# GUIDANCE DOCUMENT FOR THE GENERATION OF DATA ON THE PHYSICAL, CHEMICAL AND TECHNICAL PROPERTIES OF PLANT PROTECTION PRODUCTS UNDER REGULATION (EC) No. 1107/2009 OF THE EU PARLIAMENT AND COUNCIL ON PLACING PLANT PROTECTION PRODUCTS ON THE MARKET.

## INTRODUCTION

The intention of this guidance document is to describe the chemical and physical data required to support the registration/authorisation of a preparation. These notes are intended to provide guidance on the scientific data requirements for plant protection products as they are set out in Regulation (EC) No. 284/2013 in accordance with Regulation (EC) 1107/2009 concerning the placement of Plant Protection Products (PPPs) on the market.

This document covers the principal preparation types currently used. Where a preparation type has not been included in the guidance, then applicants are encouraged to discuss this with the appropriate Member State Regulatory Authority at the earliest opportunity.

In considering the chemical, physical and technical characteristics and shelf-life requirements for a Plant Protection Product, the aim should be to show the PPP may be safely and efficaciously applied according to label instructions, and that the preparation retains the active substance and physical-chemical characteristics on storage.

These guidelines are intended to cover the data requirements for chemical pesticides. They are not intended for biological pesticides. Biological pesticides will be considered on a case by case basis, but as with the case of chemical pesticides, the basis of the data required will be to show the preparation can be safely and efficaciously applied.

**In general, guidance is provided by Annex point. Where an Annex point is self-explanatory no guidance is provided.**

### 1 IDENTITY OF THE PLANT PROTECTION PRODUCT

### 1.1 Applicant

### 1.2 Producer of the plant protection product and the active substance(s)

### 1.3 Trade name or proposed trade name and producer's development code number of the preparation if appropriate

### 1.4 Detailed quantitative and qualitative information on the composition of the plant protection product

#### 1.4.1 Composition of the plant protection product

Full details of the composition of the plant protection product must be submitted with each application. This includes the content of the active substance and co-formulants.

The declared (nominal) active substance content should be stated with tolerance limits. The content should be declared in terms of pure active substance in accordance with the identity established in the implementing regulation. This nominal target should also be the declared label content. In addition the content of the technical active substance(s) (based on the specified minimum purity) and where relevant the corresponding content of the variant (e.g. salts and esters) of the active substance(s) should be given.

In the case of encapsulated formulations (e.g. capsule suspension, CS), in addition to the content of the active substance (total content), the free (non-encapsulated) and

encapsulated active substance content should be provided where the CS is intended to have slow or controlled-release properties, or is intended to enhance operator safety or prevent impact on non-target crops for volatile actives. In the case of slow or controlled release formulations the release rate should also be provided. A test method for CS release rate may be product-specific.

If more than one active ingredient is encapsulated, limits must be provided for each active. Methods for determination of free active ingredient and release rate may be product-specific. Where encapsulation is intended to control the stability of the active ingredient(s), the levels of free and encapsulated active ingredient are required. Full details are included in the tables outlining the physical-chemical properties in Appendix 1.

The concentrations should be expressed as follows:

- For solids, aerosols, volatile liquids (maximum boiling point 50°C) as % w/w and g/kg,
- For other liquid formulations including viscous liquids/gels, as % w/w and g/L,
- For Gas formulations as % v/v and % w/w.

The tolerance limits of the active substance content normally take into account only manufacturing, sampling and analytical variations and refer to the mean analytical result obtained. The following guideline tolerances as accepted by the Manual for the development and use of FAO and WHO Specifications for pesticides[1] are applicable. Where the proposed tolerance limits for an active substance are outside the above ranges then this must be fully justified.

[1] Manual for the development and use of FAO and WHO Specifications for pesticides, November 2010 2nd revision of the 1st Edition and any additional supplements.

| **Declared content in g/kg or g/l at 20°C ± 2°C** | **Tolerance** |
|---|---|
| up to 25 | ± 15% of the declared content for homogeneous formulations (EC, SC, SL etc)<br>± 25% of the declared content for heterogeneous formulations (GR, WG etc) |
| 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 | ± 25 g/kg or g/l of the declared content |
| Note – In each range the upper limit is included. | |

Positive deviations from the upper limits may be needed if the preparation is manufactured with an overage. However, where an overage is proposed this must be justified, the overage must be kept as low as practicable and evidence must be submitted that the breakdown of the active substance does not lead to hazardous products or adversely affect the safety or efficacy of the preparation.

The maximum content of any relevant impurities, where appropriate, must be given.

### 1.4.2 Information on the active substance(s)

For active substances their ISO common names or proposed ISO common names, their CIPAC numbers, and, where available, their EC numbers should be provided.

Where an active substance is present as an ester or a salt, the active substance content should be expressed as the amount of the ester or salt present (as the technical material) with a statement declaring the amount of the active principle.

**e.g. 'x g/l or g/kg of the salt/ester which is equivalent to y g/l or g/kg of the free acid.'**

For hydrated salts such as ferrous sulfate the active substance content must be declared in terms of the amount of hydrated salt present with a statement declaring the amount of the anhydrous form present.

**e.g. x g/kg of ferrous sulfate heptahydrate equivalent to y g/kg anhydrous ferrous sulfate.**

### 1.4.3 Information on safeners, synergists and co-formulants

The information on co-formulants must include:

i) Trade name
ii) Chemical name (according to Regulation (EC) No 1272/2008, IUPAC and CAS)
iii) CAS number
iv) EC number
v) Structure, structural formula, chemical description
vi) Function
vii) Safety data sheets

Safety data sheets should be up to date, fully applicable to the formulant and in accordance with Regulation (EC) No 1907/2006 (REACH) and Regulation (EC) No 1272/2008 (CLP). Ideally safety data sheets submitted to the evaluating Member State should be less than 2 years old; however, if the supplier does not update the MSDS this frequently a statement may suffice. For commodity chemicals, a representative MSDS rather than a separate one for every supplier used may be acceptable.

The information submitted for each preparation component must be sufficient to chemically characterise that component, for example, for polyethoxylated components the information should include the degree of ethoxylation and the number of mole equivalents of ethylene oxide.

Where a co-formulant is itself a mixture full details of the composition must be submitted.

A description of the formulation process should be provided. This information is required as it is critical to the physical chemical properties of the formulation; for example, for granular preparations whether the active substance is incorporated into the granule or sprayed on the surface of the granule can impact on the properties of the preparation.

### 1.4.4 Changes in the chemical composition of a preparation

Where alternative trade names for co-formulants are given, the components must be identical in chemical composition. Separate guidance is available for changing the chemical composition of a preparation under SANCO 12638/2011. The guidance document outlines the procedures to follow under Regulation (EC) No. 1107/2009 and gives the criteria for acceptable compositional changes.

## 1.5 Type and code of the plant protection product

The type of preparation should be provided according to the codes listed in the latest edition of the Technical Monograph no. 2 by CropLife International. If a particular preparation type is not defined precisely in this publication then a full description of its physical nature and state should be provided, together with proposals for a suitable description and for the critical physical, chemical and technical properties.

## 2 PHYSICAL, CHEMICAL AND TECHNICAL PROPERTIES OF THE PLANT PROTECTION PRODUCT

Data must be submitted in the form of test reports with full details of the methods used and an explanation and justification of any deviation from standard method protocols. Reports should identify the name and batch of the preparation tested. Where the composition of the tested preparation differs from that for which authorisation is sought then this must be fully justified.

**Good Laboratory Practice (GLP)**

Full details of GLP requirements for specific studies are given in Commission Document 7109/VI/94 Rev 6. It is the UKs understanding that the requirements outlined in this document are still applicable under Regulation (EC) 1107/2009 until a decision is made otherwise. It should be noted that where individual tests have a GLP requirement this still applies in the context of a storage stability study for which overall there is no GLP requirement. However, GLP is required for active substance / impurity determination where hazardous compounds may be formed on storage.

All studies conducted must be to GLP where appropriate. Where conducting studies to GLP is not possible, they may be accepted if they are considered scientifically valid.

A GLP certificate for the test facility should be provided. The certificate dates must be relevant to the date(s) when the study was conducted.

**Methods**

**Active substance content**

For the determination of active substance content and relevant impurities (if appropriate), the methods used must be fully reported and validated. Additional guidance on method validation can be found in the most recent version of the European Commission guidance document SANCO/3030/99.

Where an active substance is present as an isomerically (including enantiomerically) pure form, then the method must be capable of separating and quantifying the isomers of the active substance. Where isomers are present in different ratios, methods of analysis must be able to separate isomers in order to demonstrate compliance with the active substance established for approval, and to ensure isomers are stable upon storage. During storage however, separation and quantification of the isomers of the active substance shall only be required when their ratio can change. Where evidence is presented that the isomer ratio cannot change during storage (e.g. non-racemisable isomers) separation and quantification following storage won't be required.

For non-racemic mixtures of stereoisomers or when the stereoisomers do not have the same biological activity, a method capable of separating and quantifying the isomers of the active substance is required. For racemic mixtures, or when the optical isomers are equally active separation would not be required.

**Physical and technical properties**

The methods used for the determination of physical properties should be in accordance with the requirements of Commission Communication 2013/C 95/02 and be standard internationally recognised or ring tested methods such as the "EC methods" (Reg (EC) No. 440/2008), OECD methods, from the UN RTDG Manual or those developed by CIPAC[2]. In this guidance document, CIPAC MT methods will be referenced as the appropriate MT number. eg CIPAC MT 75.3 ⇒ MT 75.3.

CIPAC periodically conducts a review of the methods developed under the auspices of CIPAC to take into account that older methods become obsolete or superseded and also to take into account advances in technology. A negative list of methods declared as "obsolete" or "superseded" is published on the CIPAC website.

However, methods which are "no longer supported" by CIPAC may still be considered fit for regulatory purposes,[3] depending upon when the study was conducted and whether the data meet the regulatory need. Methods no longer supported are not included in this guideline.

Where methods other than those specified are used they must be fully described and their relationship to the appropriate CIPAC method justified. If no CIPAC or other recognised method is available to determine a particular property, then an 'in-house method' may be acceptable providing appropriate validation data demonstrating the applicability, repeatability and robustness of the method are submitted.

If test results do not meet the acceptable criteria, this will not necessarily mean that an authorisation cannot be granted. The technical characteristics are assessed using laboratory based tests which have limitations. In some cases, it may be more appropriate to demonstrate how the formulation behaves under the intended conditions of use. For example, if the formulation does not meet the acceptance criteria for the suspensibility test (CIPAC MT 184) then e.g. a sprayability test should be conducted. In the sprayability test the formulation would be used as intended and samples collected at various intervals and analysed for the active content, using a validated method, to determine if the formulation is homogenous on application.

**Ready to use preparations**

The data required are the same as those for the more concentrated preparation type e.g. if the preparation is a ready to use oil in water formulation, the data for the EW formulation type must be provided. Generally, tests that are required to demonstrate acceptable performance of the product when diluted in the spray tank e.g. persistent foam will not be required as the products are already diluted. However the data submitted must show that the preparation is homogeneous and can be satisfactorily applied according to the label instructions. For example, for ready to use suspensions evidence must be supplied showing the preparation is homogeneous on application.

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[2] CIPAC Methods are published in the CIPAC Handbooks, details are available from www.cipac.org/

[3] CIPAC maintains a list of methods which are considered to no longer be supported. Refer to http://www.cipac.org/obsolet_methods.htm.

**Physical, chemical and technical properties of the plant protection product**

**2.1 Appearance**

**Physical state**

This should be described in qualitative terms such as solid, liquid, suspension etc.

**GLP - No**

**Colour**

No method is specified but the following may be appropriate:

ASTM 'Standard method for of specifying colour by the Munsell system D-1535'

ASTM 'Standard method for of specifying colour of transparent liquids (Gardner Colour Scale), D-1544

However a visual description of colour is also acceptable.

**GLP - No**

**2.2 Explosive properties and oxidising properties**

**2.2a Explosive properties**

**Method A14 of Regulation (EC) No. 440/2008**

**United Nations Recommendations on the Transport of Dangerous Goods (UN RTDG) Manual of Tests and Criteria ST/SG/AC.10/11/Rev. 5 – Part I (Test series), section 11.**

Solid preparations should be tested for the effect of flame, shock and friction.

Liquid preparations should be tested for the effect of flame or shock.

The tests for explosivity are designed to give results that can be evaluated directly against the criteria for classification and labelling.

The criteria for classification of a solid or liquid as explosive under CLP are as outlined in Regulation (EC) No 1272/2008.

In accordance with the criteria set out in Appendix 6 of the United Nations 'Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria', the tests do not have to be carried out if "A case may be made that the preparation cannot be explosive if the individual components of the preparation are not classified as explosive (i.e. the active substance and the co-formulants do not contain any groups associated with explosive properties in their structures or by reference to the MSDS for all of the co-formulants and active substance, which demonstrate that they are not explosive)". Refer to the table below for examples.

| Structural formula | Examples |
|---|---|
| C-C unsaturation | Acetylenes, actylides, 1,2-dienes |
| C-Metal, N-Metal | Grignard reagents, organo-lithium compounds |
| Contiguous nitrogen atoms | Azides, aliphatic azo compounds, diazonium salts, hydrazines, sulfonylhydrazines |
| Contiguous oxygen atoms | Peroxides, ozonides |
| N-O | Hydroxylamines, nitrates, nitro compounds, nitroso compounds, N-oxides, 1,2-oxazoles |
| N-halogen | Chloramines, fluoroamines |
| O-halogen | Chlorates, perchlorates, iodosyl compounds |

Alternatively classification may be made through the determination of the thermal characteristics of the preparation (Differential Thermal Analysis, Differential Scanning Calorimetry) which can provide supplementary data such as evidence of exothermic decomposition, rate of energy release etc. These data can be used to help demonstrate the thermal behaviour of a product. The DSC should confirm that the exothermic decomposition energy is <500 J/g and the onset of exothermic decomposition is <500°C, for the non-classification of the formulation as an explosive. It should be stated if product is thermally sensitive or sensitive to shock or friction.

