



**Australian Pesticides &
Veterinary Medicines Authority**

**Guidelines for the Generation of Storage Stability Data of
Agricultural Chemical Products**

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INTRODUCTION

Agricultural chemical products can undergo chemical and physical changes on storage. The rate at which these changes occur depends on the nature of the active constituent(s), the formulation type, the packaging and, notably, the storage conditions (temperature, light and humidity). The product remains fit for use as long as these changes have no adverse effects on application, biological performance, and the safety of operators, consumers and the environment.

(i) Outline of the data requirement

Storage stability data must be generated with the product stored in the proposed commercial packaging (or smaller packages of the same construction and material) under accelerated conditions and/or long term testing at room temperature or under ambient warehouse conditions. However, it is recommended that long term testing be conducted at 30°C, which reflects the Australian field conditions. After having carried out these storage stability studies, the following information must be supplied to the APVMA:

- Shelf life specifications with proposed limits for the active constituent content and physical characteristics of the product within which the properties of the product will remain during its shelf life.
- Actual test results with full details of the methods used for each of the test parameters listed in the shelf life specifications.
- Stability of the packaging materials showing no significant deterioration, as interaction with the product can cause embrittlement, softening, and corrosion.

The data should support the claim that the formulated product will remain within specification for at least 2 years, when stored in its unopened original container, away from direct sunlight, at or above 25°C. This should include a statement to the effect that the packaging is unaffected during the proposed shelf life.

A shorter shelf life may be approved where the data do not support a 2 year shelf life (Date-Controlled Agricultural Chemical Products).

(ii) Shelf life statement on the product label

It is a normal convention that information on storage stability (expiry date) is given on the product label only if the product cannot be stored for at least 2 years at or above 25°C. The printed labels must include the date (month and year) of manufacture of the batch and relevant information on the conditions under which the product should be stored.

If the formulation has a shelf life of less than 2 years, the expiry date must also be included on the product label e.g., 'use by date' for date controlled products. A list of date-controlled products is provided in Schedule 1 of the Agricultural and Veterinary Chemicals (Agvet) Code Regulations.

(iii) Stability guidelines

The purpose of this guideline is to provide a comprehensive and updated guide to the conduct of stability testing. A comprehensive list of pesticide specifications has recently been published by the FAO (Ref 1). The APVMA guideline has been constructed to closely follow the *FAO Pesticide Specifications*.

This guideline is divided into the following sections:

- Section 1. - Design of stability trials. Provides advice on the design of stability trial protocols and the details that should be provided in any report of stability testing.
- Section 2. - Technical characteristics of formulated products. Defines the technical characteristics of various formulation types and provides advice on the analytical methods that may be used
- Section 3. - Parameters to be tested in stability trials. Provides a consolidated list of recommended test parameters for different formulation types
- Section 4. - Definitions
- Section 5. - References

SECTION 1. DESIGN OF STABILITY TESTING TRIALS

1.1 Size and number of batches to be tested

Stability testing must be conducted on laboratory/pilot/production scale batches of the product (stability data generated on batch size of less than 5 kilograms or 5 litres are normally not acceptable). The formulation must be the same as that proposed for registration in Australia. The size of the batches produced for stability testing, batch identity and date of manufacture should be recorded and reported with the stability data.

1.2 Storage conditions and duration

Storage stability programmes may include accelerated and/or real-time tests. For some formulations, studies at lower temperatures may be necessary due to the instability of the formulation at higher temperatures (this should be reflected in the recommended storage conditions). Liquid formulations should also be tested at low temperatures ($0 \pm 2^{\circ}\text{C}$ or lower).

1.3 Accelerated testing

Stability tests at elevated temperatures are designed to increase the rate of chemical degradation or physical change of a product. Accelerated testing is performed at elevated temperatures in an attempt to obtain information on the shelf life of a product in a relatively short time. Accelerated testing involves extrapolations from higher to lower temperatures and from shorter to longer storage periods.

The FAO Pesticide Specifications recommend testing of the relevant product parameters before and after storage at 54°C for 14 days. However, some preparations may not be stable at 54°C for 14 days and the following alternative times/temperatures may be used.

Table 1. Storage Temperature's and Duration of Stability Trials

Temperature	Duration of stability trial
54°C	14 days
45°C	6 weeks
40°C	8 weeks

Products that exhibit an adequate stability profile under any of the above conditions are likely to be stable for 2 years under normal (at or above 25°C) storage conditions.

1.4 Real-time testing

The real time testing is normally performed at or above 25°C for at least 2 years. Depending on the formulation type and packaging material, testing under standardised relative humidity and light exposure conditions may also be required.

