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CCS G25

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 24753-2009

Isocarbophos technical

水胺硫磷原药

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Foreword

The standard Chapter 3, Chapter 5 is mandatory, the rest are recommended. The standard proposed by China Petroleum and Chemical Industry Association. This standard by the National Standardization Technical Committee on Pesticides (SAC/TC133) centralized. This standard is drafted by: Shenyang Chemical Research Institute. Participated in the drafting of this standard. Hubei cents Lung Chemical Co., Ltd., Hebei Weiyuan Biological and Chemical Corporation. The main drafters of this standard. Jiangmin Yi, in bright, Zhang Xue, Yang Jinrong, Ko Wing Man. Isocarbophos original drug Other name, structural formula and basic physicochemical parameters of the active ingredients of the product Isocarbophos as follows: ISO common name. Isocarbophos CAS Registry Number. 24353-61-

5 Chemical Name. O- methyl -O- (2- isopropoxy-formylphenyl) Thioxophosphamide Structure. Empirical formula. C11H16NO4PS Molecular Weight. 288.0 (according to 2003 international relative atomic mass) Biological activity. insecticide, acaricide Melting point. 45 °C ~ 46 °C Solubility. soluble in alcohol, ether, benzene, acetone and ethyl acetate, insoluble in water and petroleum ether Stability. stable under neutral and acidic conditions, the case of alkali easy to decompose

1 Scope

GB 24753-2009 is the Chinese national standard on isocarbophos technical, in the field of agriculture. It carries no /T suffix, which in the Chinese system means compliance is mandatory: a product or a practice within its scope has to meet it to be lawfully made, sold or carried out in China. It was issued on 30 November 2009 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 July 2010. Classification: ICS 65.100.10, CCS G25. This page is published from the official record of

the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This standard specifies the Isocarbophos original drug requirements, test methods and marking, labeling, packaging, storage and transportation. This standard applies to the impurities generated by Isocarbophos composition and production Isocarbophos 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 1600 Determination of Water Pesticides

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 pale yellow crystals.

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

1 Isocarbophos original drug quality control program indicators Item Index Isocarbophos mass fraction /% >=

95.0 Moisture /% <=

0.3 Acetone insolubles a /% <=

0.5 Acidity (H2SO4 meter) /% <=

0.3 Insoluble in acetone measured at least once every 3 months a normal production. 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

--- Isocarbophos sample and standard samples in the range 4000cm-1 ~ 400cm-1 infrared absorption spectrum should not significantly the difference. Isocarbophos standard infrared spectrum is shown in Figure 1. Figure

4.3.1 High performance liquid chromatography (Arbitration Act)

4.3.1.1 Method summary Sample was dissolved in methanol, methanol + water as the mobile phase, to use as filler BDSHypersilC18 stainless steel column and UV detector (225nm), the sample Isocarbophos high performance liquid chromatography and determination.

4.3.1.2 Reagents and solutions Methanol; Water. The new secondary steam distilled water; Isocarbophos standards. a known mass fraction, >=97.5%.

4.3.1.3 Instruments High performance liquid chromatography. a variable wavelength UV detector; Chromatographic data processor or workstation; Color 150mm x 4.6mm (i.d.) Stainless steel column, built BDSHypersilC185μm filler (or the equivalent effect. Column The column); Filter. filter pore size of about 0.45μm; Micro injector. 50μL; Quantitative sample line. 5μL; Ultrasonic cleaner.

4.3.1.4 HPLC operating conditions Mobile phase. psi (CH3OH.H2O) = 60.40; Flow rate. 1.0mL/min; Column temperature. room temperature (temperature change should not exceed 2 °C); Detection wavelength. 225nm; Injection volume. 5μL; Retention time. Isocarbophos about 5.6min. It said operating parameters are typical, according to the characteristics of different instruments, given operating parameters adjusted as appropriate, in order to obtain the best results. Typical Isocarbophos original drug HPLC is shown in Figure 2. 1

--- Isocarbophos. Figure 2 HPLC Isocarbophos original drug

4.3.1.5 Measurement procedure 4.3.1.5.1 Preparation of standard solution Weigh 0.1g Isocarbophos standard (accurate to 0.0002g), a 50mL volumetric flask, dilute to the mark with methanol, ultrasonic transducer Dang 5min to dissolve the sample was cooled to room temperature and shake well. Using a pipette to take the above solution 5mL to 50mL volumetric flask, dilute with methanol Release to the mark. 4.3.1.5.2 Preparation of sample solution Weigh aqueous Isocarbophos 0.1g sample (accurate to 0.0002g), a 50mL volumetric flask, dilute to the mark with methanol, ultrasonic Wave oscillation 5min make sample dissolution, cooled to room temperature and shake well. Using a pipette to take the above solution 5mL to 50mL volumetric flask A Alcohol diluted to the mark, shake.

4.3.1.6 Determination Under these operating conditions, after the instrument is stable, continuous injection of several doses of the standard solution until the two adjacent needles Isocarbophos peak area becomes relatively After less than 1.2% of the solution in

accordance with the sample solution, the sample solution and standard sequence of the standard solution was measured.

4.3.1.7 Calculation 1 sample mass fraction (%) Isocarbophos, according to equation (1). 1 = A2 (1) Where. A1

--- the standard solution, the average Isocarbophos peak area; A2

--- sample solution, the average Isocarbophos peak area;

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

4.3.1.8 allowable difference Isocarbophos poor quality score of two parallel determination results shall not be greater than 1.2%, the arithmetic mean value as a measurement result.

4.3.2 Gas Chromatography

4.3.2.1 Method summary Sample was dissolved in ethyl acetate, n-butyl sebacate to as the internal standard, using the 5% OV-3 packed column, and a hydrogen flame ionization detector , A sample of Isocarbophos gas chromatographic separation and determination.

4.3.2.2 Reagents and solutions Ethyl acetate; N-butyl sebacate. Do not interfere with the analysis of impurities; Isocarbophos standards. a known mass fraction, >=97.5%; Internal standard solution. Weigh 5g of n-butyl sebacate in 250mL volumetric flask, dissolved in ethyl acetate and diluted to the mark, Shake; Fixative. OV-3; Carrier. 101 silanized carrier body (180μm ~ 250μm).

4.3.2.3 Instruments Gas chromatograph. with flame ionization detector; Chromatographic data processor or chromatography workstation; Column. 1m x 3mm (. I.d) glass column (or stainless steel column), built-in 5% OV-3/101 silylation white support body (180μm ~ 250

4.3.2.4 GC operating conditions Temperature (°C). Room 210 column, the gasification chamber 270, detection chamber 270; Gas flow rate (mL/min). carrier gas (N2) 60, hydrogen gas 40, air 400; Injection volume. 0.6μL; Retention time. Isocarbophos about 6.1min, internal standard about 9.5min. 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. A typical gas chromatogram Isocarbophos original drug and the internal standard are shown in Figure 3. 1

--- Isocarbophos; 2

--- internal standard. Figure

4.6 Determination of acidity

4.6.1 Reagents and solutions Ethanol; Sodium hydroxide standard titration solution Ba (NaOH) = 0.02mol/L, according to GB/T 601 preparation and calibration; Bromophenol blue