**GLP – Yes (for experimental determinations only)**

## 2.2b Oxidising properties

| | | |
|---|---|---|
| **Method** | **A17** | **of Regulation (EC) No. 440/2008 for solids** |
| | **A21** | **of Regulation (EC) No. 440/2008 for liquids** |
| | **Test O.1** | **Test for oxidizing solids (Manual of tests and Criteria Part III sub-section section 34.4.1 of United Nations Recommendations on the Transport of Dangerous Goods – UN RTDG)** |
| | **Test O.2** | **Test for oxidizing liquids (Manual of tests and Criteria Part III sub-section section 34.4.2 of UN RTDG)** |

The methods are not applicable to gases and materials that are explosive or highly flammable or organic peroxides.

The oxidising properties do not have to be determined if it can be shown, without reasonable doubt, on the basis of thermodynamic information that the preparation is incapable of reacting exothermically with combustible materials, or if a case can be made showing the individual components of the preparation are not oxidising. The case should meet the criteria set out in Appendix 6 of the United Nations 'Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria'. Reference can be made to the Material Safety Data Sheets and structural characteristics of the co-formulants (i.e. that the formulation does not contain Cl, F or O or if it does contain Cl, F or O but these are bonded to C and/or H only). Organic peroxides are classified based on their chemical structure and on the available oxygen and hydrogen peroxide content of formulations.

**A17**: Burning rates of test substance and reference substance to be reported. The formulation is not oxidizing when burning rate of test substance is less than reference substance

**A21**: mean pressure rise time for test substance and reference substance to be reported. The formulation is not oxidizing when time for mean pressure rise of test substance is greater than for reference substance.

The criteria for classification of a solid or liquid as oxidising under CLP are as outlined in Regulation (EC) No 1272/2008.

**GLP - Yes (for experimental determinations only)**

## 2.3 Flammability and self-heating

### 2.3a Flashpoint

**Method A9 of Regulation (EC) No. 440/2008**

This test is only required for preparations that contain flammable liquids. Only data generated using a closed cup method are considered acceptable. The method is appropriate for all liquid preparations **except aerosols**. Aerosol flammability should be tested in accordance with the methods described in Annex I, Part 2.3 of Regulation (EC) No 1272/2008 and classified accordingly.

The preparation is classified as 'H224: Flammable liquid 1' where the flashpoint is <23°C and the boiling point ≤35°C.

The preparation is classified as 'H225: Flammable liquid 2' if the flash point is <23°C and the boiling point >35°C.

Preparations are classed as 'H226: Flammable liquid 3' where the flashpoint is ≥23°C but ≤60°C.

The test is not required if a case can be made showing the individual components of the preparation are not flammable. The case should meet the criteria set out in Appendix 6 of the United Nations 'Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria'. Reference can be made to the Material Safety Data Sheets.

### 2.3b Flammability

| | | |
|---|---|---|
| **Method** | **A10** | **of Regulation (EC) No. 440/2008 for solids** |
| | **A11** | **of Regulation (EC) No. 440/2008 for gaseous materials** |
| | **A12** | **of Regulation (EC) No. 440/2008 (Contact with water)** |
| | **Test N.1** | **test method for readily combustible solids (Manual of tests and Criteria Part III sub-section 33.2.1 of UN RTDG)** |

Data according to method A12 are only required if the preparation is designed to liberate a gas on contact with water or if data on the active substance or co-formulants show the individual components may release a gas on contact with water.

Solid preparations are classified as 'flammable' if they readily catch fire after brief contact with a source of ignition and which continue to burn or to be consumed after removal of the source of ignition. The following classifications are proposed under Regulation (EC) No 1272/2008 (CLP):

| Classification category | Criteria |
|---|---|
| 1: Danger, flammable solid | Burning rate test<br>Substances and mixtures other than metal powders:<br>(a) wetted zone does not stop fire<br>and<br>(b) burning time < 45 seconds or burning rate > 2.2 mm/s<br>Metal powders:<br>burning time ≤5 minutes |
| 2: Warning, flammable solid | Burning rate test<br>Substances and mixtures other than metal powders:<br>(a) wetted zone does stops the fire for at least 4 minutes<br>and<br>(b) burning time < 45 seconds or burning rate > 2.2 mm/s<br>Metal powders:<br>burning time >5 minutes and ≤ 10 minutes |

Gaseous substances and preparations are classified as 'Flammable' if they are flammable in contact with air at ambient temperature and pressure (20°C) and standard pressure (101.3 kPa).

For gases the lower explosion limit (LEL) and the upper explosion limit (UEL), or a statement that the gas is non-flammable over a full range of mixtures with air, must be submitted. The lower and the upper explosion limits are those limits of concentration of the flammable gas in admixture with air at which propagation of a flame does not occur.

The test is not required if a case can be made showing the individual components of the preparation are not flammable. The case should meet the criteria set out in Appendix 6 of the United Nations 'Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria'. Reference can be made to the Material Safety Data Sheets.

**2.3c Self-heating**

| | | |
|---|---|---|
| **Method** | **A15** | **of Regulation (EC) No. 440/2008 for liquids and gases** |
| | **A16** | **of Regulation (EC) No. 440/2008 for solids** |
| | **Test N.4** | **test method for self-heating substances (Manual of tests and Criteria Part III sub-section 33.3.1.6 of UN RTDG)** |

According to A15 the self-ignition temperature for liquids and gases is the lowest temperature at which the preparation will ignite when mixed with air under defined conditions.

According to A16 the self-ignition temperature for solids is the temperature of the oven at which the sample temperature reaches 400°C by self-heating.

When using Test N.4 the classification is in accordance with CLP. The criteria for classification of a self-heating substance/mixture are as outlined in Regulation (EC) No 1272/2008.

**GLP - Yes (all flammability tests)**

**2.4 Acidity/alkalinity and pH value**

**2.4a pH**

| | | |
|---|---|---|
| **Method** | **MT 75.3** | **Determination of pH values** |

In the case of aqueous preparations, the pH value of the neat preparation should be determined.

For solid and non-aqueous liquid preparations to be applied as aqueous dilution the pH of a 1% aqueous dilution, emulsion or dispersion of the preparation should be determined.

A change in pH on storage can provide an indication of instability of the active substance or preparation.

**GLP – Yes**

**2.4b Acidity/alkalinity**

| | | |
|---|---|---|
| **Method** | **MT 191** | **Free acidity or alkalinity of formulations** |
| | **MT 31** | **Free acidity or alkalinity** |

MT 191 is the preferred method.

The acidity or alkalinity should be tested if the preparation has pH <4 or pH >10. The test expresses free acidity or alkalinity calculated as $H_2SO_4$ or NaOH.

The pH only gives an indication of the ionisation of strong acids/bases. The acidity/alkalinity gives the total concentration of weak and strong acids/bases and hence is used to assess corrosive nature of formulations.

**GLP – Yes**

**2.5 Viscosity and surface tension**

**2.5a Viscosity**[⊕]

| | | |
|---|---|---|
| **Method** | **OECD** | **Test guideline No. 114** |
| | **MT 192** | **Viscosity of liquids by rotational viscometry** |

**Where the hydrocarbon content is ≥ 10 % the preparation must be considered for classification as an aspiration hazard, based on the viscosity of the formulation.**

The kinematic viscosity must be determined and reported at 20°C and 40 °C.

Dynamic viscosity can be converted to kinematic viscosity as follows:

$$\frac{\text{Dynamic viscosity (mPa s)}}{\text{Density (g/cm}^3\text{)}} = \text{Kinematic viscosity (mm}^2\text{/s)}$$

The viscosity is required for all liquids and the results should be reported with full details of the test methodology. For liquid formulations the viscosity shall be determined at a minimum of two shear rates. All tests must be conducted at 20 °C and 40°C.

[⊕] note :- the lowest dynamic viscosity of liquids occurring at room temperature is approximately 0.2 mPa.s (1mPa.s = 1 cP)

Only the rotational viscometer can be used for determination of the (dynamic) viscosity of liquids. CIPAC method MT 192, based on OECD 114, is the preferred method. CIPAC MT 192 requires at least two shear rates to be reported, beginning with the lowest one. Unless otherwise specified, shear rates within the range of 20 to $100s^{-1}$ are recommended.

**Classification criteria for aspiration hazard**

**Classification criteria under CLP**

Under Regulation (EC) No 1272/2008, the following hazard category *for mixtures* for aspiration toxicity is proposed:

> Contains at least 10% of a substance which has been classified as a Category 1 aspiration hazard (e.g. hydrocarbon) and which has a kinematic viscosity of $\leq 20.5$ $mm^2/s$ measured at 40˚C
> If a mixture is split into 2 layers whereby one of the layers is classified as Category 1 aspiration hazard as detailed above then the whole mixture shall be classified in Category 1 (hazard statement H304). This criterion applies to all preparations where separation is observed either prior to or following storage.

Note: For classification according to Regulation (EC) 1272/2008, the surface tension is not used to determine whether the product is an aspiration hazard - however these data are still required.

**GLP - Yes**

**2.5b Surface tension**

| | | |
|---|---|---|
| **Method** | **A5** | **of Regulation (EC) No. 440/2008** |
| | **OECD** | **Test guideline No. 115** |

The surface tension of all liquid preparations should be determined at the highest in-use spray concentration.

**GLP - Yes**

**2.6 Relative density and bulk density**

**2.6a Relative density**

| | | |
|---|---|---|
| **Method** | **A3** | **of Regulation (EC) No. 440/2008** |
| | **OECD** | **Test guideline No. 109** |

The specific method (from A3) must be appropriate to the preparation type.

The relative density $D_4^{20}$ should be reported. This is the ratio between the mass of a volume of substance to be examined, determined at 20 °C, and the mass of the same volume of water, determined at 4 °C. The relative density has no dimension.

**GLP - Yes**

**2.6b Bulk (pour and tap) density**

| | | |
|---|---|---|
| **Method** | **MT 186** | **Bulk density for powder or granular preparations** |

The pour and tap density of powder and granular formulations should be reported.

**GLP – yes**

## 2.7 Storage stability and shelf-life: effects of temperature on technical characteristics of the plant protection product

Data are required to demonstrate the stability of the active substance and the physico-chemical characteristics of the preparation on storage. Accelerated data and data from storage at ambient temperatures (shelf-life) must be submitted, but data from accelerated testing can give a useful indication of the stability and may be extrapolated to propose a shelf life for the product, with the proviso that shelf-life data are subsequently provided to support the proposal. In some circumstances shelf-life data alone may be sufficient. More detailed comments on temperature regimes for accelerated and ambient studies follow under sections 2.7.1 and 2.7.3 respectively. Additionally, in the case of liquid preparations, the effect of low temperatures on stability must be provided. For capsule suspensions this should be carried out as temperature cycling (i.e. freeze/thaw) instead of cold stability testing to MT 39.3 to demonstrate that capsule integrity is not adversely affected. For some formulations use of the phrase 'Protect from Frost' on the product label may be acceptable *in lieu* of cold stability test data. For other formulations either a more detailed reasoned case or test data will be required (see Section 2.7.2 Cold Stability Testing for further information).

The active substance content and the relevant physical and technical characteristics of the product must be determined before and after storage. The stability tests must be carried out on the same sample (i.e. same lot or batch) before and after storage.

With respect to relevant impurities, storage stability data are only required where the relevant impurity may form upon storage of the product or during manufacture of the formulation. For those impurities that are formed during manufacture of the active substance i.e. process impurities that are controlled within the technical specification of the active substance, and for which a scientifically valid justification can be provided to support the case that they will not form upon storage of the formulation, levels prior to and following storage do not need to be determined. A method of analysis for such impurities is still required however, since a method must be available to monitor levels of relevant impurities in formulations post-registration for compliance purposes. It is recognised that a loss of up to 5% of the active substance is unlikely to adversely affect the safety or efficacy of the preparation. Where a loss of >5% of active substance occurs then the fate of the active substance must be addressed and the breakdown products identified. For preparations in which the active substance content is relatively low (< 25 g/kg or g/L) losses of > 5% *may* be less significant in terms of breakdown products. In such cases the apparent fate of the active substance must still be addressed but reasoned cases *may* suffice. All such instances will necessarily be treated on a case by case basis.

Where a loss of ≥10% occurs during the accelerated study, ambient shelf-life data from interim time periods e.g. 3, 6, 12 months should be submitted to address the kinetics of breakdown and establish an appropriate shelf life for the product.

It should be noted that changes of active concentration on storage due to degradation are a property distinct from the active substance tolerance limits as described in

section 1.4.1 Therefore even if the loss does not result in exceedance of the tolerance limits a > 5% loss still needs to be addressed.

Although it is fully acceptable to collect data at T0 and T24 only when conducting a 2 year ambient storage stability study, it is worthwhile considering collection of data at interim time points e.g. at three month intervals, in order to collect a more thorough dataset to understand the effects of storage. Furthermore, in line with the comment above for accelerated storage, if ≥10% loss of active substance content occurs upon ambient storage, the active substance content at interim time periods e.g. 3, 6, 12 months should be submitted to address the kinetics of breakdown and establish an appropriate shelf life for the product, therefore in this instance collection of data at T0 and T24 only would not be acceptable.

It is recognised that, on storage, plant protection products may undergo changes which will be dependent on the active substance, the preparation, container and the conditions of storage. Such changes may be acceptable if they have no adverse effect on the operator, consumer or environmental safety, the application or the biological performance. Where there is a significant change in the physical characteristics of the preparation e.g. suspensibility then the relevance of the change must be discussed and if necessary labelling proposals made.

Where a repellent has been added to a preparation e.g. to reduce risk to non-target species, then the storage stability testing should also demonstrate retention of repellency. This may be achieved through e.g. analytical determination of the levels of the repellent, or through demonstration that the efficacy of the repellent is retained before and after storage.