1.5 Cold stability testing

Liquid formulations (capsule suspensions, emulsifiable concentrates, oil-in-water emulsions, micro-emulsions, soluble concentrates, suspension concentrates) may be adversely affected by storage at low temperature. Storage at low temperature may result in crystallisation of active constituent(s), significant changes in viscosity or phase separation of emulsions.

In some places in Australia, night temperatures regularly approach 0°C or lower. Therefore, the liquid formulations should also be tested at $0 \pm 2^\circ\text{C}$ or lower for 7 days.

The effect of low temperatures on stability must be determined and reported according to CIPAC method MT 39.3 (liquid formulations).

Note: Stability data generated at low temperatures is not required if the product label contains a warning against exposure to low temperatures.

1.6 Testing intervals

Samples should be tested as soon as practicable following manufacture (time zero) and then at the conclusion of the storage period for accelerated testing, and every six months for real-time testing. The dates of product testing should be recorded and reported with the stability results.

1.7 Test parameters

The stability profile of an agricultural chemical product is determined by monitoring a combination of chemical and physical properties during storage. Monitoring active constituent content alone is insufficient to make any reliable prediction about the stability of the product. On prolonged storage, a product may exhibit negligible decline in active constituent, yet the important physical properties (eg. wettability, suspensibility etc) may have changed to such an extent that the performance of the product can be compromised.

Relevant test parameters for each formulation type are given in Section 3. It is expected that all relevant parameters will be addressed in a stability trial. If certain parameters are not addressed, then relevant scientific argument must be provided as to why testing for certain parameters should not be required. Note that the relevant test parameters have been derived from the FAO/WHO Pesticide Specifications (Ref. 1).

1.8 Containers

The effect of the formulation on the primary pack and vice versa is important and, therefore, the product should be packaged in the same containers (materials and pack size) that are proposed for the marketing of the final product. If the product is to be marketed only in containers in which stability testing would be impractical (eg too large), then stability trials in smaller containers of the same materials and construction may be used to extrapolate to the larger containers.

Containers should be examined to ensure that no significant interaction with the formulation (affecting the stability/integrity of the packaging material) has taken place during storage.

1.9 Product in water soluble bags

It is considered that packaging of products in water soluble bags/sachets may have an effect on the physical characteristics of the product. Therefore, where the product is packaged in water soluble bags, the relevant physical tests must be carried out in the presence of the soluble bag material in the same ratio as will occur in the spray tank or other application equipment.

In addition, the dissolution rate of water soluble bags must be carried out using CIPAC method MT 176. The dissolution time should be reported.

Examinations for leakage and/or effects of the formulation on water soluble bags should be carried out.

Where multiple bags are to be packaged in a single container, evidence is required that the integrity of the water soluble packaging is not affected either by the opening and resealing of the outer pack or by moisture entering through routine use. This may be achieved by storing a 'multi-bag' pack at 25°C over a 6-month period and periodically removing the water soluble bags, until all bags have been removed. The integrity of the water soluble bags must be examined on removal and the 'dissolution characteristics of the water soluble bags tested using CIPAC method MT 176'.

Note: If the product is to be packaged in a water soluble pack, the packaging material is treated as part of the formulation composition. Therefore, where the product is currently registered in some other packaging, the change to water soluble packaging would require additional stability data for the product packaged in water soluble bags.

1.10 Analytical methods

Full details of the analytical methods used to monitor the product during stability trials must be provided, except where collaboratively tested standard methods (CIPAC, AOAC etc) for the analysis are used since these are regarded as validated and do not require full revalidation (for further details of the necessary degree of method validation see the APVMA's *Guidelines for the Validation of Analytical Methods for Active Constituent, Agricultural and Veterinary Chemical Products*). Details of all important operational parameters, such as instrumentation, sample preparation, method of extraction of the active constituent from the product, details of the reference standards and reagents preparation, validation data, copies of representative chromatograms and representative calculations should be provided (see *APVMA Guidelines for the Validation of Analytical Methods* for further details).

Analytical methods described in Collaborative International Pesticide Analytical Council (CIPAC) handbooks and Association of Official Analytical Chemists (AOAC) Manual for an agricultural active constituent and agricultural chemical product are legally recognised as the regulatory methods, and these procedures (if one is available) are used by the APVMA for determining compliance with the Agricultural and Veterinary Chemicals Code Act. It is recommended that analytical methods described in official and recognized publications, such as CIPAC handbooks and AOAC for a particular formulation be used, where available. Alternative analytical methods may be proposed by the registrants in place of regulatory methods.

