6.23 **WATER SOLUBLE GRANULES (****SG)**

Note for preparation of draft specifications. Do not omit clauses or insert additional clauses, nor insert limits that are more lax than those than given in the guidelines, without referring to section 4. From the “Notes” provided at the end of this guideline, incorporate only those which are applicable to the particular specification.

**...... [ISO common name] WATER SOLUBLE GRANULES**

(CIPAC No ......)/SG (month & year of publication)

**Description**

The material shall consist of granules containing technical ...... [ISO common name] complying with the requirements of the FAO/WHO specification ......, in the form of ....... (see Section 4.2), and, if required, suitable carriers and/or necessary formulants. It shall be homogeneous, free from visible extraneous matter and/or hard lumps, free flowing, and nearly dust free or essentially non-dusty. The active ingredient shall be soluble in water. Insoluble carriers and formulants shall not interfere with compliance with 6.23.4.2.

Where the material is packaged in sealed water soluble bags, the description shall be as follows (Note 1):

The material shall consist of a defined quantity of ...... [ISO common name] water soluble granules complying with the requirements of FAO/WHO specification ..... , in the form of ....... (see Section 4.2), contained in a sealed water soluble bag.

6.23.2 **Active ingredient**

6.23.2.1 **Identity tests** (Note 2)

 The active ingredient shall comply with an identity test and, where the identity remains in doubt, shall comply with at least one additional test.

6.23.2.2 **...... [ISO common name] content** (Note 2)

 The ...... [ISO common name] content shall be declared (g/kg) and, when determined, the average content measured shall not differ from that declared by more than the appropriate tolerance, given in the table of tolerances, Section 4.3.2.

6.23.3 **Relevant impurities**

6.23.3.1 **By-products of manufacture or storage** (Note 3), if required

 Maximum: .…..% of the …… [ISO common name] content found under 6.23.2.2.

6.23.3.2 **Water** (MT 30.5) (Notes 4 and 5), if required

 Maximum: ..…. g/kg.

6.23.4 **Physical properties**

 6.23.4.1 **Acidity** and/or **Alkalinity** (MT 191) or **pH range** (MT 75.3), if required

 Maximum acidity: ...... g/kg calculated as H2SO4.

 Maximum alkalinity: ...... g/kg calculated as NaOH.

 pH range: ...... to ......

6.23.4.2 **Degree of dissolution and solution stability** (MT 179.1)

 Residue of formulation retained on a ...... µm test sieve after dissolution in CIPAC Standard Water D at 25 ± 5°C (Note 6).

 Maximum: ......% after 5 min.

 Maximum: ......% after 24 h.

6.23.4.3 **Persistent foam** (MT 47.3) (Note 7)

 Maximum ...... ml after 1 min.

 In the case of water soluble bag packaging, the provisions of clause 6.23.6.3 should be applied.

6.23.4.4 **Dustiness** (MT 171.1) (Note 8)

 The formulation shall have a maximum collected dust of 30 mg by the gravimetric method or a maximum dust factor of 25 by the optical method.

6.23.4.5 **Attrition resistance** (MT 178.2)

Minimum: ......% attrition resistance.

6.23.4.6 **Flowability** (MT172.1)

 At least ......% of the formulation shall pass through a 5 mm test sieve after 20 drops of the sieve.

6.23.5 **Storage stability**

6.23.5.1 **Stability at elevated temperatures** (MT 46.3)

 After storage at 54 ± 2 °C for 14 days (Note 9) the determined average active ingredient content shall not be lower than ......% relative to the determined average content found before storage (Note 10) and the formulation shall continue to comply with the clauses for:

- by-products of manufacture or storage (6.23.3.1),

- acidity/alkalinity/pH range (6.23.4.1),

- degree of dissolution and solution stability (6.23.4.2),

- dustiness (6.23.4.4),

- attrition resistance (6.23.4.5),

as required.

 In the case of water soluble bag packaging, the package should be enclosed in a watertight sachet, box or any other container at ...... °C (Note 12) for ...... days. The determined average active ingredient content must not be lower than ......% relative to the determined average content found before storage, and the formulation shall continue to comply with the clauses for:

- by-products of manufacture or storage (6.23.3.1),

- acidity/alkalinity/pH range (6.23.4.1),

- dissolution of the bag (6.23.6.1),

- degree of dissolution and solution stability (6.23.6.2),

- persistent foam (6.23.6.3),

as required. None of the bags tested should show signs of leakage or rupture during normal handling, before and after storage.

