

FAO SPECIFICATIONS FOR PLANT PROTECTION PRODUCTS

AGP:CP/346

CHLORIDAZON

5-amino-4-chloro-2-phenylpyridazin-3(2H)-one



FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

Rome, 1997

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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 warrants 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.

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. 128, Rome 1995 (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 $1 \times 10^3 \text{ m}^2/\text{s}$ 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.

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.

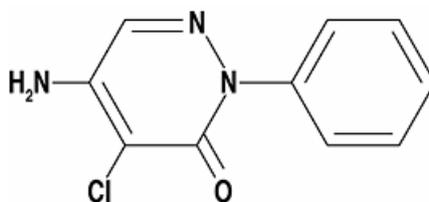
General guidelines on preparing draft specifications are given in Plant Production and Protection Paper 128, *Manual on the Development and Use of FAO Specifications for Plant Protection Products, Fourth Edition*, FAO, Rome, 1995 (available in English only).

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, Application Standards and Prior Informed Consent. The drafts are converted into FAO Provisional Specifications, or full FAO Specifications.

INFORMATION

COMMON NAME: chloridazon (ISO)

STRUCTURAL FORMULA:



EMPIRICAL FORMULA: C₁₀ H₈ Cl N₃ O

RMM: 221.6

CAS REGISTRY NUMBER: 1698-60-8

CIPAC CODE NUMBER: 111

CHEMICAL NAMES:

5-amino-4-chloro-2-phenylpyridazin-3(2H)-one (IUPAC)

5-amino-4-chloro-2-phenyl-3(2H)-pyridazone (CAS)

CHLORIDAZON TECHNICAL

FAO Specification 111/TC/S/F (1997)

1. DESCRIPTION

The material shall consist of chloridazon (Note 1) together with related manufacturing impurities and shall be a yellow to brown solid free from visible extraneous matter and added modifying agents (Note 2).

2. ACTIVE INGREDIENT

2.1 Identity tests

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

2.1.1 IR spectrum (111/TC/M/2.1, CIPAC D, p. 31).

2.1.2 HPLC (111/TC/M/2.2, CIPAC D, p. 35).

2.2 Chloridazon (111/TC/M/3, CIPAC D, p. 35)

The chloridazon content shall be declared (minimum 910 g/kg) and, when determined, the content obtained shall not differ from that declared by more than ± 25 g/kg.

3. IMPURITIES

3.1 4-Amino-5-chloro-isomer (111/TC/M/3, CIPAC D, p. 35)

Maximum: 60 g/kg.

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

Maximum: 20 g/kg.

4. PHYSICAL PROPERTIES

4.1 Acidity, alkalinity (MT 31, CIPAC F, p. 96)

Maximum acidity: 1 g/kg calculated as H₂SO₄.

Maximum alkalinity: 1 g/kg calculated as NaOH.

NOTES

1. *The spelling in French is chloridazone.
In Canada, Denmark, Poland, USA a former proposal pyrazon has become effective. PAC is the common name in Japan.*
2. *If additives are present, then they shall be declared in g/kg in general terms. If they interfere with the methods of analysis for the active ingredient or give rise to hazards in processing etc., then their composition and suitable methods of analysis shall be supplied.*

CHLORIDAZON TECHNICAL CONCENTRATES

FAO Provisional Specification 111/TK/S/F (1997)

1. DESCRIPTION

The material shall consist of chloridazon, complying with the requirements of FAO specification 111/TC/S/F (1997), together with related manufacturing impurities and shall be a solid of yellow to brown colour free from visible extraneous matter and added modifying agents except for the carrier and auxiliaries needed for the final formulation (Note 1).

2. ACTIVE INGREDIENT

2.1 Identity tests

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

2.1.1 IR spectrum (111/TC/M/2.1, CIPAC D, p. 31).

2.1.2 HPLC method (111/TC/M/2.2, CIPAC D, p. 35).

2.2 Chloridazon (cf. 111/WP/M/3, CIPAC D, p. 35)

The chloridazon content shall be declared and, when determined, the content shall not differ from that declared by more than the following amounts:

<u>Declared content</u>	<u>Permitted tolerance</u>
above 500 g/kg	± 25 g/kg

3. IMPURITIES

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

Maximum: 50 g/kg.

