FAO SPECIFICATIONS

FOR PLANT PROTECTION PRODUCTS

METHIOCARB (AGP:CP/321)



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.

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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)

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

<u>FAO Provisional Specifications [Code 'S/P', formerly ('S')]</u> 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 10³ m²/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.

METHIOCARB

4-methylthio-3,5-xylyl methylcarbamate



INFORMATION

COMMON NAME: methiocarb (ISO)

STRUCTURAL FORMULA:

EMPIRICAL FORMULA: $C_{11}H_{15}NO_2S$

RMM: 225.3

CAS REGISTRY NUMBER: 2032-65-7

CIPAC CODE NUMBER: 165

CHEMICAL NAMES:

4-methylthio-3,5-xylyl methylcarbamate (IUPAC)

3,5-dimethyl-4-(methylthio)phenyl methylcarbamate (CA)

METHIOCARB TECHNICAL

FAO Specification 165/TC/S/F (1991)

1. DESCRIPTION.

The material shall consist of methiocarb together with related manufacturing impurities and shall be a white to yellowish solid free from visible extraneous matter and added modifying agents.

2. ACTIVE INGREDIENT

2.1 <u>Identity tests</u> (165/TC/M/2, CIPAC D, p.130)

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

2.2 <u>Methiocarb</u> (165/TC/M/3, CIPAC D, p.130)

The methiocarb content shall be declared (not less than 970 g/kg) and, when determined, the content obtained shall not differ from that declared by more than ± 20 g/kg.

3. IMPURITIES

3.1 <u>Water</u> (MT 30.1, CIPAC F, p.91)

Maximum: 2 g/kg.

4. PHYSICAL PROPERTIES

4.1 <u>Acidity</u> (MT 31.1, CIPAC F, p.96)

Maximum acidity: 2 g/kg calculated as H₂SO₄.

METHIOCARB TECHNICAL CONCENTRATES

FAO Specification 165/TK/S/F (1991)

1. DESCRIPTION

The material shall consist of methiocarb, complying with the requirements of FAO specification 165/TC/S/F (1991), together with related manufacturing impurities and shall be a white to yellowish powder free from visible extraneous matter and added modifying agents except the stabilizer.

2. ACTIVE INGREDIENT

2.1 Identity tests (165/TC/M/2, CIPAC D, p.130)

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

2.2 <u>Methiocarb</u> (165/TC/M/3, CIPAC D, p.130)

The methiocarb content shall be declared (not less than 800 g/kg) and, when determined, the content obtained shall not differ from that declared by more than ± 25 g/kg.

3. IMPURITIES

3.1 <u>Water</u> (MT 30.1, CIPAC F, p.91)

Maximum: 20 g/kg.

3.2 Material insoluble in acetone (MT 27, CIPAC F, p.88)

Maximum: 180 g/kg.

4. PHYSICAL PROPERTIES

4.1 Dry sieve test (MT 59.1, CIPAC F, p.177)

Maximum: 5% retained on a 75 μ m test sieve. Not more than (0.005 x X)% of the amount of sample used for the determination shall be present as methiocarb in the residue on the sieve, where X is the methiocarb content (g/kg) found under 2.2.

METHIOCARB WETTABLE POWDERS

FAO Specification 165/WP/S/F (1991)

1. DESCRIPTION

The material shall consist of a homogeneous mixture of technical methiocarb, complying with the requirements of FAO specification 165/TC/S/F or 165/TK/S/F (1991), together with filler(s) and any other necessary formulants. It shall be in the form of a fine powder, free from visible extraneous matter and hard lumps.

2. ACTIVE INGREDIENT

2.1 Identity tests (165/WP/M/2, CIPAC D, p.132)

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

2.2 Methiocarb (165/WP/M/3, CIPAC D, p.132)

The methiocarb 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

250 up to 500 g/kg \pm 5% of the declared content

Above 500 g/kg \pm 25 g/kg

3. IMPURITIES

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

Maximum: 25 g/kg.

4. PHYSICAL PROPERTIES

4.1 <u>pH range</u> (MT 75, CIPAC F, p.205)

pH range: 7.0 to 9.0.

