

ICS 65.100.10

CCS G25



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

GB 23550-2009

35% Isocarbophos emulsifiable concentrates

35%水胺硫磷乳油

Issued on: April 27, 2009

Implemented on: November 1, 2009

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of the People's Republic of China;
Standardization Administration of China**

Table of Contents

- 1 Scope
- 2 Normative references
- 3 Requirements
- 4.2 Identification Test Liquid chromatography
- 4.3 Determination of the mass fraction of Isocarbophos
 - 4.3.1 High performance liquid chromatography (Arbitration Act)
 - 4.3.2 Gas Chromatography
- 4.5 Determination of acidity

Foreword

The standard Chapter 3, Chapter 5 are mandatory, the rest are recommended. Since the implementation date of this standard, the former Chemical Industry Standard HG2199-1991 "Isocarbophos EC" void. 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, the British prime times. 35% Isocarbophos EC 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 23550-2009 is the Chinese national standard on 35% isocarbophos emulsifiable concentrates, 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 27 April 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 November 2009. 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 35% Isocarbophos EC requirements, test methods and marking, labeling, packaging, storage and transportation. This standard applies to the original drug Isocarbophos formulated with an emulsifier dissolved in a suitable solvent from 35% Isocarbophos EC.

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 1603 Determination of emulsion stability of pesticide

GB/T 1604 Goods pesticide regulations for acceptance

GB/T 1605-2001 Sampling Method commercial pesticides

GB 4838 EC pesticide packaging

GB/T 19136 Determination of Pesticide Thermal storage stability

GB/T 19137 Pesticide cryogenic stability Determination

3 Requirements

3.1 Composition and appearance This product should be compliant with the original drug Isocarbophos formulated emulsifier dissolved in a suitable solvent, should be stable homogeneous liquid, No visible suspended matter and sediment.

3.2 Technical Specifications 35% Isocarbophos EC shall comply with the requirements of Table 1. Table 1 35% Isocarbophos EC quality control program indicators

Item	Index
Isocarbophos mass fraction	$35.0 \pm 1.8-1.8\%$
Moisture	$\text{/\%} \leq$
Acidity (H ₂ SO ₄ meter)	$\text{/\%} \leq$

0.5 Acidity (H₂SO₄ meter) $\text{/\%} \leq$

0.3 Emulsion stability (diluted 200 times) Qualified Low temperature stability a qualified Thermal storage stability of a qualified a normal production of low-temperature storage stability and thermal stability test when measured at least once every three months. Test Method 4

4.1 Sampling According to GB/T 1605-2001 in "liquid formulations sampling" approach. Determining sample package using a random number table method; the final sample size Not less than 250mL.

4.2 Identification Test Liquid chromatography

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

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 $\geq 99.0\%$.

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 BDSHypersilC18 $5\mu\text{m}$ filler (or the equivalent effect. Column The column); Filter. filter pore size of about $0.45\mu\text{m}$; Micro injector. $50\mu\text{L}$; Quantitative sample line. $5\mu\text{L}$; Ultrasonic cleaner.

4.3.1.4 HPLC operating conditions Mobile phase. $\phi(\text{CH}_3\text{OH}:\text{H}_2\text{O}) = 60:40$; Flow rate. 1.0mL/min ; Column temperature. room temperature (temperature change should not exceed 2°C); Detection wavelength. 225nm ; Injection volume. $5\mu\text{L}$; 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. A typical 35% Isocarbophos EC HPLC is shown in Figure 1. 1

--- Isocarbophos. 1 35% Isocarbophos EC HPLC diagram

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 0.3g (accurate to 0.0002g) Isocarbophos sample, placed in 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.

4.3.1.6 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). $1 = \frac{A_1}{A_2}$ Where. A_1

- the standard solution, the average Isocarbophos peak area; A_2
- 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 0.5%, 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 $\geq 99.0\%$; Internal standard solution. Weigh 5g-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 (180 μ m ~ 250 μ m).

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

m) filler. Chromatographic data processor or chromatography workstation.

4.3.2.4 GC operating conditions Temperature ($^{\circ}$ 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. 0.6 μ L; Retention time. Isocarbophos about 6.1min, internal standard about 9.5min. GC operating conditions described above, the