Specifications for formulated pesticides

Learning objectives

After completing this *Learning unit*, you should understand:

the structure and aims of specifications for formulated products;

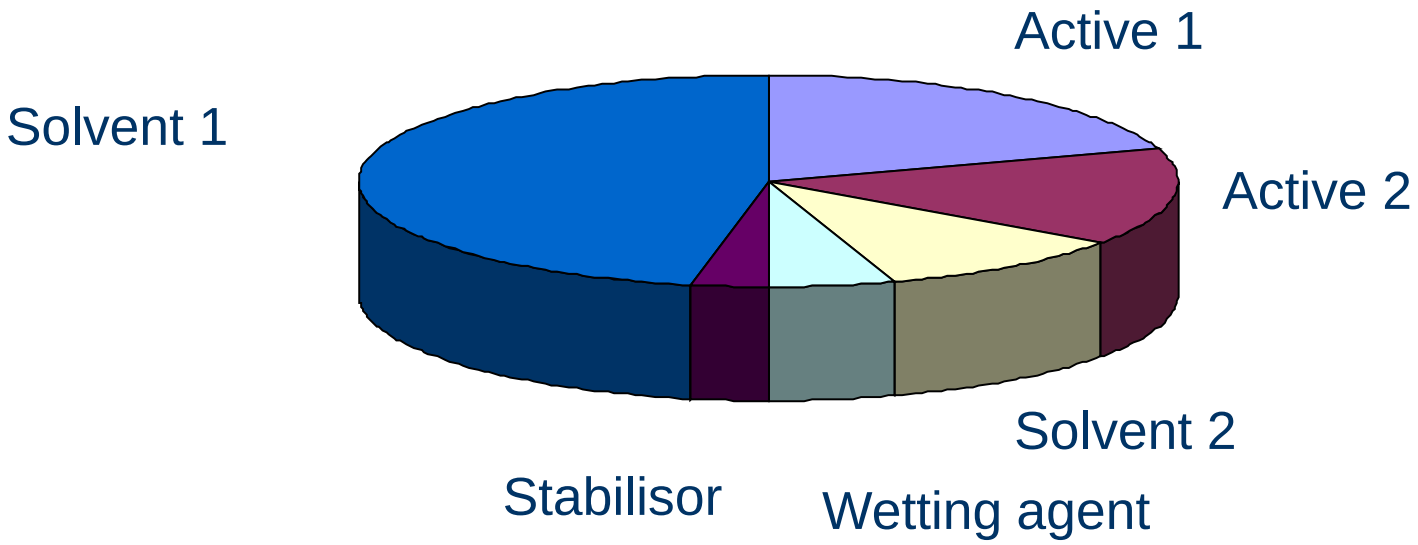
data requirements for developing specifications for formulated products

What is a formulated pesticide?

Active ingredient in the form sold for use.

Active ingredient plus formulants (excipients, “inerts”) assembled to optimize delivery to target pest, optimize activity, stabilize active ingredient, minimize user exposure, simplify use, etc.

Example Pesticide Formulation



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Prerequisites for formulation specifications

A TC or TK specification is normally required, except in unusual cases where a TC or TK is not isolated.

Because this ensures the strongest possible links to hazard and risk assessments.

Additional supporting data required?

Few additional data are normally required, over and above those supporting the TC or TK specification, except:

to support proposed specification limits which would normally be considered borderline for good quality;

non-standard clauses or limits require supporting information;

novel or unique formulations may require additional supporting information.

Efficacy data

National authorities are responsible for efficacy assessment, before developing specifications.

Existing efficacy assessments (e.g. WHO evaluations of public health pesticides), of comparable scenarios, may be used to minimize requirements for national or local testing.

Scope of formulation specifications

Similar to TC and TK specifications but also specify physical properties and synergist (if applicable).

Essential additives for safety or stability.

Unlike FAO and WHO, national authorities should control formulants.

Specifications for mixed active ingredients and formulations

In most cases, separate specifications apply to each active ingredient. SE, ZC, ZE, ZW are treated differently because of the complexity of these products.

Where the ratio of active ingredients is critically important, a specification may be developed for an individual formulated product.

Where two or more solid formulations are mixed, expanded tolerances for active ingredient content take account of the tolerance on formulation ratio and increased heterogeneity

(Appendix K, FAO/WHO specifications manual).

Description clause

- Physical appearance of product and chemical form of the active ingredient.
- Provides a simple and rapid means to determine compliance.
- Corresponding TC or TK specification is referenced.

Active ingredient identity and content

- Test methods similar to those for TC and TK, but extraction (and purification for identification) of active ingredient may be required.
- May be necessary to identify the counter-ion, etc., if it is critical for product stability or performance.
- Analytical test methods for determination of content validated by international collaborative study, to provide evidence of the reliability of the methods and the data provided.