4.2 Wet sieve test (MT 59.3, CIPAC F, p.179)

Maximum: 2% retained on a 75 µm test sieve.

4.3 Suspensibility (MT 15.1, CIPAC F, p.45. Notes 1 and 2)

A minimum of 50% of the methiocarb content found under 2.2 shall be in suspension after 30 min in CIPAC Standard Water C at 25 °C (Notes 3 and 4).

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

4.4 Persistent foam (MT 47, CIPAC F, p.152. Note 5)

Maximum: 10 ml after 1 min.

4.5 Wetting of the product without swirling (MT 53.3.1, CIPAC F, p.165)

The product shall be completely wetted in 2 min without swirling.

5. STORAGE STABILITY

5.1 <u>Stability at 54 °C</u> (MT 46.1.1, CIPAC F, p.149)

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 6) and the product shall continue to comply with 4.1, 4.2 and 4.3.

NOTES

- 1. This test will normally be carried out only after the heat stability test 5.1.
- 2. Chemical assay is the only fully reliable method of measuring the mass of active ingredient still in suspension. However, simpler methods such as gravimetric and solvent extraction determination may be used on a routine basis provided that these methods have been shown to give equal results to those of the chemical assay method. In case of dispute the chemical method shall be the 'Referee method'.
- 3. *Unless another temperature is specified.*
- 4. The product should be tested at the highest and lowest rates of use recommended by the supplier, provided this is consistent with the conditions given in the method MT 15.1, CIPAC F, p.45.
- 5. The mass of sample to be used in the test should correspond to the highest rate of use recommended by the supplier.
- 6. Samples of the product taken before and after the storage stability test should be analysed together after the test to reduce the analytical error.

METHIOCARB GRANULES

(For application by mechanical equipment)

FAO Specification 165/GR/S/F (1991)

1. **DESCRIPTION**

The material shall consist of granules containing technical methiocarb, complying with the requirements of FAO Specification 165/TC/S/F or 165/TK/S/F (1991), together with suitable carriers and any other necessary formulants. It shall be dry, free from visible extraneous matter and hard lumps, free-flowing, essentially non-dusty and intended for application by machine.

2. ACTIVE INGREDIENT

2.1 <u>Identity tests</u> (165/TC/M/2, CIPAC D, p.130)

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

2.2 <u>Methiocarb</u> (165/TC/M/3, CIPAC D, p.130)

The methiocarb content shall be declared (g/kg) 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>

Up to 25 g/kg \pm 25% of the declared content Above 25 up to 100 g/kg \pm 10% of the declared content

3. IMPURITIES

3.1 Water (MT 30.1, CIPAC F, p.91, but after extraction of water with methanol)

Maximum: 150 g/kg.

4. PHYSICAL PROPERTIES

4.1 <u>pH range</u> (MT 75, CIPAC F, p.205)

pH range: 4.5 to 9.0.

4.2 Apparent density after compaction (MT 58.3, CIPAC F, p.175)

The apparent density range of the product after compaction shall be declared.

4.3 Nominal size range (MT 58.2, CIPAC F, p.173)

The nominal size range of the product shall be declared. The ratio of the lower to the upper limit shall not normally exceed 1:4 (Note 1). Not less than 85% of the product shall be within the declared nominal size range.

4.4 Sieve analysis (MT 58.2, CIPAC F, p.173. Note 2)

4.4.1 For lower limit of nominal size range $\geq 300 \,\mu m$

Material retained on a 125 µm test sieve, minimum: 980 g/kg.

The methiocarb content of the material retained on the sieve shall not be less than 95% of that determined under 2.2.

4.4.2 For lower limit of nominal size range < 300 µm

Material passing a 63 µm test sieve, maximum: 5 g/kg.

The methiocarb content of the material passing the sieve shall not be more than the content found under 2.2.

5. STORAGE STABILITY

5.1 <u>Stability at 54 °C</u> (MT 46.1.1, CIPAC F, p.149)

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 3) and the product shall continue to comply with 4.1, 4.3 and 4.4.