**Packaging for stability tests**

Ideally storage stability tests should be performed in the commercial packaging or the material of which the commercial pack is to be manufactured; however, glass may be used in the accelerated tests as outlined in the relevant CIPAC MT methods.[4] In all cases the proposed commercial packaging must be declared. See Section 2.11 for further details of extrapolations between packaging types.

[4] The new EU PPP data requirements (stated in Commission Regulation (EU) No 284/2013) which are valid from 1st January 2016, state that "*Consideration shall be given to performing this test in packaging made of the same material as the commercial packaging*".

### 2.7.1 Accelerated testing

**Method MT 46.3 Accelerated storage procedure**

Accelerated tests are performed at elevated temperatures to ensure that the properties of formulations are not adversely affected by storage at high temperature. However, it is accepted that preparations are complex mixtures and elevated temperatures may initiate a reaction which may not occur under 'normal' conditions of storage. Unless a preparation is shown to be very stable under all conditions, care must be exercised in the interpretation of results from such tests.

Accelerated data alone are not sufficient for authorisation, however these data may also be extrapolated to propose a shelf life for the product, with the proviso that shelf-life data are subsequently provided to support the proposal.

The usual storage regime for accelerated tests is 2 weeks at 54°C (±2°C). However, some preparations may not be stable under these conditions and alternative time/temperature regimes may be used:

| Temperature (±2°C) | Time (weeks) |
|---|---|
| 54 | 2 |
| 50 | 4 |
| 45 | 6 |
| 40 | 8 |
| 35 | 12 |
| 30 | 18 |

Alternative time/temperature regimes may be proposed but the choice must be supported by a reasoned, scientific case.

**GLP - GLP is not an overall requirement for storage stability studies. However, as part of the determination of technical characteristics of the preparation, GLP is required for certain individual tests carried out within a storage stability study, e.g. pH. GLP is required for a.s. determination only where hazardous compounds may be formed on storage. To meet the GLP requirements for individual tests it may be advantageous to carry out all tests within the storage stability study in a GLP test facility.**

Where accelerated testing shows a significant change in active substance content or physical characteristics then results from ambient temperatures must demonstrate that, under 'normal' conditions of storage, the preparation retains acceptable active substance content and physical characteristics. Where a loss of ≥10% occurs ambient shelf-life data from interim time periods e.g. 3, 6, 12 months should be submitted to address the kinetics of breakdown.

### 2.7.2 Cold Stability testing

**Method MT 39.3 Low temperature stability of liquid formulations**

Liquid preparations where the active substance or preparation may crystallise, or where phase separation could occur should also be tested at 0°C or lower. For capsule suspensions (CS, ZC, ZW, ZE), the effects of low temperature should be carried out as temperature cycling (i.e. freeze/thaw). Unless otherwise agreed, the freeze/thaw stability test shall cycle the formulation between room temperature (e.g. 20 ± 2°C) and -10 ± 2°C on 18-hour-freeze/6-hour-melt cycles for a total of 4 cycles, in order to demonstrate that capsule integrity is not adversely affected.

Appropriate technical characteristics of the preparation which may be adversely affected by low temperatures should be determined after storage. These will often be related to the precipitation or separation of liquid phases. If in the test an effect is observed e.g. phase separation then the reversibility of this must be addressed. The requirements are shown for the individual preparation types in Appendix 1

Where the preparation is adversely affected by freezing conditions, then this should be indicated and the phrase 'Protect from Frost' must be included on the label.

For some formulations use of the phrase ‘Protect from Frost’ on the product label may be acceptable *in lieu* of test data. For other formulations either a more detailed reasoned case or test data will be required.

**GLP – no**

### 2.7.3 Ambient testing (shelf-life)

Real-time and ambient temperature testing is performed under ‘normal conditions’, usually over a period of 2 years. The results produced give a more accurate description of the likely properties and do not require extrapolation. However, such tests require a prolonged testing period. These tests are also the most appropriate in producing information on the stability of the packaging for a product.

The tests should be conducted at ambient temperature or, 20°C, 25°C or 30°C dependent on the expected geographical areas of use.

Ambient temperatures must reflect the maximum and minimum temperatures likely to be experienced in a warehouse, farm store or garden store for amateur products. Where tests are not conducted under ambient conditions, then the thermostatted temperature used during the storage period must be justified and recorded during the study.

For laboratory tests, the following values are appropriate for different climatic regions:

| | |
|---|---|
| temperate climate | 18 – 22 °C |
| hot climate | 23 - 27 °C |
| very hot climate | 28 – 31 °C |

In line with the CropLife International (formerly GIFAP) Monograph 17 2nd Edition,[5] it is recommended that studies are conducted at 20°C (or the nearest ambient temperature) *and* 30°C if product sales across countries with widely differing temperature ranges is expected.

The data submitted must support the proposed shelf-life of the preparation. It is normally expected that a preparation should have a shelf life of at least two years. This is based on the premise that a preparation may be purchased for use in one season but not be used in that season and kept to the following year or next season. Where a preparation has a shelf life of less than 2 years, this must be fully justified and the label must identify the manufacturing date and the 'Use by' date.

Following storage of the packaging any changes in the pack such as panelling, ballooning, condition of seals and seams and weight change must be reported in detail.

**GLP - GLP is not an overall requirement for ambient storage studies. However, as part of the determination of technical characteristics of the preparation, GLP is required for certain individual tests carried out within an ambient storage study, e.g. pH. GLP is required for a.s. determination only where hazardous compounds may be formed on storage. To meet the GLP requirements for individual tests it may be advantageous to carry out all tests within an ambient storage study in a GLP test facility.**

[5] Available from the CropLife website (https://croplife.org/wp-content/uploads/2014/05/Technical-Monograph-17-2nd-edition-June-2009.pdf)

## 2.8 Technical characteristics of the plant protection product

The properties required are in accordance with Regulation (EC) No 1107/2009 and the Manual for the Development and Use of FAO and WHO Specifications for Plant Protection Products. In this guidance document the technical characteristics for specific formulation types are presented in Appendix 1. Those properties which must be determined before and after storage are indicated. The appropriate limits for each property are described in this section.

Where a test is not considered applicable to a particular preparation then this must be explained and justified.

Where information is not given on a specific formulation then a logical approach should be taken addressing the appropriate properties and using the relevant tests. This also applies to mixed formulation e.g. ZC, ZE and ZW.

Tests of physical properties cannot replicate what happens in the field under all circumstances. Instead, the tests provide simple models against which satisfactory/unsatisfactory performance may be judged. Test results are therefore indicative of physical performance; they do not define exactly how a product will perform under specific conditions.

For some physico-chemical tests, recommended limits are stated. For example, in the case of suspensibility, not less than 60 % of the active ingredient shall remain in suspension. Where individual tests give adverse results further field tests will be required to demonstrate that the preparation can be effectively applied. For example this may take the form of a sprayability study. Additional studies should reflect worst case conditions of use according to Good Agricultural Practice.

In some test methods the test results may be described in relatively subjective terms such as 'trace of sediment' e.g. emulsion stability. In these circumstances the study reports must supply quantitative estimates based on (at least) visual inspection (e.g. trace of sediment of approx. vol. 0.25ml) and fully describe the appearance of any sediment etc.

**CIPAC Standard Water**

The physical properties of formulations that are diluted with water before use can be affected by the hardness of the water used for dilution. CIPAC Handbook F lists standard waters that may be used in laboratory tests,[6] to simulate naturally occurring waters.

With certain exceptions, Standard Water D (representing standard hard water) should be adopted in tests, even where an alternative Standard Water is recommended in the CIPAC method. Exceptions are tests of emulsion stability and dispersion stability where both Standard Waters A and D are to be used, since these properties may be affected by soft water as well as hard water.

[6] Preparation of Standard Waters A to D are described in CIPAC methods MT 18.1.1 to MT 18.1.4.

**The type of water used must be clearly indicated for all tests.**

**Test concentrations**

The test methods provide information on how to conduct the tests and may also give concentrations of product at which the tests should be conducted. In order to ensure that the product will perform acceptably when used in the field the concentration of the product tested for each technical property should relate to the recommended use rates given on the label, even where other concentrations are indicated in the test method, so long as the concentration used is within the scope of the method.

Where several use rates are recommended, the highest and lowest concentrations should be used so long as they are within the limitations of the test method, however some tests may not need to be conducted at both the high and low concentrations, where a clear "worst case" dilution can be identified. The following test concentrations have been agreed by MS:

## Recommended concentrations for use in physical-chemical property tests [7]

| Formulation types | Parameter | Concentration to be tested | Reasoning and comments |
|---|---|---|---|
| Water soluble products (SL) | Dilution stability | Highest | At lower concentrations it should be clear that solubility will be stable as events such as crystallisation are less likely to occur. |
| Preparations which form emulsions | Emulsion stability | Highest and lowest | Surfactant concentration changes, both high and low, can affect emulsification. |
| Preparations which form dispersions (e.g. SE) | Dispersion characteristics | Highest and lowest | Surfactant concentration changes, both high and low, can affect dispersion characteristics.<br>Practical difficulties when using the gravimetric methods should be considered when evaluating against existing criteria. |
| Preparations to be diluted with water | Persistent foam | Highest and Lowest | Changes in surface tension will be influenced by the concentrations. |
| Water dispersible products (WG, SC) | Spontaneity of dispersion | Highest concentration | The greater the ratio of product to water the more difficult it will be to disperse. |
| Water dispersible products (WP, WG, SC) | Suspensibility | Highest and lowest | Changes in viscosity and surface tension interact with each other. The absolute concentration of the dispersant will also affect the suspensibility. |
| WP, SG, WG | Wettability | Highest | The greater the ratio of solid to water the more difficult it will be to wet. |
| - | Dissolution of water soluble bags | Highest<br>(Highest in use concentration of bag within dilution volume.) | The greater the ratio of solid to water the more difficult it will be to disperse. |

[7] The table is based on the summary provided in the '*EFSA working document for the PRAPeR meeting of experts Section 1'* (2007). Use rates were proposed by experts during the PRAPeR 31 meeting and these were subsequently amended and accepted by the experts of PRAPeR 36

For seed treatments it is recognised that generally the concentrations at which seed treatments are applied to seeds significantly exceed those for field application of pesticides and hence many of the CIPAC tests are inappropriate. Therefore a justification may be submitted that certain tests are not required.

### 2.8.1 Wettability

**Method MT 53.3 Wetting of wettable powders**

Wettability is determined to ensure the preparation is readily wetted in use. The data are required for solid preparations which are to be dispersed in water.

The method as written describes the wetting of wettable powder preparations but it is also applicable to water soluble powders, water soluble granules and water dispersible granules.

| | |
|---|---|
| **Acceptable limits :-** | A preparation is considered acceptable if there is complete wetting in 1 minute without swirling. If the criterion is met without swirling then performance of the test with swirling is not required. |

Where a preparation is outside this limit then evidence must be submitted demonstrating acceptable wetting in the spray tank or other application equipment

**GLP – No**

### 2.8.2 Persistent foaming

**Method MT 47.3 Determination of the foaming of suspension concentrates**

Persistent foam is determined to measure the amount of foam likely to be present in a spray tank or other application equipment following dilution of the preparation.

Although MT 47.3 was standardised for the determination of persistent foam in suspension concentrates it is also applicable to other preparations which are dispersed in water.

| | |
|---|---|
| **Acceptable limits :-** | Max 60ml foam after 1 minute |

Where a preparation is outside these limits then evidence must be submitted showing that there is no unacceptable risk to operators following use of the preparation through the appropriate application equipment.

**GLP - No**

### 2.8.3 Suspensibility, spontaneity and dispersion stability

| | | |
|---|---|---|
| **Method** | **MT 184** | **Suspensibility of formulations forming suspensions on dilution with water** |
| | **MT 180** | **Dispersion stability of suspo-emulsions** |

Suspensibility/dispersion stability 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.

For the determination of suspensibility using CIPAC MT 184, chemical assay ('active' suspensibility) is the only fully reliable method to measure the mass of active substance still in suspension. However, gravimetric determination (total suspensibility) or solvent extraction determination may be used providing that these methods have been shown to give equivalent results to those of the chemical assay.

Where there is more than one insoluble active substance present in the preparation, chemical assay ('active' suspensibility) is the only acceptable method. Full validation data should be submitted to support the analytical method in accordance with SANCO/3029/99.

| | |
|---|---|
| **Acceptable limits :-** | MT 184 the mean measured active suspensibility must not be less than 60% or greater than 105%. |
| | MT 180 maximum 2ml cream after 30 minutes, trace of oil. If any separation observed, re-emulsification should be complete after 24 hours. |

Where a preparation is outside these limits then evidence must be submitted demonstrating that the preparation is homogeneous on application through appropriate application equipment e.g. determination of active substance content in the spray at the beginning, middle and end of a spraying operation at highest and lowest use rates on the label. Observations on any nozzle blockages should also be included.

**GLP – No**

### 2.8.3a Spontaneity of dispersion

| | | |
|---|---|---|
| **Method** | **MT 160** | **Spontaneity of dispersion of suspension concentrates** |
| | **MT 174** | **Dispersiblity of water dispersible granules** |

The spontaneity of dispersion is determined to show the preparation is rapidly dispersed when diluted with water.

As for the determination of suspensibility, chemical assay is the only reliable means to measure the mass of active substance in suspension.

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

Where there is more than one insoluble active substance present in the preparation, chemical assay is the only acceptable method. Full validation data should be submitted to support the analytical method in accordance with SANCO/3029/99.

| | |
|---|---|
| **Acceptable limits :-** | The mean measured minimum active spontaneity of dispersion or dispersibility must not be less than 60% or greater than 105%. |

Where a preparation is outside these limits then evidence must be submitted demonstrating that the preparation is homogeneous on application through appropriate application equipment with no blockages.