The results and interpretation of the measurement of physical properties are highly dependent on the analytical procedures used. Wherever possible, it is recommended that standard CIPAC or equivalent accepted methods be used to measure the physical properties of agricultural chemical products. Where in-house company methods or other methods are used, then a full description of the procedure, with validation data, is required.

1.11 Validation of analytical methods

1.11.1 Determination of active constituent content

Validation data should be provided to confirm that the analytical procedures used in stability testing give reliable and accurate results. The type of validation data required is dependent on the analytical technique, but typically includes demonstration of linearity over a suitable concentration range, specificity, precision and accuracy (see [APVMA Guidelines for the Validation of Analytical Methods](#) for further information).

Analytical methods described in official and recognised publications, such as CIPAC handbooks and the AOAC manual for pesticidal products are regarded as validated and do not require revalidation. However, the suitability of these methods should be verified under actual conditions of use, i.e. the specificity and accuracy of the method should be demonstrated for the published method when applied to the relevant sample matrix and laboratory conditions.

1.11.2 Determination of physical properties

Validation of methods used to determine physical parameters will not be required, provided that CIPAC or equivalent accepted methods are used.

SECTION 2. TECHNICAL CHARACTERISTICS OF PRODUCTS

The data requirements that the APVMA adopts are derived from the *‘Manual on development and use of FAO and WHO specifications for pesticides’*, first edition 2002. Any divergences from FAO specifications must be described in detail, and justified.

2.1 Appearance and Physical State

These tests are performed visually and are described in qualitative terms such as solid, liquid, suspension etc.

2.2 Colour

The following test methods may be used to describe this parameter:

- American Society for Testing and Materials (ASTM) ‘Standard method for specifying colour by the Munsell system D-1535’.
- ASTM ‘Standard method for specifying colour of transparent liquids (Gardner Colour Scale, D-1544)’.

A visual description of colour is also acceptable.

2.3 Odour

This test is performed organoleptically and involves the use of descriptive terms e.g., thymol-like odour, characteristics of aromatic compounds, garlic-like etc.

2.4 Acidity/alkalinity and pH

This test is required for any product that is acidic ($\text{pH} < 4$) or alkaline ($\text{pH} > 10$).

The acidity or alkalinity is determined by titration with standard acid or alkali according to CIPAC method MT 31.

Where relevant (the product to be applied as aqueous dilution) the pH of a 1% aqueous dilution, emulsion or dispersion of the product must be determined and reported according to CIPAC method MT 75.3. A change in pH on storage can give an indication of instability of the active substance or product.

2.5 Wettability

Wettability of solid products which are diluted for use (e.g. wettable powders, water soluble powders, water soluble granules and water dispersible granules) is determined to ensure the product is adequately wetted before use.

CIPAC method MT 53

Acceptable limits: A product is considered acceptable if there is complete wetting in one minute without swirling.

If the product is outside these limits, then evidence must be submitted demonstrating acceptable dispersion in the spray tank or other application equipment.

2.6 Persistent foaming

Persistent foam is a measure the amount of foam likely to be present in a spray tank or other application equipment following dilution of the product with water in accordance with the label instructions.

CIPAC method MT 47.1 or 47.2

Although MT 47.2 was standardised for the determination of persistent foam in suspension concentrates, it is also applicable to other products that are dispersed in water.

Acceptable limits:	MT 47.1	max 25 mL foam after 1 minute
	MT 47.2	max 60 mL foam after 1 minute

2.7 Suspensibility

Suspensibility of water dispersible products (eg wettable powders, water dispersible granules and suspension concentrates) is determined to demonstrate that a sufficient amount of the active substance is suspended in the spray liquid to give a satisfactory, homogeneous mixture during spraying.

CIPAC method	MT 15.1	wettable powders
CIPAC method	MT 161	aqueous suspension concentrates
CIPAC method	MT 168	water dispersible granules
CIPAC method	MT 177	water dispersible powders
CIPAC method	MT 184	formulations forming suspensions on dilution with water

For the determination of suspensibility, chemical assay (active suspensibility) is the only fully reliable method to measure the mass of the active substance still in suspension. However, gravimetric determination (total suspensibility) or solvent extraction determination may be used on a routine basis, provided that these methods have been shown to give equivalent results to those of the chemical assay.

When the solvent extraction method is used, the product must be assayed using the same technique to allow comparison of the results.

Where there is more than one insoluble active substance present in the product, chemical assay (active suspensibility) is the only acceptable method.

The suspensibility test should be performed at the highest and lowest dilutions recommended on the product label.