NOTES

- 1. Higher ratios may increase the risk of segregation which may adversely affect the flow rate. This should be checked with the machine to be used. The purchaser should check that the nominal size range is suitable for his requirements, since the size range may affect the biological activity.
- 2. *Not applicable to extruded granules.*
- 3. Samples of the product taken before and after the storage stability test should be analysed together after the test to reduce the analytical error.

METHIOCARB SUSPENSION CONCENTRATES FOR SEED TREATMENT

FAO Specification 165/FS/S/F (1991)

1. **DESCRIPTION**

The material shall consist of a suspension of fine particles of technical methiocarb, complying with the requirements of FAO specification 165/TC/S/F or 165 (1991)/TK/S/F (1991), in an aqueous phase together with suitable formulants including colouring matter (Note 1) for seed treatment. After gentle stirring or shaking of the commercial container, the material shall be homogeneous (Note 2) and suitable for dilution with water if necessary.

2. ACTIVE INGREDIENT

2.1 <u>Identity tests</u> (165/TC/M/2, CIPAC D, p.130)

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

2.2 <u>Methiocarb</u> (165/SC/M/3, CIPAC D, p.133)

The methiocarb content shall be declared (g/kg or g/l at 20 °C, Note 3) 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>

250 up to 500 g/kg or g/l \pm 5% of the declared content

Above 500 g/kg or g/l \pm 25 g/kg or g/l

3. PHYSICAL PROPERTIES

3.1 Mass per millilitre at 20 °C (MT 3.3, CIPAC F, p.18)

If required, the range of the mass per millilitre (g/ml) at 20 °C shall be declared (Note 4).

3.2 <u>pH range</u> (MT 75, CIPAC F, p.205, except that the pH is measured on the undiluted sample)

pH range: 2.5 to 4.5.

3.3 Pourability (MT 148, CIPAC F, p.348)

Maximum 'rinsed residue': 0.25%.

3.4 Wet sieve test (method under consideration. Note 5)

Maximum: 0.5% retained on a 75 µm test sieve.

3.5 <u>Persistent foam</u> (MT 47.2, CIPAC F, p.152. The determination is carried out using the undiluted sample. Note 6).

Maximum: 10 ml after 1 min.

3.6 Flash point (MT 12, CIPAC F, p.31. Note 7)

If required, the flash point of the product shall not be lower than the minimum declared flash point. The procedure used shall be stated.

4. STABILITY

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

After storage at 0 ± 1 °C for 7 days, the product shall continue to comply with 3.4.

4.2 <u>Stability at 54 °C</u> (MT 46.1.1, CIPAC F, p.149)

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, 3.3 and 3.4.

NOTES

- 1. Where applicable, the product shall contain a dye that permanently colours the seed after treatment (red is recommended) and cannot be removed by washing with water. In some countries, there may be a legal requirement that a specific colour shall be used. The same colour should not be used for denaturing seed to be used as livestock feeding-stuffs.
- 2. Before sampling to verify the product quality, inspect the commercial container carefully. On standing, suspension concentrates usually develop a concentration gradient from the top to the bottom of the container. This may even result in the appearance of a clear liquid on the top and/or of sediment on the bottom. Therefore, before sampling, homogenize the product according to the instructions given by the manufacturer or, in the absence of such instructions, gently shake the commercial container (for example by inverting the closed container several times; large containers must be opened and stirred adequately). After this procedure, the container should not contain a sticky layer of non-dispersed product at the bottom. A suitable and simple method of checking for a non-dispersed sticky layer ('cake') is by probing with a glass rod or similar device adapted to the size and shape of the container.

All the physical and chemical tests must be carried out on a laboratory sample taken after the recommended homogenization procedure.

3. If the buyer requires both g/kg and g/l at 20 °C, then in case of dispute the analytical result shall be calculated in g/kg.

- 4. Unless homogenization is carried out carefully, it is possible for the sample to become aerated. This can lead to errors in the determination of the density and in calculation of the active ingredient content (in g/l) if methods other than MT 3.3 are used.
- 5. The test should be carried out at the application concentration.
- 6. The sample is taken in undiluted form because it is applied without dilution.
- 7. Attention is drawn to the appropriate national and international regulations on the handling and transport of flammable materials.
- 8. Samples of the product taken before and after the storage stability test should be analysed together after the test to reduce the analytical error.