4. PHYSICAL PROPERTIES

4.1 Acidity, alkalinity (MT 31, CIPAC F, p. 96)

Maximum acidity: 50 g/kg calculated as H₂SO₄.

Maximum alkalinity: 50 g/kg calculated as NaOH.

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

pH range: 5.0 to 10.0.

5. STORAGE STABILITY

5.1 Stability at 54°C (MT 46.1, CIPAC F, p. 149)

After storage at $54 \pm 2^\circ\text{C}$ for 14 days, the determined average active ingredient content must not be lower than 97% relative to the determined average content before storage (Note 2) and the product shall continue to comply with 4.1 and 4.2.

NOTES

1. *If additives are present, then they shall be declared in g/kg in general terms. If they interfere with the methods of analysis for the active ingredient or give rise to hazards in processing etc., then their composition and suitable methods of analysis shall be supplied.*
2. *Analysis of the product before and after storage stability test should be carried out at the same time (i.e. after storage) to reduce analytical error.*

CHLORIDAZON WETTABLE POWDERS

FAO Provisional Specification 111/WP/S/F (1997)

1. DESCRIPTION

The material shall consist of a homogeneous mixture of technical chloridazon, complying with the requirements of FAO specification 111/TC/S/F (1997), together with the filler(s) and 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 test (111/WP/M/2, CIPAC D, p. 37)

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

2.2 Chloridazon (111/WP/M/3, CIPAC D, p. 37)

The chloridazon 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>
above 100 up to 250 g/kg	± 6 % of declared content
above 250 up to 500 g/kg	± 5 % of declared content
above 500 g/kg	± 25 g/kg

3. IMPURITIES

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

Maximum: 50 g/kg.

4. PHYSICAL PROPERTIES

4.1 pH range (MT 75.2, CIPAC F, p. 206)

pH range: 7.0 to 10.0.

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

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

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

A minimum of 70 % of the chloridazon content found under 2.2 shall be in suspension (Note 3) after 30 minutes in CIPAC Standard Water D at 30°C ± 2°C (Notes 4 and 5).

4.4 Persistent foam (MT 47.1, CIPAC F, p. 152)

Maximum: 20 ml after 1 minute (Note 6).

4.5 Wetting of the product (MT 53.3.1, CIPAC F, p. 164)

It shall be completely wetted in 3 minutes without swirling (Note 7).

5. STORAGE STABILITY

5.1 Stability at 54°C (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 before storage (Note 8) and the product shall continue to comply with 4.1, 4.2, 4.3 and 4.5.

NOTES

1. *The product should be tested at the highest and lowest rates of use recommended by the supplier, provided this does not exceed the conditions given in Method MT 15.1, CIPAC F, p. 45.*
2. *This test will normally only be carried out after the heat stability test 5.1.*
3. *Chemical assay is the only fully reliable method to measure 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 the methods have been shown to give results equal to those of the chemical assay. In case of dispute the chemical method shall be the "Referee method".*
4. *Alternatively, if the buyer requires other CIPAC Standard Waters to be used, then this shall be specified when ordering.*
5. *Unless another temperature is specified.*

6. *The mass of sample to be used in the test should be at the highest rate of use recommended by the supplier.*
7. *The properties of chloridazon and the low bulk weight of chloridazon WPs may cause wettabilities that exceed the 1 min. time frame usual for WPs.*
8. *Analysis of the product before and after storage stability test should be carried out at the same time (i.e. after storage) to reduce analytical error.*

CHLORIDAZON WATER DISPERSIBLE GRANULES

FAO Provisional Specification 111/WG/S/F (1997)

1. DESCRIPTION

The material shall consist of a homogeneous mixture of technical chloridazon, complying with the requirements of FAO specification 111/TC/S/F (1997), together with carriers and any other necessary formulants. It shall be in the form of granules (Note 1) for application after disintegration and dispersion in water. The product shall be dry, free flowing and free from visible extraneous matter and hard lumps.

