Food and Agriculture Organization
of the United Nations

FAO SPECIFICATIONS FOR PLANT PROTECTION PRODUCTS

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ETHEPHON

2-chloroethylphosphonic acid

2000

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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, Application Standards and Prior Informed Consent wishes to emphasize that, owing to the complexity of the problems 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 of the use of a particular compound for a given purpose by either the Group of Experts or FAO.

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, Application Standards and Prior Informed Consent 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 regulatory 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 revisions may be printed in the *FAO Plant Protection Bulletin* during the interval between editions.

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, Application Standards and Prior Informed Consent after consultations with official government scientists, the pesticides industry through GCPF (Global Crop Protection Federation) and, where appropriate, with individual manufacturers.[1]

FAO has published a *Manual on the development and use of FAO Specifications for Plant Protection Products*, FAO Plant Production and Protection Paper No. 149, Rome 1999 (available in English 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, 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, 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 Information Officer, Plant Production and Protection Division, FAO, Viale delle Terme di Caracalla, 00100 Rome, Italy.

General guidelines on preparing draft specifications are given in Plant Production and Protection Paper 149, *Manual on the Development and Use of FAO Specifications for Plant Protection Products, Fifth Edition,* FAO, Rome, 1999 (available in English or 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 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, Application Standards and Prior Informed Consent. The drafts are converted into FAO Provisional Specifications, or full FAO Specifications.

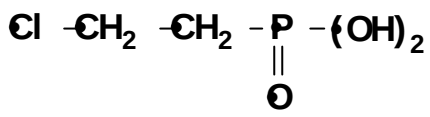
FAO SPECIFICATIONS FOR PLANT PROTECTION PRODUCTS

ETHEPHON

INFORMATION

COMMON NAME: ethephon

STRUCTURAL FORMULA:



RMM: 144.5

EMPIRICAL FORMULA: $C_2 H_6 Cl O_3 P$

C.A.S. NUMBER: 16672 - 87 – 0

CIPAC CODE NUMBER: 373

CHEMICAL NAME: 2-chloroethylphosphonic acid.(IUPAC)

(2-chloroethyl)phosponic acid (CA)

ETHEPHON TECHNICAL

FAO Specification 373/TC/S/F(2000)

.1 DESCRIPTION

The material shall consist of ethephon together with related manufacturing impurities and shall be a grayish white colored waxy solid free from visible extraneous matter and added modifying agents.

.2 ACTIVE INGREDIENT

.2.1 Identity tests (373/TK/M/2, CIPAC H, p.166)

An identity test is required if the identity of the active ingredient is in doubt (e.g. gas chromatographic retention time under analytical conditions described in the analysis of Mono 2-chloroethyl ester, 2-chloroethyl phosphonic acid : MEPHA).

.2.2 Ethephon (373/TK/M/3, CIPAC H, p.168)

The ethephon content shall be declared (not less than 910 g/kg) and, when determined, the content obtained shall not be lower than the declared content.

.3 IMPURITIES

.3.1 Manufacturing impurities (Note 1)

MEPHA : Mono 2-chloroethyl ester, 2-chloroethyl phosphonic acid : Maximum 20 g/kg

1,2-Dichloroethane : Maximum 0.5 g/kg

.4 PHYSICAL PROPERTIES

.4.1 pH range (MT 75, CIPAC F, p.205)

pH range : 1.5 to 2.0

NOTE

1.Click here to download the analytical method

ETHEPHON TECHNICAL CONCENTRATE

FAO Specification 373/TK/S/F (2000)

.1 DESCRIPTION

The material shall consist of an aqueous solution of ethephon, complying with the requirements of FAO specification 373/TC/S/F (2000), together with related manufacturing impurities and shall be a colorless to tan liquid or waxy solid, free from visible extraneous matter and added modifying agents. The aspect of the product shall be stated.

.2 ACTIVE INGREDIENT

.2.1 Identity tests (373/TK/M/2, CIPAC H, p.166)

An identity test is required if the identity of the active ingredient is in doubt (e.g. gas chromatographic retention time under analytical conditions described in the analysis of Mono 2-chloroethyl ester, 2-chloroethyl phosphonic acid : MEPHA).