**GLP - No**

### 2.8.4 Degree of dissolution and dilution stability

| | | |
|---|---|---|
| **Method** | **MT 179.1** | **Dissolution degree and solution stability** |
| | **MT 41.1** | **Dilution stability of aqueous solutions** |
| | **MT 196** | **Solution properties of ST formulations** |

The scope of MT 179.1 states the method applies to water soluble granules only and MT 196 only applies to water soluble tablets.

The dilution stability is determined to ensure that water-soluble preparations dissolve readily and/or, when diluted, produce stable solutions without precipitation, flocculation, etc. The results submitted should fully describe the appearance and amount of any separation or sediment.

| | |
|---|---|
| **Acceptable limits :-** | MT 41.1 'trace' of sediment after 30 minutes – the amount and appearance of any sediment should be fully described.<br><br>If any material has separated after 24 h determine the amount of residue on a 75 µm sieve acording to a procedure adapted from MT 185 (b) Wet Sieving and the content of the a.i. in this residue.<br><br>MT 179.1 max 2% on 75µm sieve |
| Where a preparation is outside this limit then evidence must be submitted showing the material separated will not block application equipment or present an unacceptable risk to the operator or lead to unacceptable residues or crop safety concerns. | |

**GLP – no**

### 2.8.5 Particle size distribution, dust content, attrition and mechanical stability

#### 2.8.5.1 (a) Wet sieve test

**Method** **MT 185** **Wet sieve test**

A wet sieve test is required for water dispersible products. The residue remaining on a sieve is determined after dispersion to ensure no unacceptable residue remains which might cause the blockage of nozzles or filters on application equipment.

| | |
|---|---|
| **Acceptable limits :-** | maximum 2% retained on a 75 µm sieve.<br><br>Where a preparation is outside this limit then evidence must be submitted showing the preparation may be satisfactorily applied through appropriate application equipment with no blockages. |

**GLP – No**

#### 2.8.5.1 (b) Particle size distribution

**Method** **MT 170** **Dry sieve analysis of water dispersible granules**

**OECD 110** **Powders/dusts**

**MT 187** **Particle size analysis by laser diffraction**

**The use of MT 187 should be appropriate to the preparation type and in accordance with ISO 13320-1:1999(E).**

The nominal size range for solid materials for direct application and solid materials for dispersion in water must be determined.

For dustable powders using method MT 170: if > 5% of the preparation is retained on a 75 µm sieve, the active substance content of material remaining on the sieve must be determined to demonstrate there was no separation of the active substance from the carrier.

Where relevant the particle size of formulations classed as 'dusty' according to MT 171.1 must be determined using OECD method 110.

Where a formulation is classed as 'dusty' according to MT 171.1 (2.8.5.2) and/or where a significant proportion of particles (>1% by weight) have a diameter of <50 µm then inhalation toxicity data may be required.

| | | |
|---|---|---|
| **Acceptable limits for dustable powders** | MT 170 | maximum 5% retained on a 75 µm sieve.<br>Not more than (0.005 × a.s. content in g/kg) % should be present as the a.s. in the residue on the sieve. |

**GLP – Yes**

### 2.8.5.2 Dust content

**Method** **MT 171.1** **Dustiness of granular products**

**OECD 110** **powders/dusts**

The dust content of solid preparations must be determined to ensure there is no unacceptable risk to operators or bystanders or potential for blockage of application equipment.

MT 171.1 describes two methods for the determination of dustiness but the gravimetric method is regarded as the 'Referee' method. Where a preparation is described as 'dusty' under MT 171.1, data on inhalation toxicity may be required.

The dustiness categories for products (as stated in MT 171.1) are as follows:

| Category | Range of results | | Interpretation |
|---|---|---|---|
| | Gravimetric collected dust (mg) | Optical dust factor | |
| 1 | 0 – 12 | 0 – 10 | Nearly dust free |
| 2 | >12 – 30 | >10 – 25 | Essentially non dusty |
| 3 | >30 | >25 | Dusty |

| | |
|---|---|
| **Acceptable limits :-** | The amount of dust (as a %) must be stated. Where the apparent dust content is >1% (by weight), the implications for the potential risk to operators and bystanders must be addressed.<br>(1) For the impact on operators and bystanders the particle size and nature of the dust must be investigated. CIPAC Method MT 187 particle size by laser diffraction should be used to establish the particle size distribution. |

**GLP – Yes**

### 2.8.5.3 Attrition resistance

**Method** **MT 178** **Attrition resistance of granules (GR)**

**MT 178.2** **Attrition resistance of dispersible granules**

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

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

| | |
|---|---|
| **Acceptable limits :-** | Where the material has an attrition resistance of <98% then the particle size of the dust must be determined and the risk to operators and bystanders must be addressed.<br><br>For granules, where the material has an attrition resistance of <98% then evidence is required that the material may be satisfactorily applied through application equipment. |

**GLP – No**

### 2.8.5.4 Hardness and integrity

**Method** **MT 193** **Attrition of tablets**

CIPAC MT 193 method was originally titled “Friability of tablets”, however it was noted that the method determined the attrition of the tablets rather than the attrition resistance. The test is required in order to demonstrate that tablets do not break-up in the sales pack.

### 2.8.6 Emulsifiability, re-emulsifiability, emulsion stability

**Method MT 36.3 Emulsion characteristics and re-emulsification properties (0.1% - 5% dilution)**

The data are required to determine whether a preparation forms and maintains a stable emulsion.

MT 36.3 is designed to be conducted over a 24 hour period. However, if no separation of cream or oil is observed after 2 hours then no further testing is required. If separation is observed then the 24-hour test should be carried out.

| | |
|---|---|
| **Acceptable limits :-** | MT 36.3 maximum 2 ml cream after 30 minutes, trace of oil. If any separation observed re-emulsification should be complete after 24 hours. |

Where a preparation is outside these limits then evidence must be submitted showing the preparation remains homogeneous when applied through appropriate application equipment.

If more than a trace of oil separates consideration should be given to reformulation.

**GLP - No**

### 2.8.7 Flowability, pourability and dustability

### 2.8.7a Flowability

**Method** **MT 172.1** **Flowability of granular preparations after accelerated storage under pressure**

The data are required to demonstrate that granular materials remain free flowing after storage under pressure.

The method is not appropriate to those granules where water has been added as a formulant. For such granules, alternative data must be provided to demonstrate that the granular materials may be satisfactory applied through the application equipment.

| | |
|---|---|
| **Acceptable limits :-** | The sample should flow through the sieve after a maximum of 5 liftings. |

**GLP – No**

### 2.8.7b Pourability

**Method** **MT 148 OR MT 148.1** **Pourability of suspension concentrates**

**MT 148** **Pourability of suspension concentrates (includes rinsed residue)**

The data are required to demonstrate that the user can make use of the maximum amount of the preparation and that an excessive amount of the material does not remain in the container. The method is not suitable to address effectiveness of cleaning procedures. The method is appropriate to suspension concentrates, capsule suspensions, oil in water emulsions, and suspo-emulsions.

| | |
|---|---|
| **Acceptable limits :-** | Max. 5% residue<br><br>Where a preparation is outside these limits then evidence is required that the residue remaining in the commercial pack following recommended rinsing procedures is acceptable (if appropriate). In these circumstances an acceptable limit would be max 0.25% rinsed residue. |

**GLP – No**

### 2.8.7c Dustability

**Method MT 34 Dustability tests after tropical storage**

Reference is made to the use of MT 34. However, the equipment used in this method is not readily available. It is therefore acceptable for applicants to use their own equipment, provided that this is described and it is indicated that there is no unacceptable compaction or caking following a heat test under pressure.

Additionally, data are required showing the preparation may be satisfactorily applied as a dust through the proposed application equipment.

**GLP – No**

## 2.9 Physical compatibility with other products including plant protection products with which its use is to be authorized

**Tank mix compatibility**

Data on physical compatibility of the preparation are required where a positive recommendation is made for tank mixes or where it is indicated on the label that a mixture is compatible. The ASTM standard method is E 1518-05. If another test method is used then it must be justified.

Full study reports are required for a new active substance and its preparations. In all other cases a signed Compatibility Assurance Statement (CAS) may be sufficient.

Chemical compatibility must be supported by data[8] and/or reasoned scientific cases. These must demonstrate that the products can be used safely and efficaciously together in the proposed mixture. In the context of Part A section 2, data supporting chemical compatibility does not need to be submitted; instead regulators will accept a Compatibility Assurance Statement certifying that data or evidence is available to demonstrate that the products in the proposed mixture are physically and chemically compatible.

Information on the data requirements to support the biological compatibility of tank mixtures is available in Efficacy Guideline 604: Efficacy Data Required for Tank Mixtures and Sequences of Pesticides

[8] For example a theoretical consideration based on the chemical composition can be made to demonstrate that a reaction is impossible. Signs of a reaction, such as heat development, colour change or gas formation, should be monitored in the test for physical compatibility and report, if observed.

## 2.10 Adherence and distribution to seeds

The requirements for seed treatments include the determination of the physical and chemical properties of the preparation itself in addition to evidence that the preparation may be satisfactorily applied to the seed.

## 2.10a Loading and distribution of active substance

**Active substance loading**

Data are required to show a seed treatment preparation may be applied to give acceptable and uniform loading. The data will also support other areas of the risk assessment such as ecotoxicology, residues and efficacy.

The seed loading should be a determination of the active substance content of the seeds, and a validated method of analysis should be used to determine this. Where a preparation contains two active substances then data for both active substances must be submitted. Where a preparation contains more than two active substances, then loading results for two of the components may be acceptable. In this case, the active substances chosen must be representative of pesticide types present e.g. if one or more insecticides and fungicides are applied then the analysis must include one of each pesticide type.

Tests should be carried out on seed from each crop or crop group on which the preparation is proposed for use. Extrapolations may be accepted from one seed type to another on the basis of comparable seed size, shape and surface morphology.

Data from small scale laboratory machines are acceptable but because different seed treatment machines (both commercial and small scale) employ different mechanisms it is advisable to test a seed treatment using commercially available seed treatment methods. This is especially important should preliminary small scale testing show any suggestion of possible formulation/machinery problems. The data must be supported by a full description of all procedures used in the test.

**GLP – No**

## 2.10b Uniformity of distribution

**Method** **MT 175** **Determination of seed-to-seed uniformity of distribution for liquid seed treatment formulations**

The uniformity of distribution on treated seed may be determined using MT 175. This method is dependent on a dye being present in the preparation. Where a dye is not present then the uniformity of distribution may be determined by measurement of the active substance content on the seed using a validated method of analysis.

| | | |
|---|---|---|
| **Acceptable limits :-** | seed loading | 70% of target dose |
| | uniformity, seed-to-seed | % RSD ±25% |

Where the loading and distribution are outside these limits then evidence must be submitted showing the treatment is efficacious. This may be done by reference to efficacy data.

**GLP – No**

## 2.10c Adhesion

**Method** **MT 194** **Adhesion to treated seeds**

**European Seed Association, 2011. Assessment of free floating dust and abrasion particles of treated seeds as a parameter of the quality of treated seeds: Heubach test. ESA STAT Dust Working Group.**

The method MT 194 determines the amount of product retained on treated seed after the seed falls from a funnel for a set distance. The method is not designed to classify or reliably quantify the part of the seed treatment product that is not retained on the seeds. The method is applicable to cereals, maize and sunflower seeds. It applicability to other seed types must be carefully assessed and justified.

The Heubach test involves passing air through a horizontal rotating drum containing a weighed amount of seed at a controlled flow rate over a fixed period of time. The dust generated is collected on a pre-weighed filter paper, and re-weighed to determine the amount.

Either CIPAC MT 194 or the Heubach tests are considered acceptable.

**GLP - No**

### 2.10d Storage of treated seed

There is no requirement to submit data to support stability/retention of the active substance on the stored seeds.

If no data are provided then the following statement is added to the label:

*Sowing treated seed that has been stored for prolonged periods (beyond the season of treatment) may adversely affect effectiveness and/or crop safety*

The fact that retention of the active substance is not a data requirement does not preclude companies from choosing to support a specific label claim concerning the period of time treated seed may be stored before sowing. In such instances, data should be generated as described in CRD Efficacy Guideline 208 (Seed Treatments – Efficacy and physical/mechanical data requirements) with the storage period in any studies reflecting the length of time proposed on the label.

**GLP – No**

## 2.11 Other studies

### 2.11a Packaging (type materials, size etc), compatibility of the preparation with proposed packaging materials.

**General requirements**

The packaging shall be designed in order to limit as much as possible exposure of operators and of the environment.[9]

The packaging shall be designed and constructed so that its contents cannot escape (unless special safety devices have been prescribed); the materials constituting the packaging and fastenings must not be susceptible to attack by the contents, or liable to form harmful or dangerous compounds with the contents; the packaging and fastenings must be strong and solid throughout so as to ensure that they will not come apart and will safely withstand normal handling; containers with fastening devices must be so designed that the container can be repeatedly refastened so that the contents cannot escape.

Full details of packaging must be included. This must include:

a) materials and manner of construction (e.g. extruded, welded, flexible, rigid)
b) barrier material
c) details of closures and seals
d) minimum and maximum container size and capacity
e) minimum wall thickness
f) neck size
g) details of any application device included with packaging
h) whether the container is refillable/returnable
i) outer packaging/sales pack
j) any other features e.g. pressure release

A statement must be provided that all packaging used complies with all relevant EU legislation on transportation and safe handling.

Following storage of the packaging any changes such as panelling, ballooning, condition of seals and seams and weight change must be reported in detail.

[9] Guidelines for the Packaging and Storage of Pesticides (FAO, Rome 1985)

**Extrapolation of packaging types**

As a general principle data in the form of ambient storage data are required to support each packaging type in which a plant protection product is authorised, although in certain instances it is possible to extrapolate data supporting one packaging type to another.

The following packaging extrapolations are acceptable:

**Rigid containers for liquid preparations**

For aqueous based formulation types e.g. SL and SC extrapolation between any plastic material types is acceptable. Extrapolation from plastic material to metals is not acceptable.