Acceptable limits: the mean measured active suspensibility must not be less than 60% and not greater than 105%

Where a product is outside these limits, evidence must be submitted to demonstrate that the product is homogeneous on application through appropriate application equipment e.g., determination of active content in the spray at the beginning, middle and end of spraying operation.

2.8 Spontaneity of dispersion (suspension stability)

The spontaneity of dispersion of water dispersible products (eg water dispersible granules and suspension concentrates) is determined to show the product is easily and rapidly dispersed when diluted with water.

CIPAC method	MT 160	suspension concentrates
CIPAC method	MT 174	water dispersible granules

Chemical assay is the only reliable means to measure the mass of the active substance in suspension.

However, gravimetric determination or solvent extraction determination may be used on a routine basis, provided that these methods have been shown to give equivalent results to those of the chemical assay.

When the solvent extraction method is used, the product must be assayed using the same technique to allow comparison of the results.

Where there is more than one insoluble active substance present in the product, chemical assay is the only acceptable method.

Acceptable Limits: the mean measured active suspensibility or dispersibility must not be less than 60% and not greater than 105%

Where a product is outside these limits, evidence must be submitted to demonstrate that the product is homogeneous on application through appropriate application equipment.

2.9 Dilution stability

Dilution stability is determined to ensure water soluble products dissolve readily and, when diluted, produce stable solutions without precipitation, flocculation etc.

CIPAC method	MT 179	degree of dissolution and solution stability
CIPAC method	MT 41	dilution stability of herbicide aqueous solutions

Acceptable limits:	MT 41	'trace' of sediment after 30 minutes
	MT 179	max 2% on 75µm sieve

Where a product is outside these limits, evidence must be submitted showing the material separated will not block nozzles in application equipment.

2.10 Dry sieve test

The dry sieve test is designed to determine the particle size distribution of dustable powders and granules that are intended for direct application, to ensure acceptable application.

CIPAC method	MT 59.1	dusts
CIPAC method	MT 59.2	granular formulations
CIPAC method	MT 170	water dispersible granules

For dustable powders, if >5% of the product is retained on a 75 µm sieve, then the active content of material remaining on the sieve must be determined to demonstrate there was no separation of the active substance from the carrier.

Acceptable Limits:	maximum 5% retained on 75 µm sieve (dustable powders)
	Not more than (0.005 X active content in g/kg)% should be present as the active in the residues on the sieve.

2.11 Wet sieve test

For water dispersible products, a wet sieve test must be conducted. Wet sieve analysis determines the quantity of particles in a formulation collected on a screen after dilution in water.

CIPAC method	MT 185	wet sieve test, a revision of methods MT 59.3 and MT 167
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The residue remaining on a sieve is determined after dispersion to ensure that no unacceptable residue remains, which can cause blockage of nozzles in application equipment.

This test is applicable to wettable powders, suspension concentrates, water dispersible granules, aqueous capsule suspensions, dispersible concentrates, suspo-emulsions, water soluble granules and water soluble powders.

Acceptable limits:	Maximum 2% retained on a 75 µm sieve.
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Where a product is outside these limits, evidence must be submitted showing the product may be satisfactorily applied through appropriate application equipment with no blockages.
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2.12 Particle size distribution

The nominal size range for solid materials for direct application (eg dustable powders and granules) and solid materials for dispersion in water (eg wettable powder and granules) must be determined. The data are required to ensure that an acceptable proportion of the product is within an appropriate size range.

CIPAC method	MT 170	water dispersible granules
CIPAC method	MT 187	particle size analysis by laser diffraction
OECD method	110	powders/dusts

2.13 Dust content

The dust content of solid preparations must be determined to ensure the risk to operators is acceptable and to determine the potential for blockage of application equipment.

CIPAC method	MT 171	granular products
OECD method	110	powders/dusts

MT 171 describes two methods for the determination of dustiness but the gravimetric method is regarded as the 'Referee' method.

Acceptable limits: Where 1% by weight of the preparation has a particle size <50 µm, inhalation toxicity data required.

2.14 Emulsifiability, re-emulsifiability and emulsion stability.

For products which form emulsions, emulsifiability, emulsion stability and re-emulsifiability data are required to determine whether a product forms and maintains a stable emulsion.

CIPAC method	MT 36.1	5% dilution
CIPAC method	MT 36.2	1% dilution
CIPAC method	MT 36.3	emulsion characteristics and re-emulsification properties
CIPAC method	MT 173	0.1 - 2% dilution

MT 36.1 is designed to be conducted over a 24 hour period. If no separation of cream or oil is observed after 2 hours, then no further testing is required. However, if separation is observed, then the 24 hour test should be carried out.

