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Paclobutrazol technical

多效唑原药

(English Translation)

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Foreword

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

The standard proposed by China Petroleum and Chemical Industry Association.

This standard by the National Standardization Technical Committee on Pesticides (CSBTS/T C133) centralized.

1 Scope

This standard specifies the requirements for MET original drug, test methods and marking, labeling, packaging, storage and transportation.

This standard applies to the impurities generated in the production of MET and MET composed the original drug.

2 Normative references

The following documents contain provisions which, through reference in this standard and become the standard terms. For dated references, subsequent Amendments (not including errata content) or revisions do not apply to this standard, however, encourage the parties to the agreement are based on research Whether the latest versions of these documents. For undated reference documents, the latest versions apply to this standard.

Determination of GB/T 1601 Pesticides pH value 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 acetone insoluble pesticide

3 Requirements

3.1

Composition and Appearance. This product should be related to the production of MET and impurities, should be white to tan solid, no visible foreign matter and Filling modifier.

3.2

MET original drug should be consistent with the requirements in Table 1.

Table 1 MET original drug quality control program indicators

Item Index

MET mass fraction /% ≥ 95.0 Loss on drying /% ≤ 0.5 pH range 4.0 to 9.0 Acetone insolubles a /% ≤ 0.5 when a normal production, insoluble in acetone was tested once every three months at least.

Test Method 4

4.1 Sampling

According to GB/T 1605-2001 "on the original drug product sampling" approach. Determining sample package using a random number table method; the final sample size not less than 250g.

4.2 Identification Test

IR spectra of infrared spectroscopy --- sample with MET standard infrared spectra (see Figure 1), there should be no significant difference.

Liquid chromatography --- The identification test can be carried out simultaneously with the determination of MET Quality Score. Under the same chromatographic operating conditions, the test the retention time of the sample solution and the standard solution to a peak in the MET peak retention time, the relative difference should be less than 1.5%.

Gas Chromatography --- The identification test can be carried out simultaneously with the determination of MET Quality Score. Under the same chromatographic operating conditions, the test the retention time of the sample solution and the standard solution to a peak in the MET peak retention time, the relative difference should be less than 1.5%.

Figure 1 standard infrared spectra of MET

Determination of the mass fraction of 4.3 MET

4.3.1.1 Method summary

Sample was dissolved in methanol, - acetonitrile - water as mobile phase, the choice of Nova-pakC18,5 μ m stuffing column and UV variable Wavelength detector to sample the MET liquid chromatography separation and determination.

4.3.1.2 Reagents and solutions

Methanol. HPLC grade;

Acetonitrile. chromatographic pure;

Water. The new secondary steam distilled water;

MET standard. a known mass fraction $\geq 99.0\%$.

4.3.1.3 Instruments

LC. a variable wavelength UV detector and quantitative injection valve;

Chromatographic data processor or chromatography workstation;

Column. 3.9mm (id) x 150mm stainless steel columns, built-Nova-pakC18,5 μ m filler (or a column with the same efficiency He reversed-phase column);

Ultrasonic bath;

Micro Injector. not less than 50 μ L.

4.3.1.4 LC operating conditions

Mobile phase. psi (CH₃OH.CH₃CN.H₂O) = 35.20.45;

The mobile phase flow rate. 1.0mL/min;

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

Detection wavelength. 230nm;

Injection volume. 10 μ L;

Retention time (min). 4-H 3.8 MET, MET II body 6.3, 7.7 MET, chlorine trazodone 9.6.

HPLC operating conditions described above, the Department of typical operating parameters. According to the characteristics of different instruments, given operating parameters adjusted as appropriate, in order to obtain the best results. Typical liquid chromatogram MET original drug is shown in Figure 2.

1 --- 4-H MET;

2 --- MET II body;

3 --- MET;

4 --- chlorine trazodone.

Figure 2 MET liquid chromatogram of the original drug

4.3.1.5.1 Preparation of standard solution

Weigh MET standard 0.10g (accurate to 0.0002g), a 50mL volumetric flask, add 40mL of methanol, in an ultrasonic Bath sonication 5min. Remove cooled to room temperature, add methanol to volume, shake; pipette imbibe 5mL, set another 50mL volumetric flask, add methanol to volume, shake.

4.3.1.5.2 Preparation of the sample solution

Weigh the sample 0.10g (accurate to 0.0002g), a 50mL volumetric flask, add 40mL of methanol, in an ultrasonic bath Sonication 5min. Remove cooled to room temperature, add methanol to volume, shake; pipette imbibe 5mL, set another 50mL Volumetric flask, add methanol to volume, shake.

4.3.1.5.3 Determination

Under the above chromatographic operating conditions, after the instrument is stable, continuous injection of several doses of the standard solution until the two adjacent needles MET relative peak area After the change is less than 1.0%, according to the order of the standard solution, sample solution, sample solution, standard solution injection analysis.

4.3.1.6 Calculation

The two needle sample solution measured before and after the peak area of the sample and two doses of the standard solution MET are averaged. Sample pleiotropic Azole mass fraction of 1 (%) according to equation (1).

Where.

A1 --- the average peak area of standard solution MET;

The average peak area A2 --- MET in the sample solution;

--- Paclobutrazol standard mass fraction, expressed as a percentage.

4.3.1.7 Allowable difference

The difference between two parallel determination results shall not be greater than 1.2%, the arithmetic mean value as a measurement result.