.2.2 Ethephon (373/TK/M/3, CIPAC H, p.168)

The ethephon 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
Above 500 g/kg	± 25 g/kg

.3 IMPURITIES

.3.1 Manufacturing impurities (Note 1)

MEPHA : Mono 2-chloroethyl ester, 2-chloroethyl phosphonic acid : Maximum 2 % of the ethephon declared content

1,2-Dichloroethane : Maximum 0.04 % of the ethephon declared content

.3.2 Material insoluble in water (MT 10.3 B, CIPAC F, p.29)

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

.3.3 Water (MT 30.5, CIPAC I, to be published) (Note 1)

The water content shall be measured (g/kg), and the value obtained shall not be less than the following figure:

{1000 – (measured ethephon content in g/kg)/0.91} - 15

.4 PHYSICAL PROPERTIES

.4.1 pH range (MT 75, CIPAC F, p.205)

pH range : 1.5 to 2.0

NOTES:

1.Click here to download the analytical method

ETHEPHON SOLUBLE CONCENTRATES

FAO Specification 373/SL/S/F (2000)

.1 DESCRIPTION

The material shall consist of a solution of technical ethephon, complying with the requirements of FAO specification 373/TC/S/F (2000), dissolved in suitable solvents and with other necessary added formulants. It shall be a clear or opalescent colorless to tan liquid, free from visible suspended matter and sediment, to be applied as a true solution of the active ingredient in water. (Note 1)

.2 ACTIVE INGREDIENT

.2.1 Identity tests (373/SL/M/2, CIPAC H, p.170)

An identity test is required if the identity of the active ingredient is in doubt (e.g. gas chromatographic retention time under analytical conditions described in the analysis of Mono 2-chloroethyl ester, 2-chloroethyl phosphonic acid : MEPHA).

.2.2 Ethephon (373/SL/M/3, CIPAC H, p.170)

The ethephon content shall be declared (g/kg or g/L at 20 °C, Note 2) and, when determined, the content obtained shall not differ from that declared by more than the following amounts.

Declared content	Permitted tolerance
Above 100 up to 250 g/kg or g/L	± 6 % of the declared content
Above 250 g/kg up to 500 g/kg or g/L	± 5 % of the declared content
Above 500 g/kg or g/L	± 25 g/kg

.3 IMPURITIES

.3.1 Manufacturing impurities (Note 3)

MEPHA : Mono 2-chloroethyl ester, 2-chloroethyl phosphonic acid : Maximum 2 % of the ethephon declared content

1,2-Dichloroethane : Maximum 0.04 % of the ethephon declared content

.4 PHYSICAL PROPERTIES

.4.1 pH range (MT 75, CIPAC F, p.205)

pH range : 1.5 to 2.0

.4.2 Dilution stability in water (MT 41, CIPAC F, p.131)

The product after dilution with CIPAC Standard Water D shall give a clear or opalescent solution at 20 ± 2°C. After standing for one hour, any visible particles should pass through a 75 µm test sieve.

.5 STORAGE STABILITY

.5.1 Stability at 0 °C (MT 39, CIPAC F, p.128)

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

.5.2 Stability at 54 °C (MT 46.1.3, CIPAC F, p.150) (Note 4)

After storage at 54 °C ± 2 °C for 14 days, the determined average active ingredient content must not be lower than 95 % relative to the determined average content found before storage and the product shall continue to comply with .4.1 and .4.2.

NOTES

1. *Some products content opalescent additives for commercial identification.*

2. *If the buyer requires both g/kg and g/L at 20 °C then, in the case of dispute, the analytical results shall be calculated as g/kg.*

3. *Click here to download the analytical method*

4. *Analysis of the product before and after storage stability test should be carried out at the same time (i.e. after storage) to reduce the analytical error.*

DETERMINATION OF 1,2-DICHLOROETHANE IN ETHEPHON TECHNICAL MATERIALS AND FORMULATIONS

Scope

This method determines the amount of 1,2-dichloroethane in technical ethephon and its formulations, using a GC internal standard method.

Limitations

Any compound which has the same retention time as a component of interest will interfere.

Safety precautions

Personnel must be thoroughly acquainted with the potential hazards of the reagents, products, solvents, equipment, and procedures used in this facility. The current MSDS for the chemicals used for this method should be consulted. Due to the nature of the compounds, all solutions should be prepared in a negative air fume hood.

