.1 Salt(s)

The name(s) of the salt(s) present shall be declared and the approximate content of each shall be stated.

.2.2 Identity tests (1.1 or 1.4/SL/M2/2, CIPAC 1C, p.2066)

An identity test is required if the identity of the active ingredient is in doubt.

.2.3 2,4-D (1.1 or 1.4/SL/M2/4.1, GLC method, CIPAC 1C, p.2067, or 1.1 or 1.4/SL/M2/4.2, HPLC as referee method, CIPAC 1C, p.2067)

The 2,4-D acid equivalent content shall be declared (g/kg or g/l at 20×, 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	± 4% of the declared content
above 500 g/kg or g/l	± 20 g/kg or g/l

.3 IMPURITIES

.3.1 Free phenols (MT 69.1, CIPAC 1, p.998)[6] (Note 2)

[6] Alternative method available from the Plant Protection Officer, FAO Plant Production and Protection Division

Maximum: 3 g/kg, calculated as 2,4-dichlorophenol, of the 2,4-D acid equivalent content found under .2.3.

.3.2 Water insolubles (MT 10.3 B, CIPAC 1, p.844)

The product shall pass through a 250 µm test sieve and not more than 1 g/kg shall remain on a 150 µm test sieve.

.4 PHYSICAL PROPERTIES

.4.1 Stability on dilution (MT 41, CIPAC 1, p.933)

The product, after dilution with CIPAC Standard Water C (Note 3), shall give a clear or opalescent solution at 20×. After standing for 1 h, any visible particles should pass through a 45 µm test sieve (Note 4).

.5 STORAGE STABILITY

.5.1 Stability at 54× (MT 46.1.3, CIPAC 1, p.952)

After storage at 54 ± 2× for 14 days,the determined average active ingredient content must not be lower than 97% relative to the determined average content found before storage (Note 5) and the product shall continue to comply with .3.2 and .4.1.

Note 1 If the buyer requires both g/kg and g/l at 20×, then in case of dispute the analytical results shall be calculated as g/kg.

Note 2 The content of free phenols is limited to avoid possible taint of neighbouring crops and foodstuffs. Moreover, free phenols are a potential source for generating chlorinated micro-contaminants.

Note 3 The dilution rate should be 5% v/v.

Note 4 Unless other temperatures, times and/or CIPAC Standard Waters are specified for particular products.

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