For organic solvent containing formulations e.g. EC, EW, SE extrapolation from HDPE to HDPE co-extruded with any of the following:- EVOH, fluorinated HDPE and polyamide is acceptable.

Extrapolation between plastic material types e.g. HDPE to PET is not acceptable.

Permitted extrapolations are summarised below:

| **Packaging used in shelf life study** | **Acceptable extrapolations** |
|---|---|
| **Water based formulations e.g aqueous suspension concentrates, soluble concentrates** | |
| Any, except metal | All packaging types, apart from metal are supported with no further data |
| **Organic Solvent based formulations e.g. emulsifiable concentrates** | |
| HDPE | HDPE/EVOH, HDPE/F, HDPE/PA packs would all be supported without further data |
| HDPE/EVOH or<br>HDPE/F or<br>HDPE/PA | Data generated in one of these three packaging types will support authorisation in the other two types with acceptable seepage data in the required packaging.<br>HDPE packs would be supported with acceptable seepage data*. |

*Seepage data
Data are only required to demonstrate that the required packaging is stable for the required shelf life (e.g. no leakage, no ballooning, no panelling of the packaging, no deformations) rather than a new shelf life study in which all chemical and physical properties are investigated prior to and after storage. The weight change on storage should also be determined.

**Flexible containers for powders and granules**

Extrapolation is possible between all container types (with the exception of water-soluble packaging, e.g. PVA packs). The material used must be waterproofed or have a waterproof lining.

When authorisation is sought for a flexible container, the issue of compaction or loss of granule integrity potentially caused by stacking containers in a pile such as in a store or warehouse must be addressed.

**Bulk containers**

Where it is proposed that a liquid preparation is to be packaged in a bulk container (a container of size greater than 20 litres), it is recognised that it is impractical to conduct stability tests in the large containers. Therefore results from smaller volume containers may be used to extrapolate to the larger containers.

Providing the container is constructed from the same material, results from a 20 litre container may be used to extrapolate to bulk containers.

However full details must be provided of the means of mixing the preparation in the larger container before use (if required).

For solid preparations data may be extrapolated from a smaller sample size, providing the sample is stored under pressure equivalent to the maximum that will be encountered in the bulk container. However in this case both containers must have similar mechanical properties i.e. both rigid containers or both flexible containers.

**Water soluble packaging**

The packaging of preparations in water soluble bags/sachets may have an effect on the physical characteristics of the preparation and vice versa. Therefore, when seeking authorisation for such packaging, the relevant physical tests for that preparation type must be carried out in the presence of the soluble bag material in the same ratio(s) as that which will occur in the spray tank or other application equipment.

In addition MT 176 must be carried out to determine the dissolution rate of the water soluble bag.

| | |
|---|---|
| **Acceptable limits :-** | The dissolution time should not exceed 30 seconds. |
| | Where a preparation is outside this limit then a practical sprayability test showing acceptable dissolution in a spray tank must be performed. |

**GLP - No**

When conducting storage stability tests with water soluble bags it is considered that storage under ambient temperature conditions is preferable to storage at elevated temperatures. There have been cases where the physical and chemical properties of the preparation have been such that the bag material has deteriorated when stored and this may not have been observed under short term, elevated temperature testing. Consideration should also be given to the effects of low temperature storage on water soluble packaging.

**Multiple bags in a container**

Where multiple water soluble 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 re-sealing of the outer pack or by moisture entering through routine use. Therefore the following data are required:

**a) Ambient testing – short term studies (only required if long term studies are unavailable)**

Evidence must be submitted to demonstrate that the integrity and performance of the water soluble sachets/bags is unaffected by the repeated opening and resealing of the outer pack, under recommended conditions of storage and use. This may be achieved by storing a 'multi-bag' pack at ambient temperatures over a 6 month period and periodically removing the water soluble sachets/bags from the outer pack under normal handling and use conditions, until all the bags have been removed. On removing the water soluble sachets/bags, the integrity of each of the bags must be examined and the 'dissolution' characteristics of the water soluble sachets/bags tested (using MT 176).

**b) Ambient testing long term studies**

Ambient testing may be achieved by storing a 'multi-bag' pack at ambient temperatures over a 2 year period and periodically removing the water soluble sachets/bags from the outer pack under normal handling and use conditions, until all the bags have been removed. On removing the water soluble sachets/bags, the integrity of each of the bags must be examined and the 'dissolution' characteristics of the water soluble sachets/bags tested (using MT 176).

**Trigger packs or hand held sprayers**

Evidence must be provided that the preparation may be satisfactorily applied throughout the use of the pack and that this is maintained on storage (e.g. demonstration that no nozzle blockage occurs on storage). This can be achieved by demonstrating that the quantity of spray dispensed is consistent for multiple spray actions and this is maintained after storage (note: the same individual trigger pack(s) should be used to generate the data at each of the time points tested and the determination of the quantity of spray following intermittant storage periods throughout the two year period should be recorded, not just the determination prior to and following 2 years storage). In addition observations of any build-up of crystallised material in the nozzles, nozzle blockage or leaks from the trigger head should be provided. Evidence is also required on the spray pattern before and after storage and at intermittant time periods during the 2 year storage. If multiple types of trigger packs are requested with differing trigger mechanisms, then data generated to support each mechanism type will be required.

**Re-usable/refillable packaging**

If a container is re-useable it is the responsibility of the container owner when containers are returned to ensure that the package continues to be fit for purpose, and still complies with all relevant transport requirements.

## 4.2 Effectivness of cleaning procedures

Regulation (EC) No. 284/2013 Paragraph 4.2 states that the effectiveness of the recommended tank cleaning procedures must be addressed. Data submitted must demonstrate that residues of the plant protection product do not remain in the spray tank after cleaning such that there is a risk to the operator or crops. The method used must be fully reported and justified; no standard agreed method is available at present; however CRD Efficacy Guideline 305 may be appropriate.

## Appendix 1 Requirements for the technical characteristics of the plant protection product

The technical characteristics have been arranged according to the preparation type. Those properties which must be determined before and after storage are indicated. The properties required are in accordance with the Regulation (EC) No 1107/2009 and the Manual for the Development and Use of FAO and WHO Specifications for Plant Protection Products.

Where a test is not considered applicable to a particular preparation then this must be explained and justified.

Where information is not given on a specific formulation then a logical approach should be taken addressing the appropriate properties and using the relevant tests. This also applies to mixed formulation e.g. ZC, ZE and ZW.

# 1 Common Liquid preparations

## 1.1 Soluble concentrate (SL)

A clear to opalescent liquid to be applied as a solution of the active ingredient after dilution in water. The liquid may contain water-insoluble formulants.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include whether any separation or crystallisation occurs. |
| **2.2** | Explosive properties | A 14, UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21, Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity (if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or OECD 115 | not required after storage | |
| **2.6** | Relative density | A 3 or OECD 109 | not required after storage | |
| **2.7** | Low temperature stability | MT 39.3 | | |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.4** | Dilution stability | MT 41.1 | data required before and after storage | |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required for any tank mixes recommended on the label. |
| **2.11** | Stability of packaging and packaging/preparation interactions | | data required before and after storage | Observation of pack stability including weight change. |

## 1.2 Suspension concentrates (SC)

A stable suspension of active ingredient(s) with water as the fluid, intended for dilution with water before use.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include whether any separation or sedimentation/claying occurs. Comment on re-dispersibility. |
| **2.2** | Explosive properties | A 14, UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or OECD 115 | not required after storage | |
| **2.6** | Relative density | A 3 or OECD 109 | not required after storage | |
| **2.7** | Low temperature stability | MT 39.3 | Suspensibility and wet sieve test should be determined after storage | |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.3** | Suspensibility | MT 184 | data required before and after storage | |
| **2.8.3** | Spontaneity of dispersion | MT 160 | data required before and after storage | |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | |
| **2.8.7** | Pourability | MT 148 OR MT 148.1 | data required before and after storage | |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | | data required before and after storage | Observation of pack stability including weight change. |

## 1.3 Capsule suspensions (CS)

A stable suspension of capsules in a fluid, normally intended for dilution with water before use.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | To include the determination of 'free' and 'encapsulated' active substance if required.*<br><br>For controlled release capsules the release rate must be determined. Reference may be made to biological efficacy data.[10] |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include whether any separation or sedimentation/claying occurs. Comment on re-dispersibility |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or OECD 115 | not required after storage | |
| **2.6** | Relative density | A 3 or OECD 109 | not required after storage | |

[10] Refer to section 1.4.1 (page 6) for further details.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **2.7** | Low temperature stability | | The low temperature stability testing should be carried out under a 'freeze/thaw' cycle to demonstrate that capsule integrity is not adversely affected.<br>Unless otherwise agreed, the freeze/thaw stability test shall cycle the formulation between room temperature (e.g. 20 ± 2°C) and -10 ± 2°C on 18-hour-freeze/6-hour-melt cycles for a total of 4 cycles.<br>See FAO manual Pg 157, note 12 for further details. | 'Free' and 'encapsulated' active substance should be determined before and after storage.*<br><br>Acidity/alkalinity/pH, pourability, spontaneity of dispersion, suspensibility and wet sieve test should be determined after storage. |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.3** | Suspensibility | MT 184 | data required before and after storage | |
| **2.8.3** | Spontaneity of dispersion | MT 160 | data required before and after storage | |
| **2.8.5.1** | Particle size distribution | OECD 100 or MT 187 | not required after storage | Use of MT 187 should be appropriate to the preparation type and in accordance with ISO 13320-1:1999(E) |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | |
| **2.8.7** | Pourability | MT 148 OR MT 148.1 | data required before and after storage | |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | | data required before and after storage | Observation of pack stability including weight change. |

* Determination of free active ingredient is usually required where encapsulation is intended to control the release or stability of the active ingredient to address volatility issues and potential impact on non-target crops or to decrease the risk to users from accidental exposure to the active ingredient.

## 1.4 Emulsifiable concentrate (EC)

A liquid, homogeneous formulation to be applied as an emulsion after dilution in water.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include whether any separation occurs. Comment on ease of re-homogenisation |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or OECD 115 | not required after storage | |
| **2.6** | Relative density | A 3 or OECD 109 | not required after storage | |
| **2.7** | Low temperature stability | MT 39.3 | Emulsion stability should be determined after storage | |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.6** | Emulsifiability<br>Re-emulsifiability<br>Emulsion stability | MT 36.3 | data required before and after storage | In MT 36.3: where no oil or cream separation after 2 hours, then the 24 hour test is not required |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | | | Observation of pack stability including weight change. Indicate if there is any seepage of solvent through the container walls or seal. |

## 1.5 Suspo-emulsions (SE)

A fluid, heterogeneous formulation consisting of a stable dispersion of active ingredients in the form of solid particles and fine globules in a continuous water phase.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include whether any separation occurs. Comment on ease of re-homogenisation |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or OECD 115 | not required after storage | |
| **2.6** | Relative density | A 3or OECD 115 | not required after storage | |
| **2.7** | Low temperature stability | MT 39.3 | pH, dispersion stability and wet sieve should be determined after storage. | |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.3** | Dispersion stability | MT 180 | data required before and after storage | |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | |
| **2.8.7** | Pourability | MT 148 OR MT 148.1 | data required before and after storage | |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | | data required before and after storage | Observation of pack stability including weight change. Indicate if there is any seepage of solvent through the container walls or seal. |

## 1.6 Dispersible concentrate (DC)

A liquid homogeneous formulation to be applied as a solid dispersion after dilution in water. (Note: there are some formulations which have characteristics intermediate between DC and EC).

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include whether any separation or sedimentation/claying occurs. Comment on re-dispersibility. |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or OECD 115 | not required after storage | |
| **2.6** | Relative density | A 3 or OECD 109 | not required after storage | |
| **2.7** | Low temperature stability | MT 39.3 | pH and dispersion stability should be determined after storage | |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.3** | Dispersion stability | MT 180 | data required before and after storage | |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | | data required before and after storage | Observation of pack stability including weight change. |

## 1.7 Oil-in-water emulsions (EW) and micro emulsions (ME)

EW - A fluid, heterogeneous formulation consisting of a solution of pesticide in an organic liquid dispersed as fine globules in a continuous water phase.

ME - A clear to opalescent, oil and water containing liquid, to be applied directly or after dilution in water, when it may form a diluted micro-emulsion or a conventional emulsion.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include whether any separation occurs. Comment on ease of re-homogenisation |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or OECD 115 | not required after storage | |
| **2.6** | Relative density | A 3 or OECD 109 | not required after storage | |
| **2.7** | Low temperature stability | MT 39.3 | Emulsion stability should be determined after storage | |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.6** | Emulsifiability<br>Re-emulsifiability<br>Emulsion stability | MT 36.3 | data required before and after storage | In MT 36.3: where no oil or cream separation after 2 hours, then the 24 hour test is not required |
| **2.8.7** | Pourability | MT 148 OR MT 148.1 | data required before and after storage | for EWs only |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | | | Observation of pack stability including weight change. Indicate if there is any seepage of solvent through the container walls or seal. |

## 1.8 Oil Dispersion (OD)

A stable suspension of active ingredient(s) in a water- immiscible fluid, which may contain other dissolved active ingredient(s), intended for dilution with water before use.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include whether any separation occurs. Comment on ease of re-homogenisation |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or OECD 115 | not required after storage | |
| **2.6** | Relative density | A 3 or OECD 109 | not required after storage | |
| **2.7** | Low temperature stability | MT 39.3 | separated material should not be greater than 0.3 ml | |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.3** | Dispersion stability | MT 180 | data required before and after storage | |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | |
| **2.8.5.1** | Particle size distribution | MT 187 | data required before and after storage | |
| **2.8.7** | Pourability | MT 148 OR MT 148.1 | data required before and after storage | |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | | data required before and after storage | Observation of pack stability including weight change. Indicate if there is any separation or seepage through the container walls or seal. |

## 1.9 Ultra low volume preparations (UL)

Ultra low volume preparations are generally designed for application through special equipment. Hence the physical characteristics of the preparation are also dependent on the type of equipment recommended.