Tolerances for active ingredient content

Declared content, g/kg or g/l tolerance

- up to 25 ± 15% for “homogeneous” products (e.g. EC, SC, SL)
- ± 25% for “heterogeneous” products (e.g. GR, WG)
- above 25 up to 100 ± 10% g/kg or g/l
- above 100 up to 250 ± 6% g/kg or g/l
- above 250 up to 500 ± 5% g/kg or g/l
- above 500 ± 25 g/kg or g/l

Relevant impurities

Criteria as for TC and TK but insolubles (particulates) and acidity/alkalinity are treated as physical properties.

Limits usually based on active ingredient content but may be higher if concentrations can increase in storage or through reactions with formulants.

An impurity relevant in TC or TK may become non-relevant in formulations containing only low levels of active ingredient, e.g. if the impurity concentration is diluted to a level too low to measure.

Physical properties

Specified properties are the minimum to distinguish good and bad products.

Clauses and limits may differ from FAO/WHO guidelines, if justified for a particular product.

Test methods for physical properties are simple models; they do not demonstrate field performance.

Results are method-dependent, so test methods must be performed exactly as described.

If the test method for a physical property has not been suitably validated and/or published, the specification cannot be developed.

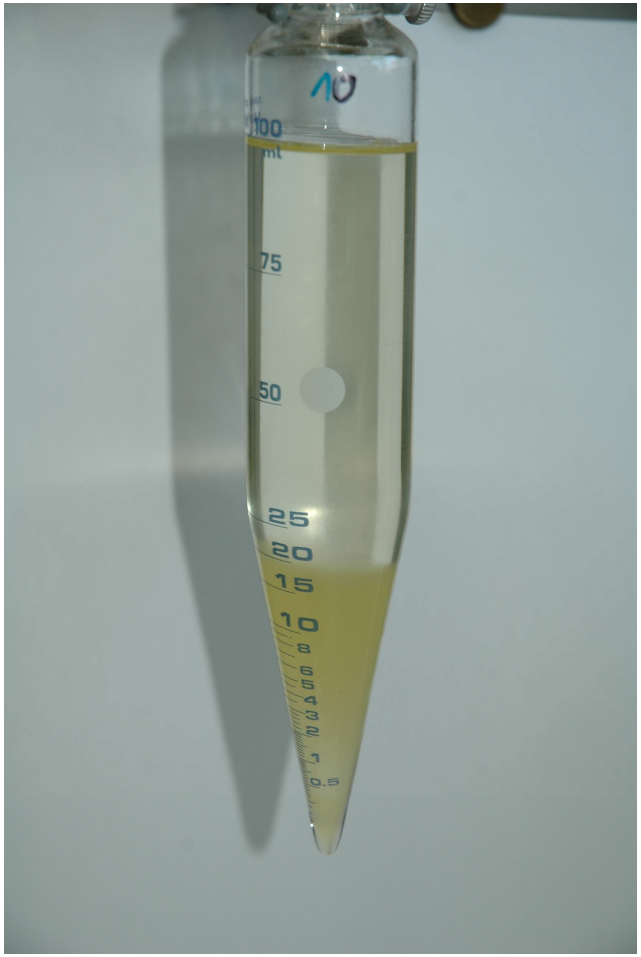
Low temperature storage stability

- Storage test at 0 ºC required for liquid formulations, which may grow crystals, aggregate particles or develop separate phases.

CS formulations may require freeze-thaw test to show that capsules are not weakened by freezing.

Low temperature storage stability

- Storage test at 0 ºC



High temperature storage stability

Test required for all formulations.

- Simulates two years' storage under "cool" conditions.
- Standard requirement is 54 ºC for 14 days.
- If 54 ºC is not appropriate for the product, alternative conditions are:
- 45 ºC for 6 weeks
- 40 ºC for 8 weeks
- 35 ºC for 12 weeks
- 30 ºC for 18 weeks.

High temperature storage stability

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Post-storage tests required

- Active ingredient content – usual minimum is ≥95% of pre-storage level.
- Relevant impurities, if they could increase in storage.
- Physical properties, if they could worsen with storage.

HAPPYFOS WATER DISPERSIBLE GRANULES

FAO Specification 999/WG (December 2007*)

*This specification, which is PART ONE of this publication, is based on an evaluation of data submitted by the manufacturer whose name is listed in the evaluation report (*999/2007*). It should be applicable to relevant products of this manufacturer, and those of any other formulators who use only TC from the evaluated source. The specification is not an endorsement of those products, nor a guarantee that they comply with the specification. The specification may not be appropriate for the products of other manufacturers who use TC from other sources. The evaluation report (*999/2007*), as PART TWO, forms an integral part of this publication.*

1 **Description**

The material shall consist of a homogeneous mixture of technical happyfos, complying with the requirements of FAO specification 999/TC (December 2007), together with carriers and any other necessary formulants. It shall be in the form of granules for application after disintegration and dispersion in water. The product shall be dry, free-flowing and free from visible extraneous matter and hard lumps.

