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Prometryn technical

扑草净原药

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Foreword

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

Appendix A of this standard is an informative annex.

Since the implementation of this standard, the original industry standard HG2201-1991 "Prometryne original drug" void.

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 Prometryne original drug requirements, test methods and marking, labeling, packaging, storage and transportation.

This standard applies to the impurities generated by its production Prometryne composed Prometryne 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.

Preparation of GB/T 601 chemical reagent standard titration solution 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

Appearance. white to off-white powder.

3.2

Prometryne original drug should meet the requirements of Table 1.

Table 1 Prometryne original drug control project indicators

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Prometryne mass fraction /% ≥ 96.0 Loss on drying /% ≤ 1.0 Sodium chloride mass fraction a /% ≤ 1.0 Dimethylformamide insolubles a /% ≤ 0.5 a mass fraction of sodium chloride, dimethylformamide insolubles in normal production, 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 method". Determining sample package using a random number table method; the final sample the amount of not less than 100g.

4.2 Identification Test

Gas Chromatography --- The identification test can be carried out simultaneously with the determination of the mass fraction of Prometryne. Under the same chromatographic operating conditions, the test Retention time with the standard solution of a sample solution peaks in Prometryne chromatographic retention time, the relative difference should be less than 1.5%.

Infrared spectroscopy --- sample and standard samples Prometryne in 4000cm⁻¹ ~ 400cm⁻¹ wave number range of the infrared spectrum should not significantly Differences. Prometryne Sample standard infrared spectrum shown in Figure 1.

Figure 1 Prometryne standard infrared spectrum

4.3.1 Method summary

Sample was dissolved in chloroform to triadimefon as internal standard, the use of the inner wall of the bonded Carbowax 20M capillary column and flame Ionization detector to sample Prometryne gas chromatographic separation and determination. You can also use packed column gas chromatography, chromatographic conditions Senate See Appendix A.

4.3.2 Reagents and solutions

Chloroform;

Triadimefon. should not interfere with the analysis of impurities, ≥95%;

Prometryne Sample. Known Prometryne mass fraction, ≥99.0%;

Internal standard solution. Weigh 8.6g triadimefon, placed 1000mL volumetric flask with chloroform to dissolve and dilute to volume, shake.

4.3.3 Instruments

Gas chromatograph. with a hydrogen flame ionization detector;

Chromatographic data processor or chromatography workstation;

Column. 30m x 0.32mm (. I.d) capillary column, the inner wall of the bonding polyethylene glycol 20M, film thickness 0.25 m;

Micro injector. 10μL.

4.3.4 GC operating conditions

Temperature (°C). 195 Column temperature gasification chamber 230, detection chamber 230;

Gas flow rate (mL/min). carrier gas (N₂) 2.0, hydrogen gas 30, air 300;

Injection volume (μL). 1.0;

Retention time (min). Prometryne about 4.2, internal standard (triadimefon) about 5.6.

GC 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 Prometryne original drug

and the internal standard gas chromatography is shown in Figure 2.

1 --- Prometryne;

2 --- internal standard (triadimefon).

Figure 2 Prometryne gas chromatogram of the drug and the internal standard

4.3.5.1 Preparation of standard solution

Weigh Prometryne standard 0.1g (accurate to 0.0002g), placed in 15mL stoppered glass bottle with a pipette moved 10mL internal standard Solution, shake.

4.3.5.2 Preparation of the sample solution

It weighs about 0.1g sample containing Prometryne of (accurate to 0.0002g), placed in 15mL stoppered glass bottle, in use with 4.3.5.1 with a pipetted into internal standard solution 10mL, shake.

4.3.5.3 Determination

Under these operating conditions, the instrument until a stable baseline, the number of continuous injection doses of the standard solution, calculated for each needle Prometryne and the internal standard peak area Repeatability ratio, when two needles Prometryne relative change compared with the internal standard peak area of less than 1.2%, according to the standard solution, sample to be adjacent Solution, sample solution, the order of the standard solution was measured.

4.3.6 Calculation

The two needle sample solution and measured before and after the two doses of the standard sample solution Prometryne and the internal standard peak area ratio in the average respectively.

Prometryne sample mass fraction of 1 (%), according to equation (1).

Where.

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

4.3.7 Allowable difference

The difference between two parallel determination results shall not be greater than 1.5%, the arithmetic mean value as a measurement result.