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Tebuconazole suspension concentrates

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1 Scope

The standard sets out the requirements for tebuconazole suspension concentrate, the test methods, and the marking, labelling, packaging, storage and transport rules together with the guarantee period.

It applies to tebuconazole suspension concentrate processed from tebuconazole technical material with suitable adjuvants and fillers.

A note refers the reader to Annex A for the other names, the structural formula and the basic physico-chemical parameters of tebuconazole.

2 Normative references

The listed documents are indispensable for the application of the document. For dated references only the edition cited applies; for undated references the latest edition, including all amendments, applies.

The references given are: GB/T 1601 Method for the determination of pH values of pesticides; GB/T 1604 Acceptance rules for commodity pesticides; GB/T 1605-2001 Sampling methods for commodity pesticides; GB 3796 General rules for the packaging of pesticides; GB/T 6682-2008 Water for analytical laboratory use - Specification and test methods; GB/T 8170-2008 Rules of rounding off for numerical values and expression and judgement of limiting values; GB/T 14825-2006 Method for the determination of the suspensibility of pesticides; GB/T 16150 Methods for the determination of the fineness of pesticide dusts and wettable powders; GB/T 19136 Method for the determination of the heat storage stability of pesticides; and GB/T 19137 Method for the determination of the low-temperature stability of pesticides.

3.1 Composition and appearance

The product is to be made from tebuconazole technical material conforming to its standard together with suitable adjuvants and fillers. It is to be a suspension liquid that flows and whose volume can be measured easily. Sediment may appear during storage, but the original state is to be recovered after shaking by hand and no caking is allowed.

3.2 Technical indices

The product is also required to satisfy Table 1, headed control item indices for tebuconazole suspension concentrate.

Table 1 has two indicator columns, headed 430 g/L and 250 g/L. For the 430 g/L grade the tebuconazole mass fraction is 38.0 % with a tolerance of +/-2.0 and the mass concentration at 20 °C is 430 g/L with a tolerance of +/-20; for the 250 g/L grade the corresponding figures are 23.5 % with a tolerance of +/-1.2 and 250 g/L with a tolerance of +/-15.

The remaining rows carry a single value that spans both columns: suspensibility not less than 90 %; pH in the range 5.0 to 8.0; pourability, with residue after pouring not more than 5.0 % and residue after washing not more than 0.5 %; wet sieve test through a 75 μm test sieve not less than 98 %; persistent foam, that is the foam volume after 1 min, not more than 20 mL; and low-temperature stability and heat storage stability both rated as passing.

Two footnotes apply to the table: where the content is in dispute, the mass fraction is the

basis for arbitration; and during normal production, low-temperature stability and heat storage stability are determined at least once every three months.

4.1 General provisions

A safety notice states that people using the standard should have practical experience of laboratory work, that the standard does not point out all the safety problems involved, and that it is the user's responsibility to take suitable safety and health measures and to ensure compliance with the relevant national regulations.

Unless otherwise stated, the reagents and water used in the standard mean analytically pure reagents and grade three water as specified in GB/T 6682-2008. Test results are judged by the rounded value comparison method given in 4.3.3 of GB/T 8170-2008.

4.2 Sampling

Sampling is carried out by the liquid formulation sampling method of GB/T 1605-2001. The packages to be sampled are determined with a random number table, and the final sample quantity is not to be less than 1 000 mL.

4.3 Identification tests

Liquid chromatographic method - this identification test may be carried out at the same time as the determination of the tebuconazole mass fraction. Under the same chromatographic operating conditions, the relative difference between the retention time of a chromatographic peak in the sample solution and the retention time of the tebuconazole peak in the standard solution is to be within 1.5 %.

Gas chromatographic method - this identification test may likewise be carried out at the same time as the determination of the tebuconazole mass fraction. Under the same chromatographic operating conditions, the relative difference between the retention time of a chromatographic peak in the sample solution and the retention time of the tebuconazole peak in the standard solution is to be within 1.5 %.

4.4.1 Determination of the tebuconazole mass fraction by high performance liquid chromatography (referee method)

Outline of the method: the sample is dissolved in methanol and the tebuconazole in it is separated and determined by high performance liquid chromatography on a stainless steel column packed with Nova-Pak C18, with methanol plus water as the mobile phase and an ultraviolet detector at 220 nm.

Reagents and solutions: methanol; water, freshly prepared double-distilled water; and a tebuconazole standard of known mass fraction, not less than 99.0 %.

Apparatus: a high performance liquid chromatograph with a variable wavelength ultraviolet detector; a chromatographic data processor; a chromatographic column of 150 mm by 3.9 mm internal diameter, stainless steel, packed with Nova-Pak C18 5 μm packing or an equivalent column; a filter with a membrane pore size of about 0.45 μm; a 50 μL microsyringe; a 5 μL sample loop; and an ultrasonic cleaner.

Operating conditions: mobile phase, methanol to water 65 to 35 by volume; flow rate 1.0 mL/min; column temperature, room temperature, with the temperature variation not greater than 2 °C; detection wavelength 220 nm; injection volume 5 μL; retention time of tebuconazole about 7.0 min. The standard notes that these parameters are typical and may be adjusted to suit the characteristics of different instruments, and it gives a typical chromatogram of tebuconazole suspension concentrate as Figure 1.

Preparation of the standard solution: 0.1 g of the tebuconazole standard is weighed to the nearest 0.000 2 g into a 50 mL volumetric flask, diluted to the mark with methanol, dissolved by ultrasonic vibration for 5 min, cooled to room temperature and shaken; 5 mL of that solution is transferred with a pipette into a further 50 mL volumetric flask, diluted to the mark with methanol and shaken.

Preparation of the sample solution: a sample containing 0.1 g of tebuconazole is weighed to the nearest 0.000 2 g into a 50 mL volumetric flask, diluted to the mark with methanol, dissolved by ultrasonic vibration for 5 min, cooled to room temperature and shaken; 5 mL of that solution is transferred with a pipette into a further 50 mL volumetric flask, diluted to the mark with methanol, shaken and filtered.

Determination: under the operating conditions above and once the instrument is stable, several injections of the standard solution are made until the relative change between the tebuconazole peak areas of two adjacent injections is less than 1.5 %; the determination then proceeds in the order standard solution, sample solution, sample solution, standard solution.

Calculation: the tebuconazole peak areas of the two sample solution injections and of the standard solution injections made before and after them are averaged separately. Two equations are given, one for the tebuconazole mass fraction of the sample in per cent and one for the mass concentration. Their symbols denote the mass fraction of tebuconazole in the sample in per cent, the mean tebuconazole peak area of the sample solution, the mass of the tebuconazole standard in grams, the mass fraction of the tebuconazole standard in per cent, the mean tebuconazole peak area of the standard solution, the mass of the sample in grams, the mass concentration of tebuconazole in the sample at 20 °C in grams per litre, and the density of the sample at 20 °C in grams per millilitre, which is determined according to Annex B.

Permissible difference: the difference between two parallel determinations of the tebuconazole mass fraction is to be not greater than 0.5 %, and their arithmetic mean is