2.1 **Identity tests** (999/WG/M/2, CIPAC X, p.196, 2003)The active ingredient shall comply with an identity test and, where the identity remains in doubt, shall comply with at least one additional test.

2.2 **Happyfos content** (999/WG/M/3, CIPAC X, p.196, 2003)

The happyfos content shall be declared (g/kg) and, when determined, the average measured content shall not differ fromthat declared by more than the following amounts.

Declared content, g/kg tolerance

above 100 up to 250 ± 6% g/kg

above 250 up to 500 ± 5% g/kg

3 **Physical properties**

3.1 **pH range** (MT 75.3, CIPAC J, p. 131, 2000)

pH range: 5.0 to 7.0.

3.2 **Wettability** (MT 53.3, CIPAC F, p. 160, 1995)

The formulation shall be completed wetted in 5 seconds without swirling.

3.3 **Wet sieve test** (MT167, CIPAC F, p. 416, 1995) A maximum of 0.5 % w/w shall be retained on a 75 µm test sieve.

3.4 **Degree of dispersion** (MT 174, CIPAC F, p. 435, 1995)
The minimum dispersibility shall be 70% after 1 minute of stirring.

3.5 **Suspensibility** (MT 168, CIPAC F, p. 417, 1995) A minimum of 50% of the happyfos content found under 2.2 shall be in suspension after 30 minutes in CIPAC standard water D at 30 ± 2 ° C.

3.6 **Persistent foam** (MT 47.2, CIPAC F, p. 152, 1995)
There shall be a maximum of 10 ml after 1 minute.

3.7 **Dustiness** (MT 171, CIPAC F, p. 425, 1995))
The formulation shall be essentially non-dusty.

3.8 **Flowability** (MT 172, CIPAC F, p. 430, 1995)
At least 98% of the formulation shall pass through a 5 mm test sieve after 20 drops of the sieve.

3.5 **Suspensibility** (MT 168, CIPAC F, p. 417, 1995) A minimum of

50% of the happyfos content found under 2.2 shall be in suspension after 30 minutes in CIPAC standard water D at 30 ± 2 ° C.

3.6 **Persistent foam** (MT 47.2, CIPAC F, p. 152, 1995)

There shall be a maximum of 10 ml after 1 minute.

4 **Storage stability**

4.1 **Stability at elevated temperature** (MT 46.3, CIPAC J, p.128, 2000)