For dilute emulsion, MT 173 is the preferred method. However, MT 36.1 may be used as a 'screening' method. If no separation of the 5% dilution is seen after 2 hours, then no further testing is required.

The test should be conducted in CIPAC waters A and D.

Acceptable limits: MT 36.1 maximum 2 mL cream, trace of oil after 30 minutes. If any separation observed, re-emulsification should be complete after 24 hours.

MT 173	min	98% after 4 hours
	max	102% after 4 hours

Where a product is outside these limits, evidence must be submitted showing the product remains homogeneous when applied through appropriate application equipment. If more than a trace of oil separates, consideration should be given to re-formulation of the product.

2.15 Dispersion stability of suspo-emulsions

Data must be provided to ensure that a sufficient amount of active constituent is homogeneously dispersed in suspension and emulsion in the spray liquid, to give a satisfactory and effective mixture during spraying.

CIPAC method MT 180

Acceptable limits: Maximum 2 mL cream, trace of oil after 30 minutes. If any separation observed, re-emulsification should be complete after 24 hours.

Where a product is outside these limits, evidence must be submitted showing the product remains homogeneous when applied through appropriate application equipment. If more than a trace of oil separates, consideration should be given to re-formulation of the product.

2.16 Pourability (rinsability) of suspension concentrates

Data are required to demonstrate that the user can make use of the maximum amount of the product in the container and that an excessive amount of the material does not remain in the container. This test should be conducted with suspension concentrates, capsule suspensions and suspo-emulsions.

CIPAC method MT 148, MT 148.1 (revised method)

Acceptable limits: max 5% residue; max 0.25% rinsed residue

Where a product is outside these limits, evidence must be submitted on the residue remaining in the commercial pack following recommended rinsing procedures.

2.17 Attrition and friability

Attrition is defined as the wearing away of the surface of a granule by friction or impact, particularly by granule-to-granule interaction.

Friability is defined as the tendency of the granule to crumble, breaking down to smaller particles.

Data are required to determine whether a granular material is robust under normal conditions of use and transport.

CIPAC method MT 178 measures attrition resistance of granules.

CIPAC method MT 178.2 measures attrition resistance of dispersible granules

Acceptable limits: where the material has an attrition resistance of < 98%, evidence is required that the material may be satisfactorily applied through application equipment.

2.18 Viscosity

The viscosity of a fluid is the property that determines the resistance offered to a shearing force under laminar flow conditions, eg resistance to slow stirring, or to flow through a capillary or narrow channel.

The kinematic viscosity of liquid formulation for direct application (ultra low volume products) must be determined.

CIPAC method	MT 22
OECD method	114

2.19 Flowability

CIPAC method	MT 44	powders
CIPAC method	MT 172	water dispersible granules

Acceptable limits: the sample should flow through the sieve after a maximum of 5 liftings

2.20 Dissolution rate of water soluble bags

CIPAC method	MT 176
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The dissolution rate of water soluble bags must be carried out to demonstrate particles of water soluble material will not block nozzles of application equipment.

Acceptable limits: 30 seconds

2.21 Disintegration time, and degree of dispersion or dissolution

Data are required to demonstrate that soluble or dispersible tablets disintegrate rapidly on addition to water and that the formulation is readily dispersed or dissolved.

SECTION 3. PARAMETERS TO BE TESTED IN STABILITY TRIALS

In addition to appearance and active constituent content, the relevant physical chemical properties of each formulation type must be monitored before and after storage. For the following shelf life specifications, the physical properties mentioned in the *Manual on development and use of FAO and WHO specifications for Pesticides* have been selected insofar as they are applicable to the given formulation type.

International codes used below are based on *Catalogue of Pesticide Formulation Types and International Coding System*. Technical Monograph No. 2, 5th Edition, Crop Life International, Brussels., Belgium.

In this guideline CIPAC MT methods are referenced as the appropriate MT number e.g. CIPAC MT 75 = MT 75.