In addition to the characteristics described below, the potential for loss of droplet mass is critical for UL preparations as this can affect spray drift during application. For this reason determination of droplet size is required both before and after storage. In determining the stability of the preparation, either the physical characteristics such as viscosity or droplet size may be determined or evidence must be provided that the preparation may be satisfactorily applied through the recommended application equipment.

| **Annex point** | **Property** | **Test Method** | **Storage stability requirements** | **Comments** |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or OECD 115 | not required after storage | |
| **2.6** | Relative density | A 3 or OECD 109 | not required after storage | |
| **2.7** | Low temperature stability | MT 39.3 | | |
| **2.8.5.1** | Wet sieve test | MT 185 | data required before and after storage | Only applicable to water dispersible products |
| **2.11** | Stability of packaging and packaging/preparation interactions | | data required before and after storage | Observation of pack stability including any evidence of seepage. Record any weight change during storage |
| **-** | Droplet size | appropriate validated method | data required before and after storage | |

## 1.10 Oil miscible liquids (OL)

A liquid, homogeneous formulation to be applied as a homogeneous liquid after dilution in an organic liquid.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or OECD 115 | not required after storage | |
| **2.6** | Relative density | A 3 or OECD 109 | not required after storage | |
| **2.7** | Low temperature stability | MT 39.3 | | |
| **2.11** | Stability of packaging and packaging/preparation interactions | | data required before and after storage | Observation of pack stability including any evidence of seepage. Record any weight change during storage |
| - | Miscibility with hydrocarbon oil | MT 23 | data required before and after storage | The formulation shall be miscible with the appropriate hydrocarbon oil |

## 1.11 Mixed formulations of CS and SC (ZC)

A stable suspension of capsules and active ingredient(s) in fluid, normally intended for dilution with water before use.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | To include the determination of 'free' and 'encapsulated' active substance if required.*<br><br>For controlled release capsules the release rate must be determined. Reference may be made to biological efficacy data[11]. |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include whether any separation or sedimentation/claying occurs. Comment on re-dispersibility |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or OECD 115 | not required after storage | |
| **2.6** | Relative density | A 3 or OECD 109 | not required after storage | |

[11] Refer to section 1.4.1 (page 6) for further details.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **2.7** | Low temperature stability | | The low temperature stability testing should be carried out under a 'freeze/thaw' cycle to demonstrate that capsule integrity is not adversely affected.<br>Unless otherwise agreed, the freeze/thaw stability test shall cycle the formulation between room temperature (e.g. 20 ± 2°C) and -10 ± 2°C on 18-hour-freeze/6-hour-melt cycles for a total of 4 cycles.<br>See FAO manual Pg 169, note 12 for further details. | 'Free' and 'encapsulated' active substance should be determined before and after storage.*<br><br>Acidity/alkalinity/pH, pourability, spontaneity of dispersion, suspensibility and wet sieve test should be determined after storage. |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.3** | Suspensibility | MT 184 | data required before and after storage | |
| **2.8.3** | Spontaneity of dispersion | MT 160 | data required before and after storage | |
| **2.8.5.1** | Particle size distribution | OECD 100 or MT 187 | not required after storage | Use of MT 187 should be appropriate to the preparation type and in accordance with ISO 13320-1:1999(E) |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | |
| **2.8.7** | Pourability | MT 148 OR MT 148.1 | data required before and after storage | |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | | data required before and after storage | Observation of pack stability including weight change. |

* Determination of free active ingredient is usually required where encapsulation is intended to control the release or stability of the active ingredient to address volatility issues and potential impact on non-target crops or to decrease the risk to users from accidental exposure to the active ingredient.

## 1.12 Mixed formulations of CS and EW (ZW)

A fluid, heterogeneous formulation consisting of a stable dispersion of active ingredient(s) in the form of capsules and fine globules in a continuous water phase, normally intended for dilution with water before use.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | To include the determination of 'free' and 'encapsulated' active substance if required.*<br><br>For controlled release capsules the release rate must be determined. Reference may be made to biological efficacy data.[12] |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include whether any separation or sedimentation/ claying occurs. Comment on re-dispersibility. |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or OECD 115 | not required after storage | |
| **2.6** | Relative density | A 3 or OECD 109 | not required after storage | |

[12] Refer to section 1.4.1 (page 6) for further details.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **2.7** | Low temperature stability | | The low temperature stability testing should be carried out under a 'freeze/thaw' cycle to demonstrate that capsule integrity is not adversely affected. Unless otherwise agreed, the freeze/thaw stability test shall cycle the formulation between room temperature (e.g. 20 ± 2°C) and -10 ± 2°C on 18-hour-freeze/6-hour-melt cycles for a total of 4 cycles. See FAO manual Pg 174, note 11 for further details. | 'Free' and 'encapsulated' active substance should be determined before and after storage.*<br><br>Acidity/alkalinity/pH, pourability, spontaneity of dispersion, suspensibility and wet sieve test should be determined after storage. |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.3** | Dispersion stability | MT 180 | data required before and after storage | |
| **2.8.5.1** | Particle size distribution | OECD 100 or MT 187 | not required after storage | Use of MT 187 should be appropriate to the preparation type and in accordance with ISO 13320-1:1999(E) |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | |
| **2.8.7** | Pourability | MT 148 OR MT 148.1 | data required before and after storage | |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | | data required before and after storage | Observation of pack stability including weight change. |

* Determination of free active ingredient is usually required where encapsulation is intended to control the release or stability of the active ingredient to address volatility issues and potential impact on non-target crops or to decrease the risk to users from accidental exposure to the active ingredient.

## 1.13 Mixed formulations of CS and SE (ZE)

A fluid, heterogeneous formulation consisting of a stable dispersion of active ingredient(s) in the form of capsules, solid particles, and fine globules in a continuous water phase, normally intended for dilution with water before use.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | To include the determination of 'free' and 'encapsulated' active substance if required.*<br><br>For controlled release capsules the release rate must be determined. Reference may be made to biological efficacy data. [13] |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include whether any separation or sedimentation/ claying occurs. Comment on re-dispersibility |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or OECD 115 | not required after storage | |
| **2.6** | Relative density | A 3 or OECD 109 | not required after storage | |

[13] Refer to section 1.4.1 (page 6) for further details.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **2.7** | Low temperature stability | | The low temperature stability testing should be carried out under a 'freeze/thaw' cycle to demonstrate that capsule integrity is not adversely affected.<br>Unless otherwise agreed, the freeze/thaw stability test shall cycle the formulation between room temperature (e.g. 20 ± 2°C) and -10 ± 2°C on 18-hour-freeze/6-hour-melt cycles for a total of 4 cycles.<br>See FAO manual Pg 180, note 11 for further details. | 'Free' and 'encapsulated' active substance should be determined before and after storage.*<br><br>Acidity/alkalinity/pH, pourability, spontaneity of dispersion, suspensibility and wet sieve test should be determined after storage. |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.3** | Dispersion stability | MT 180 | data required before and after storage | |
| **2.8.5.1** | Particle size distribution | OECD 100 or MT 187 | not required after storage | Use of MT 187 should be appropriate to the preparation type and in accordance with ISO 13320-1:1999(E) |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | |
| **2.8.7** | Pourability | MT 148 OR MT 148.1 | data required before and after storage | |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | | data required before and after storage | Observation of pack stability including weight change. |

* Determination of free active ingredient is usually required where encapsulation is intended to control the release or stability of the active ingredient to address volatility issues and potential impact on non-target crops or to decrease the risk to users from accidental exposure to the active ingredient.

## 2 Common Solid preparations

### 2.1 Granules (GR)

A free-flowing solid formulation of a defined granule size range ready for use.

The guidelines for granules are intended only to cover those preparations that are designed to be applied in dry form by mechanical means.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br><br>physical state<br>colour | | data required before and after storage | Observations of granule integrity are required before and after storage |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A16<br>Or<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.5.1** | Dry sieve test | MT 170 | data required before and after storage | |
| **2.8.5.2** | Dust content | MT 171.1<br>OECD 110 | data required before and after storage | If results show > 1% w/w dust then the particle size of the dust generated shall be determined in accordance with MT 187. |
| **2.8.5.3** | Attrition | MT 178 | data required before and after storage | |
| **2.8.7** | Flowability | MT 172.1 | Determined after accelerated storage under pressure in accordance with the method. | |
| **2.11** | Stability of packaging and packaging/preparation interactions | | | Observation of pack stability including weight change.<br>There should be no loss of granule integrity or caking on storage.<br>For flexible packs the effect of stacking packs should be determined after storagethrough conducting the ambient storage stability study under pressures encountered in commercial practice. |

## 2.2 Water dispersible granules (WG)

A formulation consisting of granules to be applied after disintegration and dispersion in water.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations of granule integrity are required before and after storage |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.1** | Wettability | MT 53.3 | data required before and after storage | WSB |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | WSB |
| **2.8.3** | Suspensibility | MT 184 | data required before and after storage | WSB |
| **2.8.3** | Spontaneity of dispersion | MT 174 | data required before and after storage | WSB |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | WSB |
| **2.8.5.1** | Dry sieve test | MT 170 | data required before and after storage | |
| **2.8.5.2** | Dust content | MT 171.1 | data required before and after storage | If results show > 1% w/w dust then the particle size of the dust generated shall be determined in accordance with MT 187. |
| **2.8.5.3** | Attrition | MT 178.2 | data required before and after storage | |
| **2.8.7** | Flowability | MT 172.1 | Determined after accelerated storage under pressure in accordance with the method. | |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | Observation of pack stability including weight change.<br>There should be no loss of granule integrity or caking.<br>For flexible packs the effect of stacking packs should be determined after storagethrough conducting the ambient storage stability study under pressures encountered in commercial practice. | | |

**WSB:** Where the preparation is packaged in a water soluble bag then the wet sieve, persistent foam, wettability and solution/dilution stability tests must be carried out using the preparation and the water soluble bag in the same ratio as in the recommended application. In addition the dissolution rate of water soluble bags according to MT 176 should be determined

## 2.3 Water soluble granules (SG)

A formulation consisting of granules to be applied as a true solution of the active ingredient after dissolution in water, but which may contain insoluble inert ingredients.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations of granule integrity are required before and after storage |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.1** | Wettability | MT 53.3 | data required before and after storage | WSB |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | WSB |
| **2.8.4** | Dilution stability | MT 179.1 | data required before and after storage | WSB |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | WSB |
| **2.8.5.1** | Dry sieve test | MT 170 | data required before and after storage | |
| **2.8.5.2** | Dust content | MT 171.1 | data required before and after storage | If results show > 1% w/w dust then the particle size of the dust generated shall be determined in accordance with MT 187. |
| **2.8.5.3** | Attrition | MT 178.2 | data required before and after storage | |
| **2.8.7** | Flowability | MT 172.1 | Determined after accelerated storage under pressure in accordance with the method. | |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **2.11** | Stability of packaging and packaging/preparation interactions | Observation of pack stability including weight change.<br>There should be no loss of granule integrity or caking.<br>For flexible packs the effect of stacking packs should be determined after storage through conducting the ambient storage stability study under pressures encountered in commercial practice. | | |

**WSB:** Where the preparation is packaged in a water soluble bag then the wet sieve, persistent foam, wettability and solution/dilution stability tests must be carried out using the preparation and the water soluble bag in the same ratio as in the recommended application. In addition the dissolution rate of water soluble bags according to MT 176 should be determined.

## 2.4 Emulsifiable granule (EG)

A granular formulation, which may contain water-insoluble formulants, to be applied as an oil-in-water emulsion of the active ingredient(s) after disintegration in water.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations of granule integrity are required before and after storage |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.1** | Wettability | MT 53.3 | data required before and after storage | WSB |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | WSB |
| **2.8.4** | Dispersion stability | MT 180 | data required before and after storage | WSB |
| **2.8.5.1** | Wet sieve | MT185 | data required before and after storage | WSB |
| **2.8.5.1** | Dry sieve test | MT 170 | data required before and after storage | |
| **2.8.5.2** | Dust content | MT 171.1 | data required before and after storage | If results show > 1% w/w dust then the particle size of the dust generated shall be determined in accordance with MT 187. |
| **2.8.5.3** | Attrition | MT 178.2 | data required before and after storage | |
| **2.8.6** | Emulsifiability<br>Re-emulsifiability<br>Emulsion stability | MT 36.3 | data required before and after storage | In MT 36.3: where no oil or cream separation after 2 hours, then the 24 hour test is not required |
| **2.8.7** | Flowability | MT 172.1 | Determined after accelerated storage under pressure in accordance with the method. | |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **2.11** | Stability of packaging and packaging/preparation interactions | | Observation of pack stability including weight change. There should be no loss of granule integrity or caking. For flexible packs the effect of stacking packs should be determined after storage through conducting the ambient storage stability study under pressures encountered in commercial practice. | |

**WSB:** Where the preparation is packaged in a water soluble bag then the wet sieve, persistent foam, wettability and dispersion/emulsion stability tests must be carried out using the preparation and the water soluble bag in the same ratio as in the recommended application. In addition the dissolution rate of water soluble bags according to MT 176 should be determined.