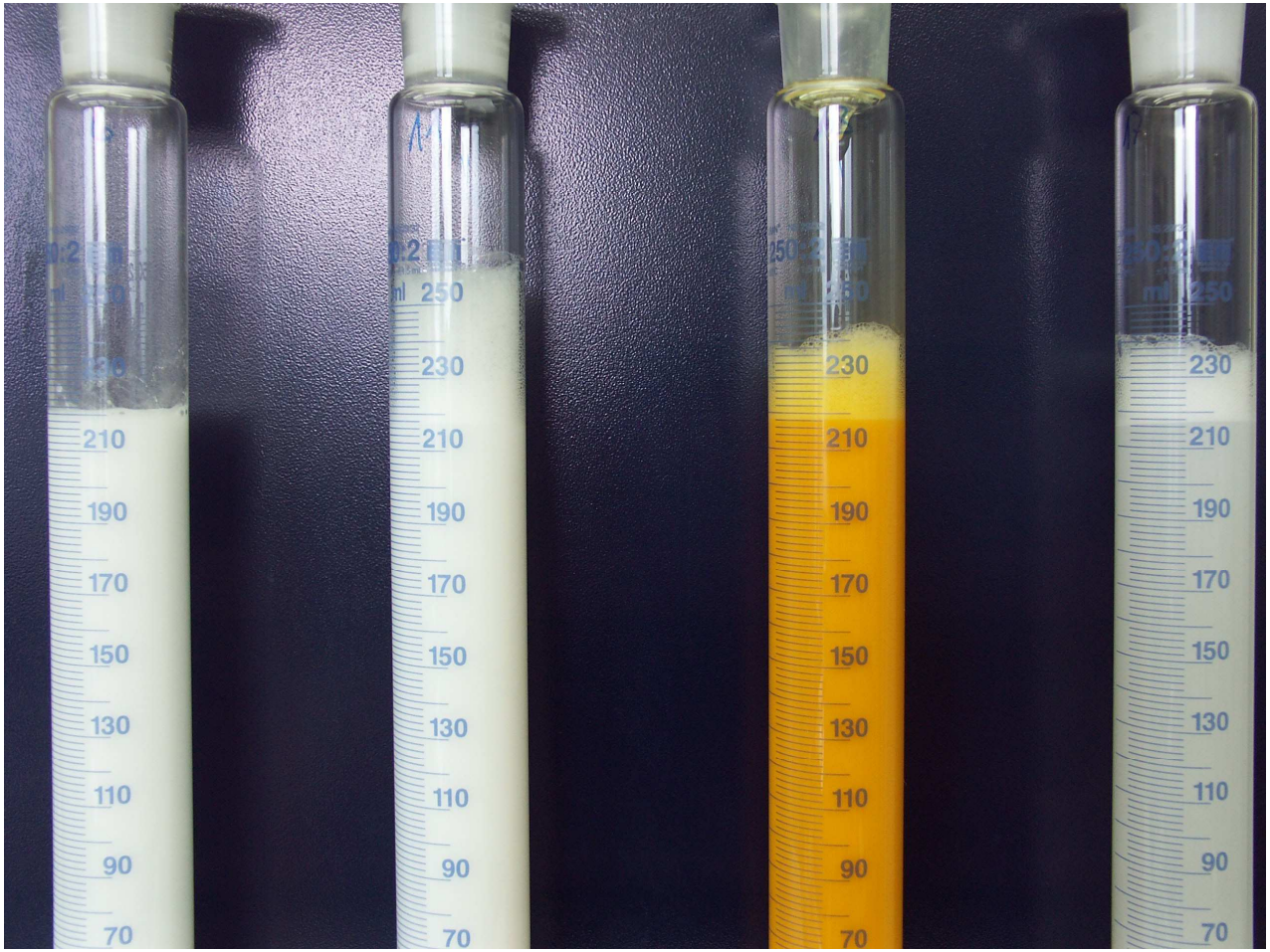
After storage at 54 ± 2° C for 14 days, the determined average active ingredient content shall not be lower than 95%, relative to the determined average content found under 2.2 before storage, and the material shall continue to comply with clauses for:

pH range (3.1),
wet sieve test (3.3),
degree of dispersion (3.4),
suspensibility (3.5),
dustiness (3.7),
flowability (3.8).

Note 1....

* *Specifications may be revised and/or additional evaluations may be undertaken.Ensure the use of current versions by checking at: http://www.fao.org/ag/agp/agpp/pesticid/.*

Persistent foam



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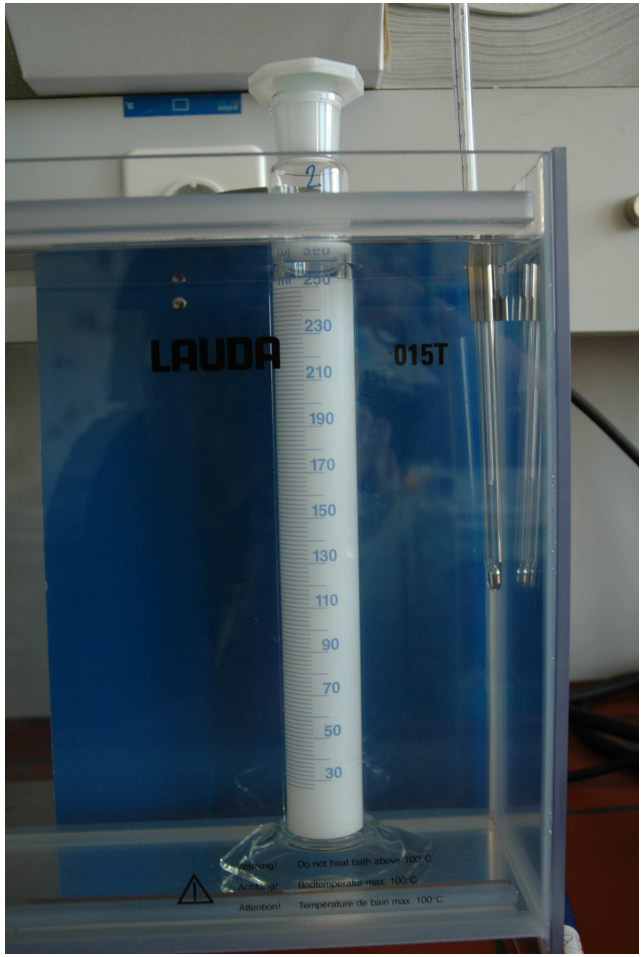
Suspension stability



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Dustiness

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