3.1 DUSTABLE POWDERS (DP)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Dry sieve test	MT 59.1	
Packaging stability	Observation of packaging stability; there should be no caking in the pack on storage	

3.2 POWDERS FOR DRY SEED TREATMENT (DS)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Dry sieve test	MT 59.1	
Particle size distribution	OECD 110	
Adhesion to seeds	Appropriate validated method	
Packaging stability	Observation of packaging stability	

3.3 GRANULES (GR)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	

Pour and bulk density	MT 186	Only applicable to controlled release granules.
Particle size distribution	MT 58.2 or 58.3	
Dust content	MT 171	
Friability and attrition characteristics	MT 178	
Release rate of active constituent	Suitable validated method	
Packaging stability	Observation of packaging stability; there should be no loss of granule integrity or caking on storage	

3.4 TABLETS FOR DIRECT APPLICATION (DT)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		No broken tablets
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Tablet Integrity	Visual observation	
Tablet hardness		
Degree of attrition	MT 193	
Packaging stability	Observation of packaging stability	

3.5 WETTABLE POWDERS (WP)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	

Acidity /alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	Only for the product packaged in a sealed water soluble bag.
Wet sieve test*	MT 185	
Suspensibility*	MT 15.1, MT 177, MT 184	
Wettability*	MT 53.3	
Persistent Foam*	MT 47.2	
Dissolution of water soluble bags	MT 176	
Packaging stability	Observation of packaging stability: include a statement no caking on storage	

*Where the product is packaged in a water soluble bag then the wet sieve test, suspensibility, wettability test and persistent foam test must be carried using a solution of the product and water soluble bag in the actual ratio of application.

3.6 WATER DISPERSIBLE POWDERS FOR SLURRY SEED TREATMENT (WS)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity /alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Wet sieve test	MT 185	
Wettability	MT 53.3	
Persistent Foam	MT 47.2	
Packaging stability	Observation of packaging stability	

3.7 WATER DISPERSIBLE GRANULES (WG)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		

Active content	Appropriate validated method	Only for the product packaged in a sealed water soluble bag.
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Wet sieve test*	MT 185	
Degree of Dispersion	MT 174	
Suspensibility*	MT 184	
Wettability*	MT 53.3	
Persistent Foam*	MT 47.2	
Dust content	MT 171	
Flowability	MT 172	
Dissolution of water soluble bags	MT 176	
Attrition resistance	MT 178.2	
Packaging stability	Observation of packaging stability	

*Where the product is packaged in a water soluble bag then the wet sieve test, suspensibility, wettability test and persistent foam test must be carried using a solution of the product and water soluble bag in the same ratio as in the recommended application.

3.8 WATER DISPERSIBLE TABLETS (WT)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		No broken tablets.
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Tablet Integrity	Visual observation	
Suspensibility	MT 184	
Disintegration time	Appropriate method	
Wet sieve test	MT 185	

Persistent Foam	MT 47.2	
Packaging stability	Observation of packaging stability	

3.9 EMULSIFIABLE GRANULES (EG)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Wettability	MT 53.3	
Dispersion Stability	MT 180	
Wet Sieve Test	MT 185	
Dustiness	MT 171	
Persistent Foam	MT 47.2	
Packaging stability	Observation of packaging stability	

3.10 EMULSIFIABLE POWDERS (EP)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Wettability	MT 53.3	
Dispersion Stability	MT 180	
Wet Sieve Test	MT 185	
Persistent Foam	MT 47.2	

Packaging stability	Observation of packaging stability	
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3.11 WATER SOLUBLE POWDERS (SP)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Persistent foam	MT 47.2	
Wettability*	MT 53.3	
Degree of dissolution and solution stability*	MT 179	
Persistent foam*	MT 47.2	
Dissolution of water soluble bags	MT 176	Only required for the product packaged in a sealed water soluble bag.
Packaging stability	Observation of packaging stability	

*Where the product is packaged in a water soluble bag then the wettability, degree of dissolution, solution stability test and persistent foam test must be carried using a solution of the product and water soluble bag in the same ratio as in the recommended application.

3.12 WATER SOLUBLE POWDERS FOR SEED TREATMENT (SS)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Degree of dissolution and solution stability	MT 179	

Packaging stability	Observation of packaging stability	
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3.13 WATER SOLUBLE GRANULES (SG)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Dust content	MT 179	
Degree of dissolution and solution stability*	MT 176	
Dissolution of water soluble bags		Only for the product packaged in a sealed water soluble bag.
Packaging stability	Observation of packaging stability	

*Where the product is packaged in a water soluble bag then the wettability, degree of dissolution and solution stability test must be carried using a solution of the product and water soluble bag in the same ratio as in the recommended application.

