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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

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Buprofezin technical

噻嗪酮原药

(English Translation)

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Foreword

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

From the date of implementation of this standard, HG2463.1-1993 "buprofenzin original drug" abolished.

Appendix A of this standard is an informative annex.

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

This standard applies to the impurities generated by the buprofezin and production consisting of buprofezin 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 603 chemical reagent in test methods - Preparations with 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. crystalline powder should be white to pale yellow or white, no visible foreign matter.

3.2

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

Table 1 bupropfenzin original drug quality control program indicators

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Bupropfenzin mass fraction /% ≥ 97.0 Loss on drying /% ≤ 0.3 Acidity (H₂SO₄ dollars) or alkalinity (NaOH meter) /% ≤ 0.1 Acetone insolubles a /% ≤ 0.2 a insoluble in acetone under normal production conditions, 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. Determining sample package using a random number table method; the final sample size not less than 100g.

4.2 Identification Test

Infrared spectroscopy --- sample and standard samples in the IR 4000cm⁻¹ ~ 400cm⁻¹ Scope absorption spectrum should be no significant difference. Thiophene Piperazinone standard infrared spectrum is shown in Figure 1.

Figure 1 bupropfenzin standard infrared spectrum

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

4.3.1 Method summary

Sample was dissolved in chloroform, dicyclohexyl phthalate esters as internal standard, using the 5% HP-5 capillary column coated walls and hydrogen Flame ionization detector, the sample of buprofezin gas chromatography separation and determination. You can also use packed column gas chromatography, strip Member See Appendix A.

4.3.2 Reagents and solutions

Chloroform;

Buprofezin standard. a known mass fraction, $\geq 98.0\%$;

Dicyclohexyl phthalate esters. interference analysis should not contain impurities;

Internal standard solution. Weigh 4.0g dicyclohexyl phthalate esters, placed in 500mL flask with chloroform dissolved and diluted to the mark Degree, 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, wall coated HP-5 (5% + 95% diphenyl dimethyl silicone), the film thickness 0.25 μ m;

Micro injector. 10 μ L.

4.3.4 GC operating conditions

Temperature ($^{\circ}$ C). 230 column temperature, the gasification chamber 270, the detector chamber 280;

Gas flow rate (mL/min). carrier gas (N₂) 2.0, hydrogen gas 30, air 300, the compensation gas (N₂) 25;

Split ratio. 20.1;

Injection volume (μ L). 1.0;

Retention time (min). 3.8 buprofezin, internal standard 7.0.

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 bupropfen original drug capillary column gas chromatography is shown in Figure 2.

1 --- solvent;

2 --- bupropfen;

3 --- internal standard (phthalate, dicyclohexyl phthalate).

Figure 2 bupropfen original drug and the internal standard capillary column gas chromatogram

4.3.5.1 Preparation of standard solution

Weigh bupropfen standard 0.08g (accurate to 0.0002g) in 15mL stoppered glass bottle with a pipette accurately added 10mL within Standard solution, shake.

4.3.5.2 Preparation of the sample solution

Weigh containing bupropfen 0.08g (accurate to 0.0002g) sample in 15mL stoppered glass bottle with a pipette and 4.3.5.1 with Tube accurately added 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 until the two needles bupropfen and the internal standard peak adjacent when the area ratio changes by less than 1.2%, according to the solution, the sample solution, the sample solution and standard sequence of the standard solution was measured.

4.3.6 Calculation

The two needle sample solution and measured before and after the two doses of the standard sample solution piperazinone and thiophene internal standard peak area ratios are averaged. test 1 sample mass fraction (%) of bupropfen according to formula (1).

Where.

--- Standard samples bupropfen mass fraction, expressed as a percentage.

4.3.7 Allowable difference

Thiophene-one hydrochloride poor quality score measurement results of two parallel, should not exceed 1.2%, and the arithmetic mean as a measurement result.