Reagents

Methylene chloride (DCM), HPLC grade
Monochlorobenzene, analytical grade
1,2-dichloroethane (EDC), analytical grade

Apparatus

Negative air fume hood

Laboratory analytical balance, readable to 1 mg

Pipets, volumetric 1, 2, 5, 10, and 25 ml, Corning No. 7100, or equivalent

Flask, volumetric, 100 ml

Bottle, 2 oz., glass, narrow-neck with Teflon-lined septum and screw top

Shaker, wrist-action, Burrel Model 30 or equivalent

Instrument:	Perkin-Elmer 8500 Gas Chromatograph (or equivalent) with data handling system (Chrom 3)
Detector:	Flame Ionization
Column:	30 m x 0.53 mm DB1701 Megabore film thickness 1.0 um; J& W Scientific, S/N 9518014 inserted in packed column injector for on-column injection
Autosampler:	Perkin-Elmer AS 8300
Temperature:	
Injection port	250^{o}C
Detector	250^{o}C

Column*	40°C, 4 min. initial hold.6°C/min. to 136°C: ramp to 240°C at 20°C/min.; hold 5 min.
Helium flow:	Approx. 15 ml/min
Attenuation:	4 (high sensitivity setting)
Report run time:	20 minutes

*Similar results have been obtained with a glass packed column (10% SP1000/100/120 Suplecoport, 10'x 1/4" x 2 mm I.D.) using a column flow of 28 ml/min and temperature programming of 65-150°C/min; injection volume-2ul

Procedure

Caution: The standard solution should be prepared every two weeks and be refrigerated when not in use.

Internal Standard Solution (ISS)

Into a 100 ml volumetric flask, transfer 2.000g of chlorobenzene weighed to the nearest mg. Prepare solution A by diluting to volume with DCM. Mix well.

Using a volumetric pipet, transfer 2 ml of solution A into a 100 ml volumetric flask. Dilute to the mark with DCM and mix well. This is solution B.

Standard

Into a 100 ml volumetric flask transfer 1.500 g of 1,2-dichloroethane weighed to the nearest 0.1 mg. Dilute to the mark with DCM and mix well.

Prepare standards for calibration by transferring 1, 2, 5,10,and 25 ml of the above solution into respective 10 ml volumetric flasks, using volumetric pipets.

Transfer 2 ml of ISS solution A into each flask, using a volumetric pipet. Dilute to the mark with DCM and mix well.

Sample

Into a 2 oz. glass, narrow-neck bottle, transfer the appropriate amount of sample, each weighed to the nearest mg, using the table below:

Test Material	Approximate Weight
Base 250	10.00 grams (7ml)
EPR-2	33.00 grams (30 ml)
EPR-4	18.00 (ml)

Dilute each sample by bringing the total weight of sample and solvent to 40.00 grams by dilution with deionized water.

Using a volumetric pipet, transfer 10 ml of ISS solution B to each sample.

Secure each sample bottle with a Teflon-lined septum and open top screw cap.

Shake for 15 min on a wrist-action horizontal shaker. Invert each bottle, and wait until two separate layers are obtained.

DETERMINATION

Inject 1 ul of the lower DCM layer from each standard and sample into the GC. Obtain a chromatogram and calculate using the formula given below.

CALCULATIONS

1. Standard concentration or Internal Standard concentration

$$\frac{\text{Wt of component(g)}}{\text{Volume(ml)}} \times 10^6 = \text{ppm/ml (wt/volume)}$$

2. Standard Relative Response Factor (R.R.F.)

$$\frac{\text{Standard conc.(ppm/ml)}}{\text{Area of component}} \times \frac{\text{Area of Int. Std.}}{\text{ppm Int. Std. conc.(ppm/vol)}}$$

3. Sample Concentration ppm (Wt/Wt)

$$\text{R.R.F.} \times \frac{\text{Area of Component}}{\text{Area of Int. Std.}} \times \frac{\text{Conc. of Std. (ppm/ml)}}{\text{Sample Wt. (g)}} \times \text{Dilution Volume(ml)}$$

Experimental Data

Component	Retention Time (minutes)	Relative Response Factor (R.R.F.)
1,2-Dichloroethane	3.75	2.574
Chlorobenzene	9 08	1.000

Recovery Determination

1. Into a 2oz. glass bottle, weigh the appropriate amount of a test sample of known concentration as outlined in Procedure
2. To each test sample, pipette 10 ml of internal standard solution(B)
3. Pipette 2 ml of stock solution to one test sample and 4 ml of stock standard solution to the other test preparation.
4. Secure each sample bottle with a Teflon-lined septum and screw top and prepare as with other test samples.

Recovery Data

Recovery for 1,2-dichloroethane averaged 98% using either water or Base 250 to extrct known standards in DCM. The method has excellent linearity within the range of standards.