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Imidacloprid technical material

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(English Translation)

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5.1 Marking, labeling, packaging
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Foreword

This standard in Chapter 3, Section 5 is mandatory, the rest are recommended.

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

This draft standard method using the reformed modified using FAOSpecification582/TC (May2006) "imidacloprid" the main technical differences and the reasons are as follows.

--- This increases the control over the standard loss on drying, pH value range, dimethylformamide insolubles, more comprehensive index item, the FAO Specifications not control these three indicators.

Please note that some of the content of this document may involve patents. The issuing authority of this document does not assume responsibility to identify these patents.

The standard proposed by China Petroleum and Chemical Industry Association.

This standard by the National Pesticide Standardization Technical Committee (SAC/TC133).

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1 Scope

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

This standard applies to imidacloprid and imidacloprid impurities generated in the production thereof.

Note: The other name, structural formula and basic physicochemical parameters of imidacloprid see Appendix A.

2 Normative references

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

Determination 1601 pH value pesticides GB/T GB/T 1604 pesticide regulations for acceptance of goods GB/T 1605-2001 sampling commercial pesticides GB 3796 pesticide packaging General

3.1 Appearance

White to beige powder, without visible foreign matter.

3.2 Technical Specifications

Imidacloprid should also meet the requirements of Table 1.

Table 1 imidacloprid Control Project Index

Item Index

Imidacloprid content /% ≥ 97.0 Loss on drying /% ≤ 0.5 the pH range of 5.0 to 8.0
Dimethylformamide insolubles mass fraction a /% ≤ 0.2 when a normal production,
dimethylformamide insolubles content was measured at least once every three months.

Test Method 4

4.1 Sampling

Carried out in GB/T 1605-2001 of "original drug product sampling" approach. Determining Sample packages with a random number table method; final sample size not less than 100g.

4.2 Identification Test

The present liquid chromatography --- identification test can be conducted simultaneously with the determination of the mass fraction of imidacloprid. Chromatography under the same operating conditions, the test Main peaks in the retention time of the sample solution and the standard solution imidacloprid retention time of the peaks, the relative difference should be less than 1.5%.

Infrared spectroscopy --- imidacloprid standard sample and the infrared 4000cm⁻¹ ~ 400cm⁻¹ absorption spectrum range should not significantly the difference. Imidacloprid standard infrared spectra of Figure 1.

FIG 1 imidacloprid standard IR spectrum

4.3.1 Method summary

Sample dissolved in mobile phase, methanol and water as mobile phase, used as a filler C18 stainless steel column and UV detector wavelength at 260nm, of the sample imidacloprid RP-HPLC separation and determination, external standard.

4.3.2 Reagents and solutions

Methanol. HPLC grade;

Water. The new secondary steam distilled water;

Imidacloprid Sample. A known mass fraction $w \geq 99.0\%$.

4.3.3 Instruments

HPLC. a variable wavelength UV detector;

Chromatographic data processor or chromatography workstation;

Column. 250mm x 4.6mm (id) stainless steel column, the contents C18, 5μm filler;

Filter. membrane pore size of about 0.45 μm;

Microsyringe. 50μL;

Quantitative sampling tube. 5μL;

Ultrasonic cleaning.

4.3.4 HPLC operating conditions

Mobile phase. [Psi] (methanol. water) = 40.60, by membrane filtration, and degassing;

Flow rate. 0.8mL/min;

Column temperature. room temperature (temperature change should be less than 2 deg.] C);

Detection wavelength. 260nm;

Injection Volume. 5μL;

Retention time. imidacloprid 6.0min.

Are typical operating parameters of the above, according to the characteristics of different instruments, for a given operating parameters appropriately adjusted to obtain the best result.

Typical HPLC imidacloprid shown in Figure 2.

1 --- imidacloprid.

FIG 2 FIG imidacloprid original drug HPLC

4.3.5.1 Preparation of standard solution

0.1 g of weighed (accurate to 0.0002g) imidacloprid in Sample 100mL volumetric flask, dissolved in methanol and diluted to the mark, ultrasonic 5min shaken to dissolve the sample, cooled to room temperature, shaking. Pipette with a pipette 5mL above solution in 50mL volumetric flask, with the mobile Phase was diluted to the mark.

4.3.5.2 Preparation of sample solution

Containing 0.1 g of imidacloprid was weighed (accurate to 0.0002g) sample in 100mL volumetric flask, dissolved in methanol and diluted to the mark, ultra 5min sonication to dissolve the sample, cooled to room temperature, shaking. Pipette with a pipette 5mL above solution in 50mL volumetric flask, with Mobile phase and dilute to the mark.

4.3.5.3 Determination

Under the above operating conditions, after the instrument is stable, the number of consecutive doses of the standard solution is injected until the two adjacent pins relative change in peak area imidacloprid After less than 1.2%, measured according to the standard solution, sample solution, the sample solution, the order of standard solution.

4.3.5.4 Computing

The two needle and the sample solution was measured before and after the two doses of the standard sample solution and peak areas were averaged imidacloprid. Imidacloprid sample Mass fraction by formula (1).

$$w_1 = A_2$$