6.23.6 **Material packaged in a sealed water soluble bag** (Notes 12, 13 & 14)

6.23.6.1 **Dissolution of the bag** (MT 176)

 The dissolution of the bag shall be tested on a sample of the emptied and cleaned bag in CIPAC Standard Water D taken according to the procedure described in Note 13, together with an appropriate proportion of the SG.

 Flow time of the solution: maximum ...... sec.

6.23.6.2 **Degree of dissolution and solution stability** (MT 179.1)

 The degree of dissolution and solution stability shall be tested on a solution containing the SG and the bag material in the actual ratio of application, prepared according to the procedure described in Note 14.

 Residue of formulation retained on a ...... µm test sieve after dissolution in CIPAC Standard Water D at 25 ± 5 °C (Note 6).

 Maximum: ......% after 5 min.

 Maximum: ......% after 24 h.

6.23.6.3 **Persistent foam** (MT 47.3) (Note 7)

 The persistent foam shall be tested on a solution containing the SG and the bag in the actual ratio of application, prepared according to the procedure described in Note 15.

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Note 1 For record keeping purposes, the suffix “SB” should be added to the formulation code (SG-SB).

Note 2 Method(s) of analysis must be CIPAC, AOAC or equivalent. If the methods have not yet been published then full details, with appropriate method validation data, must be submitted to FAO/WHO by the proposer.

Note 3 This clause should include only relevant impurities and the title should be changed to reflect the name of the relevant impurity. Method(s) of analysis must be peer validated.

Note 4 The method to be used shall be stated. If several methods are available, a referee method shall be selected.

Note 5 If required, a minimum water content may be specified as an alternative, or in addition, to the maximum.

Note 6 Unless other temperatures or waters are specified.

Note 7 The mass of sample to be used in the test should be specified at the highest rate recommended by the supplier. The test is to be conducted in CIPAC standard water D.

Note 8 The optical method of MT 171.1, usually shows good correlation with the gravimetric method and can, therefore, be used as an alternative where the equipment is available. Where the correlation is in doubt, it must be checked with the formulation to be tested. In case of dispute the gravimetric method shall be used.

Note 9 Unless other temperatures and/or times are specified. Refer to Section 4.6.2 of this Manual for alternative storage conditions.

Note 10 Samples of the formulation taken before and after the storage stability test may be analyzed together after the test in order to reduce the analytical error.

Note 11 If, due to irreversible changes in the characteristics of the bag material when stored above 50 °C, the test temperature should not exceed 45°C, refer to Section 4.6.2 of this Manual for alternative storage conditions.

Note 12 Sub-sampling

 Lay the bag on a bench and carefully open one side of the bag with a cutter, taking care not to damage the seals.

 Transfer the contents of the bag into a suitable flask. This material shall be used to carry out the tests for:

- active ingredient identity (6.23.2.1),

- active ingredient content (6.23.2.2),

- by-products of manufacture or storage (6.23.3.1),

- water content (6.23.3.2),

- acidity/alkalinity/pH range (6.23.4.1),

- dissolution of the bag (6.23.6.1),

- degree of dissolution (6.23.6.2),

- persistent foam (6.23.6.3),

as required.

 The bag is then opened on three sides, completely cleaned from adhering powder by brushing or suction and weighed to the nearest 0.01 g. It shall be used to carry out the dissolution test (6.23.6.1). Aliquots of an aqueous solution of the bag material shall be used in the degree of dissolution and solution stability (6.23.6.2) and persistent foam (6.23.6.3) tests. In the case of delay of the above tests, the bag shall be stored in a watertight container (glass bottle or equivalent) to avoid any change in its properties.

Note 13 The sampling of the bag for the dissolution test should be as follows:

 Lay the empty cleaned bag in its original configuration (double layer). Delineate and then cut up a test sample including part of the upper seal (5 cm) and symmetrically including the vertical seal (10 cm). If the size of the bag is less than this dimension, use the whole bag.

 Carry out the dissolution test immediately to avoid any modification of the sample.

Note 14 The procedure for adding the bag material to the solution for the degree of dissolution and solution stability and the persistent foam tests should be as follows:

 "Prepare a stock solution of the bag material (1 mg/ml) by weighing approximately a 100 mg sample (n mg) of the bag (excluding sealed parts) to the nearest mg. Dissolve this sample by stirring in the standard water used for the tests to give a final volume of n ml. Store the stock solution in a stoppered bottle before use. Calculate the volume (V ml) of the stock solution of the bag to be added to the test suspension of the water soluble granule according to the following equation:

 **V(ml) = X *x* 1000B**

 **W**

where: B (g) = weight of the emptied and cleaned bag

 W (g) = nominal weight of the SG contained in the bag

 X (g) = weight of the SG sample used in the test.