## 2.5 Wettable powder (WP)

A powder formulation to be applied as a suspension after dispersion in water.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.1** | Wettability | MT 53.3 | data required before and after storage | WSB |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | WSB |
| **2.8.3** | Suspensibility | MT 184 | data required before and after storage | WSB |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | WSB |
| **2.8.5.2** | Dust content | MT 171.1 | data required before and after storage | If results show > 1% w/w dust then the particle size of the dust generated shall be determined in accordance with MT 187. |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | Observations of pack stability including weight change and comment whether any caking or compaction has occurred on storage.<br>For flexible packs the effect of stacking packs should be determined after storage through conducting the ambient storage stability study under pressures encountered in commercial practice. | | |

**WSB:** Where the preparation is packaged in a water soluble bag then the wet sieve, persistent foam, wettability and suspensibility tests must be carried out using the preparation and the water soluble bag in the same ratio as in the recommended application. In addition the dissolution rate of water soluble bags according to MT 176 should be determined

## 2.6 Water soluble powders (SP)

A powder formulation to be applied as a true solution of the active ingredient after dissolution in water, but which may contain insoluble inert ingredients.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.1** | Wettability | MT 53.3 | data required before and after storage | WSB |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | WSB |
| **2.8.4** | Dilution stability | MT 179.1 | data required before and after storage | WSB |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | WSB |
| **2.8.5.2** | Dust content | MT 171.1 | data required before and after storage | If results show > 1% w/w dust then the particle size of the dust generated shall be determined in accordance with MT 187. |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | Observations of pack stability including weight change and comment whether any caking or compaction has occurred on storage.<br>For flexible packs the effect of stacking packs should be determined after storage through conducting the ambient storage stability study under pressures encountered in commercial practice. | | |

**WSB:** Where the preparation is packaged in a water soluble bag then the wet sieve, wettability, solution/dilution stability and persistent foam tests must be carried out using the preparation and the water soluble bag in the same ratio as in the recommended application. In addition the dissolution rate of water soluble bags according to MT 176 should be determined.

## 2.7 Dustable powders (DP)

A free-flowing powder suitable for dusting.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.5.1** | Particle size distribution | OECD 100 or MT 187 | data required before and after storage | Use of MT 187 should be appropriate to the preparation type and in accordance with ISO 13320-1:1999(E) |
| **2.8.5.1** | Dry sieve test | MT 170 | data required before and after storage | |
| **2.8.5.2** | Dust content | MT 171.1 | data required before and after storage | If results show > 1% w/w dust then the particle size of the dust generated shall be determined in accordance with MT 187. |
| **2.8.7** | Dustability | appropriate method | data required before and after storage | Evidence that the preparation may be satisfactorily applied through the equipment or from container specified. |
| **2.11** | Stability of packaging and packaging/preparation interactions | Observations of pack stability including weight change and comment whether any caking or compaction has occurred on storage.<br>For flexible packs the effect of stacking packs should be determined after storage through conducting the ambient storage stability study under pressures encountered in commercial practice. | | |

## 2.8 Tablets for direct application (DT)

Tablets for direct application (DT) are intended for application directly in the field and/or bodies of water without preparation of a spraying solution or dispersion.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include tablet integrity |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.8.5.2** | Dust content | MT 171.1 | data required before and after storage | If results show > 1% w/w dust then the particle size of the dust generated shall be determined in accordance with MT 187. |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.5.3** | Attrition | MT 193 | data required before and after storage | |
| **2.8.5.4** | Hardness and integrity | | data required before and after storage | |
| **2.11** | Stability of packaging and packaging/preparation interactions | | observation of pack stability | |

## 2.9 Water dispersible tablets (WT)

Water dispersible tablets (WT) are intended for application after disintegration and dispersion in water by conventional spraying equipment.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include tablet integrity |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A 10, A 16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | Not applicable to effervescent tablets |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | WSB |
| **2.8.3** | Suspensibility | MT 184 | data required before and after storage | WSB |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | WSB |
| **2.8.5.2** | Dust content | MT 171.1 | data required before and after storage | If results show > 1% w/w dust then the particle size of the dust generated shall be determined in accordance with MT 187. |
| **2.8.5.3** | Attrition | MT 193 | data required before and after storage | |
| **2.8.5.4** | Hardness and integrity | | data required before and after storage | |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **2.11** | Stability of packaging and packaging/preparation interactions | Observation of pack stability including weight change | | |
| | Disintegration time | No agreed method[14] | data required before and after storage | WSB. The data should demonstrate the tablet disintegrates rapidly on addition to water and that the formulation is readily dispersed. |

**WSB:** Where the preparation is packaged in a water soluble bag then the wet sieve, persistent foam and suspensibility tests must be carried out using the preparation and the water soluble bag in the same ratio as in the recommended application. In addition the dissolution rate of water soluble bags according to MT 176 should be determined.

[14] The data should demonstrate the tablet disintegrates rapidly on addition to water and that the formulation is readily dispersed and no blockages occur in the application equipment on use. A maximum disintegration time is to be specified. If continuous agitation is required, this should be specified on the instructions for use/label.

## 2.10 Water soluble tablets (ST)

Water soluble tablets (ST) are intended for application after dissolution in water by conventional spraying equipment. STs contain an active substance which is totally soluble in water at use rate concentrations.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include tablet integrity |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191 | data required before and after storage | Not applicable to effervescent tablets |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | WSB |
| **2.8.4** | Degree of dissolution<br>Dilution stability | MT 196 | data required before and after storage | WSB |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | WSB |
| **2.8.5.2** | Dust content | MT 171.1 | data required before and after storage | If results show > 1% w/w dust then the particle size of the dust generated shall be determined in accordance with MT 187. |
| **2.8.5.3** | Attrition | MT 193 | data required before and after storage | |
| **2.8.5.4** | Hardness and integrity | no method agreed | data required before and after storage | The data should demonstrate the mechanical robustness of the tablet and the stability in transport and use. |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **2.11** | Stability of packaging and packaging/preparation interactions | Observation of pack stability including weight change | | |
| | Disintegration time | No agreed method[15] | data required before and after storage | WSB. The data should demonstrate the tablet disintegrates rapidly on addition to water and that the formulation is readily dispersed. |

**WSB:** Where the preparation is packaged in a water soluble bag then the wet sieve, persistent foam, disintegration time and dilution stability must be carried out using the preparation and the water soluble bag in the same ratio as in the recommended application. In addition the dissolution rate of water soluble bags according to MT 176 should be determined.

[15] The data should demonstrate the tablet disintegrates rapidly on addition to water and that the formulation is readily dispersed and no blockages occur in the application equipment on use. A maximum disintegration time is to be specified. If continuous agitation is required, this should be specified on the instructions for use/label.

## 2.11 Emulsifiable powders (EP)

A powder formulation, which may contain water-insoluble formulants, to be applied as an oil-in-water emulsion of the active ingredient(s) after dispersion in water.

Water emulsifiable powders may contain one or more active ingredient(s), either solubilized or diluted in suitable organic solvent(s) which is (are) absorbed in a water soluble polymer powder or other type of soluble or insoluble powder.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.1** | Wettability | MT 53.3 | data required before and after storage | WSB |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | WSB |
| **2.8.3** | Dispersion stability | MT 180 | data required before and after storage | |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | WSB |
| **2.8.5.2** | Dust content | MT 171.1 | data required before and after storage | If results show > 1% w/w dust then the particle size of the dust generated shall be determined in accordance with MT 187. |
| **2.8.6** | Emulsifiability<br>Re-emulsifiability<br>Emulsion stability | MT 36.3 | data required before and after storage | In MT 36.3: where no oil or cream separation after 2 hours, then the 24 hour test is not required |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | Observations of pack stability including weight change and comment whether any caking or compaction has occurred on storage.<br>For flexible packs the effect of stacking packs should be determined after storage through conducting the ambient storage stability study under pressures encountered in commercial practice. | | |

**WSB:** Where the preparation is packaged in a water soluble bag then the wet sieve, persistent foam, disintegration time and dilution stability must be carried out using the preparation and the water soluble bag in the same ratio as in the recommended application. In addition the dissolution rate of water soluble bags according to MT 176 should be determined.

## 3 Seed treatments

### 3.1 Flowable concentrate for seed treatment (FS)

A stable suspension for application to the seed, either directly or after dilution.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include whether any separation or sedimentation/ claying occurs. Comment on re-dispersibility |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 | not required after storage | |
| **2.6** | Relative density | A 3 | not required after storage | |
| **2.7** | Low temperature stability | MT 39.3 | | |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.3** | Suspensibility | MT 184 | data required before and after storage | If the methods are not applicable to in use concentrations, evidence is required that there is no unacceptable phase separation on application after dilution with water. Evidence is required the preparation is homogeneous if it is not to be diluted before use. |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | |
| **2.8.7** | Pourability | MT 148 OR MT 148.1 | data required before and after storage | If applicable to the container and use instructions |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be applied in a mixture with other PPPs |
| **2.10** | Adherence to seeds | MT 194 | not required after storage | |
| **2.10** | Seed loading | appropriate validated method | | |
| **2.10** | Distribution to seeds | MT 175 | | |
| **2.11** | Stability of packaging and packaging/preparation interactions | | | Observation of pack stability including weight change. |

## 3.2 Solutions for seed treatments (LS)

A clear to opalescent liquid to be applied to the seed either directly or as a solution of the active ingredient after dilution in water. The liquid may contain water-insoluble formulants.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include whether any separation or crystallisation occurs |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or<br>MT 115 | not required after storage | |
| **2.6** | Relative density | A 3 or<br>OECD 109 | not required after storage | |
| **2.7** | Low temperature stability | MT 39.3 | | |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.4** | Dilution stability | MT 41.1 | data required before and after storage | The preparation should form a clear homogeneous solution on dilution with water |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be applied in a mixture with other PPPs |
| **2.10** | Adherence to seeds | MT 194 | not required after storage | |
| **2.10** | Seed loading | appropriate validated method | | |
| **2.10** | Distribution to seeds | MT 175 | | |
| **2.11** | Stability of packaging and packaging/preparation interactions | Observation of pack stability including weight change. | | |

## 3.3 Emulsions for seed treatments (ES)

A stable emulsion for application to the seed either directly or after dilution.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations after storage should include whether any separation occurs. Comment on ease of re-homogenisation |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21<br>Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 or<br>MT 115 | not required after storage | |
| **2.6** | Relative density | A 3 or<br>OECD 109 | not required after storage | |
| **2.7** | Low temperature stability | MT 39.3 | | |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.6** | Emulsifiability<br>Re-emulsifiability<br>Emulsion stability | MT 36.3 | data required before and after storage | If the methods are not applicable to in use concentrations, evidence required that there is no unacceptable phase separation on application after dilution with water.<br>Evidence is required the preparation is homogeneous if it is not to be diluted before use. |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be applied in a mixture with other PPPs |
| **2.10** | Adherence to seeds | MT 194 | not required after storage | |
| **2.10** | Seed loading | appropriate validated method | | |
| **2.10** | Distribution to seeds | MT 175 | | |
| **2.11** | Stability of packaging and packaging/preparation interactions | Observation of pack stability including weight change. Indicate if there is any seepage of solvent through the container walls or seal. | | |

## 3.4 Powders for dry seed treatments (DS)

A powder for application in the dry state directly to the seed.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.5.1** | Particle size distribution | OECD 110 | data required before and after storage | |
| **2.8.5.1** | Dry sieve test | MT 170 | data required before and after storage | |
| **2.8.5.2** | Dust content | MT 171.1 | data required before and after storage | If results show > 1% w/w dust then the particle size of the dust generated shall be determined in accordance with MT 187. |
| **2.10** | Adherence to seeds | MT 194 | not required after storage | |
| **2.10** | Seed loading | appropriate validated method | | |
| **2.10** | Distribution to seeds | appropriate method | | |
| **2.11** | Stability of packaging and packaging/preparation interactions | Observation of pack stability including weight change | | |

## 3.5 Water dispersible powders for slurry seed treatment (WS)

A powder to be dispersed at high concentration in water before application as a slurry to the seed.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.1** | Wettability | MT 53.3 | data required before and after storage | |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | |
| **2.8.3** | Suspensibility | MT 184 | data required before and after storage | If the methods are not applicable to in use concentrations, evidence is required that the dispersion is homogeneous |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | |
| **2.8.5.2** | Dust content | MT 171.1 | data required before and after storage | If results show > 1% w/w dust then the particle size of the dust generated shall be determined in accordance with MT 187. |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be applied in a mixture with other PPPs |
| **2.10** | Adherence to seeds | MT 194 | not required after storage | |
| **2.10** | Seed loading | appropriate validated method | | |
| **2.10** | Distribution to seeds | MT 175 | | |
| **2.11** | Stability of packaging and packaging/preparation interactions | observation of pack stability including weight change | | |

## 3.6 Water soluble powders for seed treatment (SS)

A powder formulation to be applied to the seed as a true solution of the active ingredient in water.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | Observations of granule integrity are required before and after storage |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.1** | Wettability | MT 53.3 | data required before and after storage | WSB |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | WSB |
| **2.8.4** | Degree of dissolution | MT 179.1 | data required before and after storage | WSB |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | WSB |
| **2.8.5.1** | Nominal size range | MT 170 | data required before and after storage | |
| **2.8.5.2** | Dust content | MT 171.1 | data required before and after storage | If results show > 1% w/w dust then the particle size of the dust generated shall be determined in accordance with MT 187. |
| **2.8.5.3** | Attrition | MT 178.2 | not required after storage | |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be used in tank mix with other PPPs |
| **2.10** | Adherence to seeds | MT 194 | not required after storage | |
| **2.10** | Seed loading | appropriate validated method | | |
| **2.10** | Distribution to seeds | appropriate method | | |
| **2.11** | Stability of packaging and packaging/preparation interactions | Observation of pack stability including weight change.<br>There should be no loss of granule integrity or caking.<br>For flexible packs the effect of stacking packs on granule integrity and particle size should be determined after storage | | |

**WSB:** Where the preparation is packaged in a water soluble bag then the wet sieve, persistent foam, wettability and solution/dilution stability tests must be carried out using the preparation and the water soluble bag in the same ratio as in the recommended application. In addition the dissolution rate of water soluble bags according to MT 176 should be determined

# 4 Miscellaneous

## 4.1 Smoke generator (FU)

A combustible formulation, generally solid, which upon ignition releases the active ingredient(s) in the form of smoke.

