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Carbosulfan Technical

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(English Translation)

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Foreword

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

This modification uses standard FAO Specification 417/T C/S/F (1991) "carbosulfan TC" (in English).

This standard modification method using foreign advanced standards for the redrafted law. This standard FAO Specifications "carbosulfan TC" major technical differences.

- 1) The standard carbosulfan quality score is $\geq 90\%$, FAO specifications $\geq 890\text{g/kg}$ ($\geq 89\%$).
- 2) The standard control carbofuran impurity content is $\leq 0.2\%$, FAO specifications $\leq 2\text{g/kg}$ ($\leq 0.2\%$).
- 3) The control standard acetone insoluble impurity content, FAO specifications without the indicators.
- 4) the actual quality of our products overall situation, alkalinity index of $\leq 0.05\%$, while FAO specifications for $\leq 0.2\text{g/kg}$ ($\leq 0.02\%$). The standard proposed by China Petroleum and Chemical Industry Association.

2 Normative references

Terms of the following documents become provisions of this standard by reference in this standard. For dated reference documents, all later Amendments (not including errata content) or revisions do not apply to this standard, however, encourage the parties to agreements based on this standard Whether the latest versions of these documents. For undated references, the latest version applies to this standard. Preparation GB/T 601 standard titration solution Chemicals

GB/T 1600-2001 Pesticide moisture determination method

GB/T 1604 pesticide regulations for acceptance of goods

GB/T 1605-2001 sampling commercial pesticides

GB 3796 pesticide packaging General

GB/T 19138 Determination of acetone insoluble pesticide

3.1 Appearance

This product should be a brown viscous liquid.

3.2 Technical Specifications

Carbosulfan original drugs should meet the requirements of Table 1.

4.1 Sampling

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

4.2 Identification Test

HPLC --- This identification test can be carried out simultaneously with the measurement of the content of carbosulfan. In the same chromatographic operating conditions Next, a peak retention time of a sample solution and the standard solution carbosulfan peak retention time, which should be in the relative difference 1.5% or less.

Infrared Spectroscopy Infrared --- sample and standard sample in the range of 4000cm⁻¹ ~ 400cm⁻¹ absorption spectrum should be no significant difference. Ding Carbosulfan standard infrared spectra of Figure 1.

FIG 1 carbosulfan standard infrared spectra Determination of the mass fraction of 4.3 carbosulfan (carbofuran)

4.3.1 Method summary

Sample was dissolved in methanol, methanol + water as the mobile phase, used as a filler AgilentTC-C18 stainless steel column and UV detector (280 nm), the sample carbosulfan (carbofuran) reverse phase high performance liquid chromatography, external standard.

4.3.2 Reagents and solutions

Methanol. HPLC grade; Water. The new secondary steam distilled water; Carbosulfan Sample. A known mass fraction $\geq 98.0\%$. Carbofuran Sample. A known mass fraction $\geq 99.0\%$.

Carbofuran Preparation of standard solution. Weigh 0.05 g of carbofuran standard (accurate to 0.0002g) was placed in a 50mL volumetric flask, washed with methanol Dissolved and diluted to the mark.

4.3.3 Instruments

HPLC. a variable wavelength UV detector; Chromatographic data processor or workstation;

Column. 250mm x 4.6mm (i.d.) Stainless steel column, built AgilentTC-C18,5 μ m filler (column or with equivalent effect); Filter. membrane pore size of about 0.45 μ m;

Microsyringe. 50 μ L; Quantitative sampling tube. 5 μ L; Ultrasonic cleaning.

4.3.4 HPLC operating conditions

Mobile phase. [Psi] (methanol. water) = 88.12, by membrane filtration, and degassing; Flow rate. 1.0mL/min;

Column temperature. room temperature (temperature change should be less than 2 deg.] C); Detection wavelength. 280nm; Injection Volume. 5 μ L; Retention time. 3.6min carbofuran, carbosulfan 13.8min.

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. A typical carbosulfan HPLC TC shown in Figure 2.

1 --- carbofuran;

2 --- carbosulfan.

FIG 2 FIG carbosulfan HPLC original drug

4.3.5.1 Preparation of standard solution

Weigh 0.1 g of carbosulfan standard (accurate to 0.0002g), a 50mL volumetric flask, dissolve and dilute to volume with methanol, Shake well. Pipette accurately pipetted solution is placed above another 10mL 50mL volumetric flask, and then another 2mL pipette take g Budweiser standard solution in the same volumetric flask, dilute to volume with methanol, shake.

4.3.5.2 Preparation of sample solution

Weigh containing 0.1 g of carbosulfan (accurate to 0.0002g) sample, placed in a 50mL volumetric flask, dissolved in methanol and diluted to the mark with Degree, shake. Pipette accurately pipetted solution is placed above another 10mL 50mL volumetric flask, diluted with methanol 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 needle carbosulfan two adjacent opposing peak area After the change is less than 1.0%, measured according to

the standard solution, sample solution, the sample solution, the order of standard solution.

4.3.6 Computing

The two doses of the standard peak area was measured before and after the two needles in the sample solution and a sample carbosulfan (carbofuran peak area) were Averaged. Sample carbosulfan (carbofuran) is a mass fraction (%), according to equation (1). (1) Where.

A1 --- the standard solution, a peak area carbosulfan (carbofuran peak area) is calculated;

A2 --- sample solution, carbosulfan peak area (carbofuran peak area) is calculated;

--- standard samples carbosulfan (carbofuran) mass fraction, expressed in%;

4.3.7 Allows poor

The measurement results of the mass fraction of carbosulfan two parallel should be less than 1.5%, mass fraction of parallel measurement results twice carbofuran The difference should not be more than 0.2%, and the arithmetic mean value as a measurement result.

4.4 Determination of acetone insolubles

GB/T 19138 Determination press.

4.5 Determination of Moisture

According to the GB/T he 1600-2001 "Karl Fischer method" T "2.1.2 Karl Fischer - coulometric titration instrument measurement method".

4.6.1 Reagents and solutions

Volume fraction of 95% ethanol;

HCl Standard Solution Ba (HCl) = 0.02mol/L, according to GB/T 601 and calibration formulation; Methyl red ethanol solution. rho (methyl red) 2g/L; Bromocresol green ethanol solution. rho (Bromocresol Green) 1g/L;

Mixed indicator. [Psi] (methyl red ethanol solution. bromocresol green ethanol solution) = 1.3.

4.6.2 Determination Step

2g sample was weighed (accurate to 0.0002g), placed in a 250mL Erlenmeyer flask, 95% ethanol was added 100mL, shaking the sample Dissolved. Add 5 drops of mixed indicator, titration with a standard solution of hydrochloric acid titration, the solution turns from green to yellow is the end. Meanwhile blank Determination.