3.14 WATER SOLUBLE TABLETS (ST)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Tablet Integrity	Visual observation	No broken tablets
Degree of dissolution and solution stability	MT 179	
Wet sieve test	MT 185	
Disintegration time	Appropriate method	

Persistent Foam	MT 47.2	
Packaging stability	Observation of packaging stability	

3.15 SOLUBLE CONCENTRATES (SL)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Solution Stability	MT 41	
Persistent Foam	MT 47.2	
Low temperature stability	MT 39.3	
Packaging stability	Observation of packaging stability	

3.16 SOLUTIONS FOR SEED TREATMENT (LS)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity or alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Solution stability	MT 41	
Low temperature stability	MT 39.3	
Packaging stability	Observation of packaging stability	

3.17 OIL MISCIBLE LIQUIDS (OL)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Miscibility with hydrocarbon oil	MT 23	
Low temperature stability	MT 39.3	
Packaging stability	Observation of packaging stability	

3.18 ULTRA LOW VOLUME LIQUIDS (UL)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, clarity, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Low temperature stability	MT 39.3	
Kinematic viscosity	MT 22, OECD 114	
Packaging stability	Observation of packaging stability	

3.19 EMULSIFIABLE CONCENTRATES (EC)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	

Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Emulsion characteristics	MT 36.1, MT 36.2, MT 36.3, MT 173 or MT 183	
Persistent Foam	MT 47.2	
Low temperature stability	MT 39.3	
Packaging stability	Observation of packaging stability	

3.20 DISPERSIBLE CONCENTRATES (DC)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Dispersion Stability	MT 180	
Wet Sieve Test	MT 185	
Persistent Foam	MT 47.2	
Low temperature stability	MT 39.3	
Packaging stability	Observation of packaging stability	

3.21 EMULSIONS, OIL IN WATER (EW)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Emulsion characteristics	MT 36.1, MT 36.2, MT 36.3, MT 173 or MT 183	

Pourability	MT 148.1	
Persistent Foam	MT 47.2	
Viscosity	MT 192	
Low temperature stability	MT 39.3	
Packaging stability	Observation of packaging stability	

3.22 EMULSIONS FOR SEED TREATMENT (ES)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Emulsion Stability on dilution with water	Appropriate method	
Persistent Foam	MT 47.2	
Low temperature stability	MT 39.3	
Packaging stability	Observation of packaging stability	

3.23 MICRO-EMULSIONS (ME)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Emulsion characteristics	MT 36.1, MT 36.2, MT 36.3, MT 173	
Persistent Foam	MT 47.2	

Low temperature stability	MT 39.3	
Packaging stability	Observation of packaging stability	

3.24 SUSPO-EMULSIONS (SE)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Dispersion Stability	MT 180	
Pourability	MT 148.1	
Wet sieve test	MT 185	
Persistent foam	MT 47.2	
Low temperature stability	MT 39.3	
Packaging stability	Observation of packaging stability	

3.25 SUSPENSION CONCENTRATES (SC)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Pourability	MT 148.1	
Suspensibility	MT 184	
Spontaneity of dispersion	MT 160	

Wet sieve test	MT 185	
Persistent Foam	MT 47.2	
Particle size distribution	MT 187	
Low temperature stability	MT 39.3	
Packaging stability	Observation of packaging stability	

3.26 SUSPENSION CONCENTRATES FOR SEED TREATMENT (FS)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Pourability	MT 148.1	
Suspensibility	MT 184	
Wet sieve test	MT 185	
Persistent Foam	MT 47.2	
Low temperature stability	MT 39.3	
Packaging stability	Observation of packaging stability	

3.27 CAPSULE SUSPENSIONS (CS)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Pourability	MT 148.1	

Suspensibility	MT 184	Testing of stability parameters (acidity/alkalinity/ pH range, pourability, suspensibility, spontaneity of dispersion, wet sieve test) required after freeze/thaw cycle
Spontaneity of dispersion	MT 160	
Wet sieve test	MT 185	
Persistent Foam	MT 47.2	
Freeze/thaw stability		
Packaging stability	Observation of packaging stability	

3.28 OIL-BASED SUSPENSION CONCENTRATES (OD)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Acidity/alkalinity or pH	MT 31 or MT 191 or pH range (MT 75.3)	
Pourability	MT 148.1	
Dispersion Stability	MT 180	
Wet sieve test	MT 185	
Persistent Foam	MT 47.2	
Low temperature stability	MT 39.3	
Packaging stability	Observation of packaging stability	

3.29 WATER SOLUBLE GELS (GW)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Dilution stability*	MT 41	
Packaging stability	Observation of packaging stability (no corrosion)	

*only required if the preparation is to be dissolved in water.

