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Fluoroglycofen-ethyl technical material

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(English Translation)

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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 B fluoroglycofen original drug requirements, test methods and marking, labeling, packaging, storage and acceptance period.

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This standard applies to the impurities generated by Fluoroglycofen ether acetate composition and production fluoroglycofen original drug.

Note: Other name, structural formula and basic physicochemical parameters Fluoroglycofen ether in 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 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

3.1 Appearance

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

3.2 Technical Specifications

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

Table 1 B fluoroglycofen original drug quality control program indicators

Item Index

Fluoroglycofen ether mass fraction /% >= 95.0 Loss on drying /% <= 0.5 Acidity (H2SO4 meter) /% <= 0.5 Acetone insoluble mass fraction /% <= 0.3 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 standard samples at 4000cm-1 ~ 400cm-1 Scope of the infrared absorption spectrum should be no significant difference. Mark Like infrared spectrum is shown in Figure 1.

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Figure 1 B fluoroglycofen standard infrared spectrum

HPLC --- The identification test can be carried out simultaneously with the determination of ethyl fluoroglycofen mass fraction. In the same chromatographic operating conditions Under parts, the sample solution to a chromatographic retention time and Fluoroglycofen ethyl ether standard solution fluoroglycofen chromatographic retention time, relative of the difference should be less than 1.5%.

4.3.1.1 Method summary

Sample was dissolved in chloroform, dicyclohexyl phthalate esters as internal standard, using a HP-5 fused silica capillary column coated walls, and hydrogen fire Flame ionization detector, a sample with acetic fluoroglycofen capillary gas chromatography and determination.

4.3.1.2 Reagents and solutions

Chloroform;

B fluoroglycofen standard. a known mass fraction w>=99.0%;

Dicyclohexyl phthalate ester. not interfere with the analysis of impurities;

Internal standard solution. Weigh 7.5g dicyclohexyl phthalate esters into 1000mL volumetric flask, dissolved with chloroform, constant volume, Shake well.

4.3.1.3 Instruments

Gas chromatograph. with flame ionization detector;

Column. 30m x 0.32mm (id) quartz capillary column, wall coated HP-5, film thickness 0.25μm;

Chromatographic data processor or chromatography workstation.

4.3.1.4 GC operating conditions

Temperature (°C). Room 230 column, the gasification chamber 260, detection chamber 280;

Gas flow rate (mL/min). carrier gas (N2) 1.8, hydrogen gas 30, air 300, the compensation gas 25;

Split ratio. 30.1;

Injection volume. 1.0μL;

Retention time. Fluoroglycofen ether about 10.8min, internal standard about 7.9min.

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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 B fluoroglycofen original drug and the internal standard gas chromatography is shown in Figure 2.

1 --- internal standard;

2 --- Fluoroglycofen ether.

Figure 2 B fluoroglycofen original drug and the internal standard gas chromatograph

4.3.1.5.1 Preparation of standard solution

Weigh Fluoroglycofen ether standard 0.05g (accurate to 0.0002g), placed in a glass bottle stopper, with a pipette accurately added 5mL within Standard solution, shake.

4.3.1.5.2 Preparation of the sample solution

Weigh containing ethyl fluoroglycofen 0.05g of the sample (accurate to 0.0002g), placed in a glass bottle stopper, and with the 4.3.1.5.1 with the use of a pipette accurately added internal standard solution 5mL, 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 Fluoroglycofen ether and the internal standard After the relative change of the peak area ratio was less than 1.5%, according to the solution, the sample solution, the sample solution and standard sequence analysis of the standard solution Determination.

4.3.1.5.4 Calculation

The measured before and after the two-needle sample solution and two doses of the standard sample solution Fluoroglycofen ether and the internal standard peak area ratios are flat all. Sample B fluoroglycofen content according to equation (1).

w1 = r2 x m1 x wr1 x m2 (1) where.

Quality w1 --- sample Fluoroglycofen ether fraction, in%;

r2 --- sample solution Fluoroglycofen ether and the average internal standard peak area ratio;

m1 --- standard mass in grams (g);

w --- standard samples Fluoroglycofen ether mass fraction, expressed as a percentage;

r1 --- standard samples Fluoroglycofen ether and the average peak area ratio of the internal

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