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Pyridaben technical material

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Foreword

The standard Chapter 3, Chapter 5 is mandatory, the rest are recommended.

This standard was drafted in accordance with GB/T 1.1-2009 given rules.

Please note that some of the content of this document may involve patents. Release mechanism of the present document does not assume responsibility for the identification of these patents.

The standard proposed by China Petroleum and Chemical Industry Association.

This standard by the National Standardization Technical Committee on Pesticides (SAC/TC133) centralized.

1 Scope

This standard specifies the requirements, test methods and marking, labeling, packaging, storage and acceptance of pyridaben the original drug.

This standard applies to the impurities from the PYRIDABEN its production into the composition of pyridaben the original drug.

Note: Other name, structural formula and basic physicochemical parameters pyridaben see Appendix A.

2 Normative references

The following documents for the application of this document is essential. For dated references, only the dated version suitable for use herein Member. For undated references, the latest edition (including any amendments) applies to this document.

Preparation of GB/T 601-2002 Chemical Reagent standard titration solution GB/T 1600 Determination of Water Pesticides GB/T 1604 Goods pesticide regulations for acceptance GB/T 1605-2001 Sampling Method commercial pesticides GB 3796 pesticide packaging General GB/T 19138 Determination of pesticide insoluble in acetone (GB/T 19138-2003, CIPACMT27, MOD)

3.1 Appearance

White granular or powdery solid, no visible foreign matter and filling the modifier.

3.2 Technical Specifications

Pyridaben the original drug should meet the requirements of Table 1.

Table 1 pyridaben the original drug control project indicators

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Pyridaben mass fraction /% ≥ 95.0 Moisture content /% ≤ 0.5 Acidity (H₂SO₄ meter) /% $\leq$ or alkalinity (NaOH meter) /% ≤ 0.3 0.2 Fraction a /% acetone insolubles ≤ 0.5 mass when a normal production, acetone insoluble content was measured at least once every three months.

Test Method 4

4.1 Sampling

According to GB/T 1605-2001 "on the original drug product sampling" approach. Determined by sampling a random number table method packages, the final sample size not less than 100g.

4.2 Identification Test

You can choose one of the following methods. When not identify one way, then use another method should be determined.

Infrared spectroscopy --- sample and pyridaben standard infrared 4000cm⁻¹ ~ 400cm⁻¹ absorption spectrum should be no significant difference.

Pyridaben standard infrared spectrum is shown in Figure 1.

Figure 1 pyridaben standard infrared spectrum

HPLC --- The identification test can be carried out simultaneously with the determination of the mass fraction of pyridaben. In the same chromatographic operating conditions , The sample solution to a chromatographic retention time and pyridaben standard solution PYRIDABEN chromatographic retention time, the relative difference should 1.5% or less.

4.3.1.1 Method summary

Sample was dissolved in methanol, acetonitrile and water as the mobile phase, use as filler in Nova-PakC18 stainless steel column and UV detector (240nm), the sample of PYRIDABEN high performance liquid chromatography and determination.

4.3.1.2 Reagents and solutions

Methanol;

Acetonitrile. chromatographic pure;

Water. The new secondary steam distilled water;

Pyridaben Sample. Known pyridaben mass fraction $w \geq 99.0\%$.

4.3.1.3 Instruments

High performance liquid chromatography. a variable wavelength UV detector;

Chromatographic data processor or workstation;

Column. 150mm x 3.9mm (id) stainless steel column, built-Nova-PakC18,5μm filler (or equally effective column);

Filter. pore size of about 0.45μm;

Micro injector. 50μL;

Quantitative sample line. 5μL;

Ultrasonic cleaner.

4.3.1.4 HPLC operating conditions

Mobile phase. Psi (CH₃CN.H₂O) = 85.15;

Flow rate. 1.0mL/min;

Column temperature. room temperature (temperature change should not exceed 2 °C);

Detection wavelength. 240nm;

Injection volume. 5μL;

Retention time. pyridaben about 5.6min.

It said operating parameters are typical, according to the characteristics of different instruments, given operating parameters adjusted as appropriate, in order to obtain the best results.

Typical pyridaben the original drug HPLC is shown in Figure 2.

1 --- pyridaben.

Figure 2 HPLC pyridaben the original drug

4.3.1.5.1 Preparation of standard solution

Weigh 0.1g PYRIDABEN standard (accurate to 0.0002g), a 50mL volumetric flask, dilute to the mark with methanol, ultrasonic oscillation 5min make sample dissolution, cooled to room temperature and shake well. Using a pipette to take the above solution 5mL to 50mL volumetric flask, diluted with methanol to the mark.

4.3.1.5.2 Preparation of sample solution

Weigh containing pyridaben 0.1g sample (accurate to 0.0002g), a 50mL volumetric flask, dilute to the mark with methanol, ultrasonic Oscillation 5min make sample dissolution, cooled to room temperature and shake well. Using a pipette to take the above solution 5mL to 50mL volumetric flask with methanol Dilute to the mark.

4.3.1.5.3 Determination

Under these operating conditions, after the instrument is stable, continuous injection of several doses of the standard solution until the two adjacent needles PYRIDABEN Lingfeng area relative change After less than 1.2%, according to the solution, the sample solution, the sample solution and standard sequence of the standard solution was measured.

4.3.1.6 Calculation

The two needle sample solution and measured before and after the two doses of the standard sample solution PYRIDABEN Lingfeng area were averaged. Sample PYRIDABEN the mass fraction according to equation (1).

$$w_1 = A_2$$

$$\cdot M_1 \cdot w$$