3.30 MOSQUITO COILS (MC)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Average weight of coils		
Burning time		
Strength of Coil		
Packaging stability	Observation of packaging stability (no corrosion)	

3.31 VAPORIZER MATS (MV)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Packaging stability	Observation of packaging stability (no corrosion)	

3.32 LIQUID VAPORIZERS (LV)

Recommended Test Parameters	Relevant CIPAC Method	Comments
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Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Packaging stability	Observation of packaging stability (no corrosion)	

3.33 BAITS:- INCLUDING GRAIN BAIT (AB), BLOCK BAIT (BB), GRANULAR BAIT (GB), READY TO USE BAIT (RB), PLATE BAIT (PB)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method Evidence of retention of biological efficacy may be acceptable	
Packaging stability	Observation of packaging stability and integrity	

3.34 PRODUCTS TO BE APPLIED AS SMOKES INCLUDING SMOKE TINS (FD), SMOKE CANDLES (FK), SMOKE CARTRIDGE (FP), SMOKE RODLET (FR), SMOKE TABLETS (FT), SMOKE GENERATORS (FU) AND SMOKE PELLET (FW)

Recommended Test Parameters	Relevant CIPAC Method	Comments
Appearance (physical state, colour, odour)		
Active content	Appropriate validated method	
Burning time		
Evidence of combustibility		
Packaging stability	Observation of packaging stability (no corrosion)	

SECTION 4. GLOSSARY OF TERMS

Accelerated stability Testing

Testing designed to increase the rate of chemical or physical degradation of a product by using exaggerated storage conditions.

Active constituent(s)	The component of a formulation responsible for the biological activity against pests and diseases, or in regulating plant growth.
Batch	A defined quantity of material produced in a single series of operations.
CIPAC methods	Analytical and physical test methods published in Collaborative International Pesticides Analytical Council (CIPAC) Handbooks or agreed by CIPAC as full methods prior to publication.
FAO specifications	International standards of quality for pesticides evaluated and published by FAO.
FAO tolerances	Tolerances (established by FAO) on the active constituent content of single determinations taking into account analytical and sampling errors and the manufacturing variations.
Content of active constituent	Where an FAO specification is available for an active constituent in a product, the tolerance limits must meet those in the FAO specification. However, where there is no appropriate FAO specification, the following guideline tolerances as detailed in the <i>Manual on the development and use of FAO specifications for plant protection products</i> are applicable:

Table 2. APVMA's Prescribed Allowable Variations of an Active Constituent in Agricultural Chemical Products.

Declared content in g/kg or g/L at 20 ± 2°C	Tolerance
Up to 25	± 15% of the declared content for homogeneous formulations (eg. emulsion concentrate, suspension concentrate and soluble liquid) or ± 25% for non-homogeneous formulations (e.g. water dispersible granule)
above 25 up to 100	± 10% of the declared content
above 100 up to 250	± 6% of the declared content
above 250 up to 500	± 5% of the declared content
above 500 g/kg or g/L	+ 25 g/kg or g/L

Pilot batches	A quantity of product, which is identical in formulation and equivalent in terms of manufacturing equipment and manufacturing method to a production batch, except for scale. Pilot batches should be of a minimum of 10% of the proposed production batch size.
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Product	A formulation containing active constituent(s) and formulant(s), which is intended for application, with or without dilution prior to use, and which is labelled with directions for use.
Real time stability Testing	Testing on the product stored in the container and closure system intended for marketing, under proposed storage conditions that support a proposed shelf life for that product.
Shelf life	Period of time during which the product remains suitable for use, and within approved shelf life specifications, provided that it is stored under conditions defined on the label in the proposed containers and closure.
Shelf life specifications	Specification with proposed limits, within which the properties of a formulation will remain after a minimum of 2 years storage, if no other statement as to the storage period is made. <i>Shelf life specifications must provide limits for the active constituent content and physical properties of the product.</i>

SECTION 5. REFERENCES

1. Pesticide Specifications, *Manual on the development and use of FAO and WHO specifications for Pesticides*, first edition, World Health Organization and Food and Agriculture Organization of the United Nations, Rome 2002.
2. Association of Official Analytical chemists, *Official Methods of Analysis of AOAC – International*; AOAC-International, Arlington, VA, U.S.
3. Collaborative International Pesticide Analytical Council (CIPAC) methods are published in the *CIPAC Handbooks*, details of the CIPAC handbooks may be obtained from Black Bear press, Kings Hedges Road, Cambridge, U.K.
4. American Society for Testing and Materials, *Annual Book of ASTM Standards*; ASTM, 1916 Race Street, Philadelphia, PA 19103, U.S.A.
5. Organisation for Economic Co-operation and Development, *Guidelines for Testing of Chemicals*, OECD 101-117; OECD, Paris, France