2. ACTIVE INGREDIENT

2.1 Identity test (111/WP/M/2, CIPAC D, p. 37)

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

2.2 Chloridazon (111/WP/M/3, CIPAC D, p. 37)(Notes 2 and 3)

The chloridazon content shall be declared (g/kg) and, when determined (Note 4), the content shall not differ from that declared by more than the following amounts:

<u>Declared content</u>	<u>Permitted tolerance</u>
above 250 up to 500 g/kg	$\pm 5\%$ of declared content
above 500 g/kg	± 25 g/kg

3. IMPURITIES

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

Maximum: 50 g/kg.

4. PHYSICAL PROPERTIES

4.1 pH range (MT 75.2, CIPAC F, p. 206)

pH range: 7.0 to 10.0.

4.2 Wetting of the material (MT 53.3.1, CIPAC F, p. 164)

It shall be completely wetted in 30 sec. without swirling (Note 3).

4.3 Wet sieve test (MT 167, CIPAC F, p. 416)

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

4.4 Suspensibility (MT 168, CIPAC F, p. 41)(Notes 5 and 6)

A minimum of 75% of the chloridazon content found under 2.2 shall be in suspension after 30 min. in CIPAC Standard Water D at 30°C ± 2°C (Notes 7 and 8).

4.5 Degree of dispersion (MT 174, CIPAC F, p. 435)

Minimum: 80% .

4.6 Persistent foam (MT 47.1, CIPAC F, p. 152)(Note 9)

Maximum: 20 ml after 1 min.

4.7 Dustiness (MT 17 1.1, CIPAC F, p. 425)(Note 10)

Maximum: 12 mg collected dust.

4.8 Flowability (MT 172, CIPAC, p. 430)

At least 99 % of the product shall pass through a 5 mm test sieve after 20 liftings of the sieve.

5. STORAGE STABILITY

5.1 Stability at 54°C (MT 46.1.1, CIPAC F, p. 149)(Note 11)

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 before storage (Note 12) and the product shall continue to comply with 4.1, 4.3, 4.4, 4.5, 4.7 and 4.8.

NOTES

1. *Depending on manufacturing conditions WG may have different forms and particle size ranges. To describe specific products it is recommended to add information about the form (e.g. irregular shape, nearly spherical, cylindrical) and the nominal size range.*

2. *If the upper limit was exceeded due to excessive desiccation, the material may be acceptable after moisture equilibration under appropriate storage conditions.*
3. *The method has been developed for water dispersible powder. It was proven suitable for water dispersible granules as well.*
4. *If additives interfere with the CIPAC method for chloridazon WPs, then the composition of the additives and suitable methods for chloridazon analysis shall be supplied.*
5. *Chemical assay is the only fully reliable method to measure 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 results equal to those of the chemical assay. In case of dispute the chemical method shall be the "Referee method".*
6. *The product should be tested at the highest and lowest use concentration recommended by the supplier, provided this does not exceed the conditions given in method MT 168.*
7. *Alternatively, if the buyers require other CIPAC Standard Waters to be used, then this shall be specified when ordering.*
8. *Unless another temperature is specified.*
9. *The mass of sample to be used in the test should be specified at the highest rate of use recommended by the supplier.*
10. *The optical method (MT 171.2) usually shows good correlation to the gravimetric method (MT 171.1) and can therefore be used as an alternative where the equipment is available. Where the correlation is in doubt, it must be checked with the product to be tested. In case of dispute, the gravimetric method shall be used. The dustiness of a chloridazon WG should not be more than 12 mg collected dust with the gravimetric method. This corresponds to a dust value of approximately 10 resulting from the optical method.*
11. *CIPAC MT 46.1.1 was meant originally for water dispersible powders. It was proven suitable for water dispersible granules, as well. Excessive desiccation can impair the active ingredient content range and the properties of the product. It may be necessary to seal the beaker in metal foil.*
12. *Analysis of the product before and after storage stability test should be carried out at the same time (i.e. after storage) to reduce analytical error.*

CHLORIDAZON SUSPENSION CONCENTRATES

FAO Provisional Specification 111/SC/S/F (1997)

1. DESCRIPTION

The material shall consist of a suspension of fine particles of technical chloridazon, complying with the requirements of the FAO specification 111/TC/S/F (1997) in an aqueous phase together with suitable formulants. After gentle agitation the material shall be homogeneous (Note 1) and suitable for further dilution in water.

