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

GB 9556-2008

Replacing GB 9556-1999

phoxim technical

辛硫磷原药

(English Translation)

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Foreword

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

This standard replaces GB 9556-1999 "phoxim the original drug." the main differences between this standard and GB 9556-1999 are as follows.

--- Canceled and classification.

--- Deleted chloride control program.

From the date of implementation of this standard, replace and repeal GB 9556-1999.

The standard proposed by China Petroleum and Chemical Industry Association.

This standard by the National Standardization Technical Committee on Pesticides (CSBTS/T C133) centralized.

1 Scope

This standard specifies requirements for phoxim the original drug, test methods and marking, labeling, packaging, storage and transportation.

This standard applies to the impurities generated by the phoxim its production consisting of phoxim the 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

3.1 Appearance

Pale yellow to reddish brown oily liquid.

3.2 Technical Specifications

Phoxim the original drug should also meet the requirements of Table 1.

Table 1 phoxim the original drug control project indicators

Item Index

Phoxim mass fraction (in cis phoxim meter) /% ≥ 90.0 Moisture /% ≤ 0.5 Acidity (H₂SO₄ meter) /% ≤ 0.3 Test Method 4

4.1 Sampling

According to GB/T 1605-2001 "on the original drug sampling" approach. Determining sample package using a random number table method, the final sample size should not be Less than 100g.

4.2 Identification Test

HPLC --- The identification test can be carried out simultaneously with the determination of phoxim content. Under the same chromatographic operating conditions, the test A sample solution peak retention time compared with standard sample solution cis phoxim retention time, the relative difference should be less than 1.5%.

4.3.1 Method summary

Sample was dissolved in methanol, + water as the mobile phase, to use as filler Capcel-PakC18 stainless steel column and UV detector (254nm), the sample of phoxim reverse phase high performance liquid chromatography, external standard.

4.3.2 Reagents and solutions

Methanol. HPLC grade;

Water. The new secondary steam distilled water;

Phoxim standard. a known mass fraction $\geq 99.0\%$.

4.3.3 Instruments

High performance liquid chromatography. a variable wavelength UV detector;

Chromatographic data processor;

Column. 150mm x 4.6mm (i.d) stainless steel column, built Capcel-PakC18, 5 μ m filler (or with equivalent effect Column);

Filter. pore size of about 0.45 μ m;

Micro injector. 50 μ L;

Quantitative sample line. 5 μ L;

Ultrasonic cleaner.

4.3.4 HPLC operating conditions

Mobile phase. psi (methanol. water) = 75.25, by membrane filtration, and degassing;

Flow rate. 1.0mL/min;

Column temperature. room temperature (temperature change should not exceed 2 °C);

Detection wavelength. 254nm;

Injection volume. 5 μ L;

Retention time. 8.6min.

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 phoxim the original drug HPLC is shown in Figure 1.

1 --- cis phoxim.

Figure 1 phoxim the original drug by high performance liquid chromatogram

4.3.5.1 Preparation of standard solution

Weigh 0.1g phoxim standard (accurate to 0.0002g), a 50mL volumetric flask, dissolved in methanol and diluted to the mark, shake uniform. Using a pipette to take the above solution 5mL to 50mL volumetric flask, dilute with mobile phase to the mark.

4.3.5.2 Preparation of sample solution

Weigh phoxim containing 0.1g sample (accurate to 0.0002g), a 50mL volumetric flask, dissolved in methanol and diluted to the mark, Shake well. Using a pipette to take the above solution 5mL to 50mL volumetric flask, dilute with mobile phase to the mark.

4.3.5.3 Determination

Under these operating conditions, after the instrument is stable, continuous injection of several doses of the standard solution until the two adjacent needles phoxim peak area relative change After less than 1.5%, according to the solution, sample solution, sample solution, standard order of 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 phoxim peak areas are averaged. Sample phoxim the mass fraction of 1 (%), according to equation (1).

$$1 = A2$$

Where.

A1 --- the standard solution, the average phoxim peak area;

A2 --- sample solution, phoxim mean peak area;

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

4.3.7 Allowable difference

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

4.4 Determination of Moisture

According to GB/T 1600 the "Karl Fischer method".