Evidence is required that the preparation generates a smoke when used according to label recommendations.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | Evidence is also required that active substance is stable at the temperatures likely to occur in smoking[16] |
| **2.1** | Appearance:<br>physical state<br>colour | | data required before and after storage | |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A15, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.11** | Stability of packaging and packaging/preparation interactions | | | Observation of pack stability and integrity including weight change on storage |
| | Burning time[17] | No standard method | data required before and after storage | Evidence is required that the preparation may be satisfactorily applied as a smoke |
| | Evidence of combustibility[18] | No standard method | data required before and after storage | The quantity of material remaining after combustion (including unvolatilised a.s.) should be determined |

[16] This is required to confirm that the smoke delivers the required active concentration as stated on the product label.

[17] The burning rate should correspond with the proposed use. The duration and burning rate of a smoke generator should be specified to establish how long it takes before the preparation stops generating smoke. Data are required, based on a representative in-use situation, to show the burning rate and duration comply with the specified rates on the product label. Where relevant the data must support intermittent use of the product.

[18] Evidence of combustibility (completeness of burning) can be determined by weighing the preparation before and after use. It should be demonstrated that by far the largest part of the active substance was volatised in the smoke composition. This also requires determination of the concentration active substance in the residue.

## 4.2 Fogging concentrates, hot fogging (HN) cold fogging (KN)

HN - A formulation suitable for application by hot fogging equipment, either directly or after dilution.

KN - A formulation suitable for application by cold fogging equipment, either directly or after dilution.

| **Annex point** | **Property** | **Test Method** | **Storage stability requirements** | **Comments** |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | evidence is also required that the active substance is stable at the temperatures likely to occur in fogging machinery |
| **2.1** | Appearance:<br><br>physical state<br>colour | | data required before and after storage | |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A15<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC |
| **2.5** | Surface tension | A 5 | not required after storage | Only required for liquid products |
| **2.6** | Relative density<br>Bulk density | A 3<br>MT 186 | not required after storage | |
| **2.7** | Low temperature stability | MT 39.3 | | Only required for liquid products |
| **2.11** | Stability of packaging and packaging/preparation interactions | observation of pack stability including weight change and comment whether any pack deterioration on storage | | |

## 4.3 Gels - water soluble gel (GW)

Gels are jelly-like colloidal systems of complex physical chemistry. Gels may either be dispersed in water before use or formulated as 'Ready to use' preparations. The data required on the technical characteristics of the preparation will be dependent on its mode of use e.g. if the preparation is a water soluble gel then data on dilution stability and wettability will be required.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance: physical state colour | | data required before and after storage | |
| **2.2** | Explosive properties | A 14 UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 21 Test O.2 | not required after storage | |
| **2.3** | Flash point and flammability | A9, A15 | not required after storage | |
| **2.4** | pH acidity/ alkalinity(if applicable) | MT 75.3 MT 191 MT 31.1 | data required before and after storage | |
| **2.5** | Viscosity | OECD 114 or MT 192 | not required after storage | Test must be conducted at 20°C and 40ºC. |
| **2.5** | Surface tension | A 5 or OECD 115 | not required after storage | |
| **2.6** | Relative density | A 3 or OECD 109 | not required after storage | It may be more appropriate to treat as a solid depending on viscosity |
| **2.7** | Low temperature stability | MT 39.3 | pH, suspensibility, and emulsion stability as relevant should be determined after storage | |
| **2.8.2** | Persistent foam | MT 47.3 | not required after storage | Required if the preparation is to be dispersed in water |
| **2.8.3** | Suspensibility | MT 184 | data required before and after storage | only required if the preparation is to be dissolved in water |
| **2.8.3** | Spontaneity | MT 160 | data required before and after storage | |
| **2.8.4** | Dilution stability | MT 179.1 | data required before and after storage | |
| **2.8.5.1** | Wet sieve | MT 185 | data required before and after storage | only required if the preparation is to be dispersed in water |
| **2.8.6** | Emulsifiability Re-emulsifiability Emulsion stability | MT 36.3 | data required before and after storage | Only required if the preparation is to be emulsified in water. |
| **2.9** | Physical compatibility | ASTM E1518-05 | not required after storage | Required if product is to be applied in a mixture with other PPPs |
| **2.11** | Stability of packaging and packaging/preparation interactions | | Observations of pack stability including weight change. Indicate if there has been seepage/migration of the a.s. | |

## 4.4 Baits:- Bait concentrate (CB) and ready-to-use bait (RB)

These requirements should be read in conjunction for those describing the efficacy of the preparation as there are some areas where data for the biological efficacy of the preparation may be used to support the preparation chemistry and storage stability requirements. For preparations with the same bait base which differ only in active substance content, there may be scope to extrapolate some data between different preparations.

| **Annex point** | **Property** | **Test Method** | **Storage stability requirements** | **Comments** |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br><br>physical state<br>colour | | data required before and after storage | Evidence must be provided that the bait retains the physical state including observations of compaction on storage and there is no obvious bio-degradation e.g. microbial growth<br>For all baits it must be demonstrated that the product can be satisfactorily applied according to the recommendations on the label |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A12, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.6** | Relative density<br><br>Bulk density | A 3 or OECD 109<br>MT 186 | not required after storage | |
| **2.11** | Stability of packaging and packaging/preparation interactions | observation of pack stability including weight change | | |
| | Retention of palatability | | After storage | evidence of retention of biological efficacy may be acceptable but consideration must be given to the number of animals required in testing |

## 4.5 Plant Rodlet (PR)

A small rodlet, usually a few centimetres in length and a few millimetres in diameter, containing an active ingredient, generally designed for direct application.

| Annex point | Property | Test Method | Storage stability requirements | Comments |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | |
| **2.1** | Appearance:<br><br>physical state<br>colour | | data required before and after storage | Observations after storage should include stick integrity. |
| **2.2** | Explosive properties | A 14<br>UN RTDG Manual of Tests and Criteria | not required after storage | |
| **2.2** | Oxidising properties | A 17<br>Test O.1 | not required after storage | |
| **2.3** | Flash point and flammability | A10, A16<br>Test N.1<br>Test N.4 | not required after storage | |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | |
| **2.6** | Bulk density | MT 186 | not required after storage | |
| **2.8.5.3** | Attrition | MT 178<br>MT 193 | data required before and after storage | The data should demonstrate the robustness of the stick and it's stability in transport and use. |
| **2.8.5.4** | Hardness and integrity | | data required before and after storage | |
| **2.11** | Stability of packaging and packaging/preparation interactions | observation of pack stability including weight change | | |

## 4.6 Aerosols (AE)

A container-held formulation which is dispersed generally by a propellant as fine droplets or particles upon the actuation of a valve. Guidance on the testing of aerosols may be found in 'The Aerosol Directive' (75/324/EEC) and the subsequent amendment 94/1/EC. Additional methods for the determination of these aerosol specific properties (internal pressure / discharge rate) are included in the FAO / WHO manual.[19]

| **Annex point** | **Property** | **Test Method** | **Storage stability requirements** | **Comments** |
|---|---|---|---|---|
| **1.4.1** | Active substance content | appropriate validated method | data required before and after storage | The net active substance content of the spray produced from the aerosol should be determined shall not be lower than that declared. |
| **2.3** | Flammability | | not required after storage | Flammability should be tested in accordance with the methods described in Annex I, Part 2.3 of Regulation (EC) No 1272/2008 and classified accordingly. |
| **2.4** | pH<br>acidity/ alkalinity(if applicable) | MT 75.3<br>MT 191<br>MT 31.1 | data required before and after storage | Applicable to water-based formulations only |
| **2.7** | Low temperature stability | | Required if ambient testing did not include low temperature storage | |
| **2.11** | Stability of packaging and packaging/preparation interactions | No standard method | Required before and after storage | Observations of pack stability including weight change. Indicate if there has been seepage/migration of the a.s.<br>Observation of can integrity. |
| | Weight loss of can after a 5 second spray (repeated three times) | No standard method | Required before and after storage | These data allow a measurement of the approximate application rate |
| | Spray pattern[20] | No standard method | Required before and after storage | Evidence is required that the container and nozzle/spray release mechanism remains intact on storage with no corrosion and that there is no nozzle blockage on storage. (required after storage only). |
| | Internal pressure | No standard method | Required before and after storage | The determination of the pressure existing in the finished aerosol packs is necessary to verify that the true pressure is compatible with the pressure limitations of the pack, and in accordance with the regulations. |
| | Discharge rate | No standard method | Required before and after storage | |

[19] Manual for the development and use of FAO and WHO Specifications for pesticides, November 2010 2nd revision of the 1st Edition and any additional supplements.

[20] Determined by measuring the diameter of the wet patch on a piece of cardboard (or similar material) when sprayed at a distance of 30 cm.

**Summary of requirements for the technical characteristics of the plant protection product**

| Point | Technical characteristic | SL | SC | CS | EC | SE | DC | EW | ME | OD | UL | OL | ZC | ZW | ZE | GR | WG | SG | EG | WP | SP | DP |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2.8.1 | Wettability | | | | | | | | | | | | | | | | S | S | S | S | S | |
| 2.8.2 | Persistence of foaming | X | X | X | X | X | X | X | X | X | | | X | X | X | | X | X | X | X | X | |
| 2.8.3 | Suspensibility | | S | S | | | | | | | | | S | | | | S | | | S | | |
| 2.8.3 | Spontaneity of dispersion | | S | S | | | | | | | | | S | | | | S | | | | | |
| 2.8.3 | Dispersion stability | | | | | S | S | | | S | | | | S | S | | | | S | | | |
| 2.8.4 | Degree of dissolution | | | | | | | | | | | | | | | | | | | | | |
| 2.8.4 | Dilution stability | S | | | | | | | | | | | | | | | | S | | | S | |
| 2.8.5.1 | Particle size distribution | | | X | | | | | | S | | | X | X | X | S | S | S | S | S | S | S |
| 2.8.5.1 | Wet sieve test | | S | S | | S | S | | | S | S | | S | S | S | | S | S | S | S | S | |
| 2.8.5.1 | Dry sieve | | | | | | | | | | | | | | | S | S | S | S | | | S |
| 2.8.5.2 | Dust content | | | | | | | | | | | | | | | S | S | S | S | S | S | S |
| 2.8.5.3 | Attrition | | | | | | | | | | | | | | | S | S | S | S | | | |
| 2.8.6 | Emulsifiability tests | | | | S | | | S | S | | | | | | | | | | S | | | |
| 2.8.6 | Emulsion stability | | | | S | | | S | S | | | | | | | | | | S | | | |
| 2.8.7 | Flowability | | | | | | | | | | | | | | | X | X | X | | | | |
| 2.8.7 | Pourability | | S | S | | S | | S | | S | | | S | S | S | | | | | | | |
| 2.8.7 | Dustability | | | | | | | | | | | | | | | | | | | | | S |
| 2.11 | Solubility of WSB | | | | | | | | | | | | | | | | S | S | S | S | S | |
| 2.11 | Miscibility with hydrocarbon oil | | | | | | | | | | | S | | | | | | | | | | |
| 2.11 | Droplet size | | | | | | | | | | S | | | | | | | | | | | |

Where: X = test required prior to storage, only S = test required before and after storage

**All formulations require active substance content, appearance, pH (acidity/alkalinity if necessary) and packaging stability following storage in addition to the storage stability tests in the table above. See separate tables in Appendix 1 for details of all properties required both before and after storage for individual formulation types.**

**Summary of requirements for the technical characteristics of the plant protection product (continued)**

| Point | Technical characteristic | DT | WT | ST | EP | FS | LS | ES | DS | WS | SS | FU | HN | KN | GW | CB | RB | PR |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2.8.1 | Wettability | | | | S | | | | | S | S | | | | | | | |
| 2.8.2 | Persistence of foaming | | X | X | X | X | X | X | | X | X | | | | X | | | |
| 2.8.3 | Suspensibility | | S | | | S | | | | S | | | | | S | | | |
| 2.8.3 | Spontaneity of dispersion | | | | | | | | | | | | | | S | | | |
| 2.8.3 | Dispersion stability | | | | S | | | | | | | | | | | | | |
| 2.8.4 | Degree of dissolution | | | S | | | | | | | S | | | | | | | |
| 2.8.4 | Dilution stability | | | S | | | S | | | | | | | | S | | | |
| 2.8.5.1 | Particle size distribution | S | S | S | S | | | | S | S | S | | | | | | | |
| 2.8.5.1 | Wet sieve test | | S | S | S | S | | | | S | S | | | | S | | | |
| 2.8.5.1 | Dry sieve test | | | | | | | | S | | S | | | | | | | |
| 2.8.5.2 | Dust content | S | S | S | S | | | | S | S | S | | | | | | | |
| 2.8.5.3 | Attrition (tablet) | S | S | S | | | | | | | X | | | | | | | S |
| 2.8.5.4 | Hardness and integrity | S | S | S | | | | | | | | | | | | | | S |
| 2.8.6 | Emulsifiability tests | | | | S | | | S | | | | | | | S | | | |
| 2.8.6 | Emulsion stability | | | | S | | | S | | | | | | | S | | | |
| 2.8.7 | Flowability | | | | | | | | | | | | | | | | | |
| 2.8.7 | Pourability | | | | | S | | | | | | | | | | | | |
| 2.10 | Adherence to seed | | | | | X | X | X | X | X | X | | | | | | | |
| 2.10 | Distribution to seed | | | | | X | X | X | X | X | X | | | | | | | |
| 2.10 | Seed loading | | | | | X | X | X | X | X | X | | | | | | | |
| 2.11 | Solubility of WSB | | | | S | | | | | | S | | | | | | | |
| 2.11 | Burning time | | | | | | | | | | | S | | | | | | |
| 2.11 | Evidence of combustibility | | | | | | | | | | | S | | | | | | |
| 2.11 | Retention of palatability | | | | | | | | | | | | | | | S | S | |
| 2.11 | Disintegration time | | S | S | | | | | | | | | | | | | | |

Where: X = test required prior to storage, only S = test required before and after storage

**All formulations require active substance content, appearance, pH (acidity/alkalinity if necessary) and packaging stability following storage in addition to the storage stability tests in the table above. See separate tables in Appendix 1 for details of all properties required both before and after storage for individual formulation types.**