2. ACTIVE INGREDIENT

2.1 Identity test (111/SC/M/2, CIPAC D, p. 38)

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

2.2 Chloridazon (111/SC/M/3, CIPAC D, p. 38)

The chloridazon 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:

<u>Declared content</u>	<u>Permitted tolerance</u>
above 100 up to 250 g/l	± 6 % of the declared content
above 250 up to 500 g/l	± 5 % of the declared content
above 500 g/l	± 25 g/l

3. IMPURITIES

Not relevant

4. PROPERTIES

4.1 Mass per millilitre at 20°C (MT 3.3.2, CIPAC F, p.19)(Note 3)

The range of the mass per millilitre (g/ml) at 20°C shall be declared. Usually 1.1 to 1.3 g/ml.

4.2 pH range (MT 75.2, CIPAC F, p. 206)

pH range: 5.0 to 9.0.

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

Maximum "residue": 8.0%.

Maximum "rinsed residue": 0.6%.

4.4 Spontaneity of dispersion (MT 160, CIPAC F, p. 391)

A minimum of 75 % of the chloridazon content found under 2.2 shall be in suspension after 5 minutes in CIPAC Standard Water D at $25^{\circ}\text{C} \pm 2^{\circ}\text{C}$

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

4.5 Suspensibility (MT 161, CIPAC F, p. 394) (Notes 4 and 5)

A minimum of 75 % of the chloridazon content found under 2.2 shall be in suspension after 30 minutes in CIPAC Standard Water D at $30^{\circ}\text{C} \pm 2^{\circ}\text{C}$ (Note 6).

Alternatively, if the buyers require other CIPAC Standard Waters or temperatures to be used, then this shall be specified when ordering.

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

Maximum: 0.2 % of the product shall be retained on a 75 μm test sieve.

4.7 Persistent foam (MT 47.2. CIPAC F, p. 152) (Note 6)

Maximum: 20 ml after 1 minute.

5. STORAGE STABILITY

5.1 Stability at 0 °C (MT 39.1, CIPAC F, p. 128) (Note 7)

After storage at $0 \pm 1^{\circ}\text{C}$ for 7 days, the product shall continue to comply with 4.4, 4.5 and 4.6.

5.2 Stability at 54°C (MT 46.1 CIPAC F, p. 951)

After storage at $54 \pm 2^\circ\text{C}$ for 14 days, the determined average active ingredient content must not be lower than 97 % relative to the determined average content before storage (Note 8) and the product shall continue to comply with 4.3, 4.4, 4.5, 4.6 and, if required, with 4.2.

NOTES

- 1. 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, by gentle shaking of 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 any 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.*
- 2. 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 the calculation of the active ingredient content in g/l, if methods other than MT 3.3, CIPAC F, p. 19, are used. 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.*
- 3. Instead of using the delicate dilution method MT 3.3.2 for density determination direct measurement using the air comparison pycnometer is mostly preferable. With this alternate method air bubbles will not cause misleading results.*
- 4. This test will normally only be carried out after the heat stability test 5.2.*
- 5. Chemical assay is the only fully reliable method to measure 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 results equal to those of the chemical assay. In case of dispute the chemical method shall be the "Referee method".*
- 6. The amount of sample to be used in the test should be specified at the highest rate of use recommended by the supplier.*

7. *The method has been developed for clear EC and SL formulations. For SCs the volume of visible deposit at the bottom and/or serum at the top of the cone shaped centrifuge tube shall be recorded omitting centrifugation.*
8. *Analysis of the product before and after storage stability test should be carried out at the same time (i.e. after storage) to reduce analytical error.*