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

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Methomyl technical

灭多威原药

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1 = A2

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Foreword

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

This standard uses redrafted law revision adopted FAO specification 264/T C (2002) "methomyl original drug" (in English).

This standard FAO Specification 264/T C (2002) The main technical differences are as follows.

Standard control --- This acetone-insoluble matter, water and pH range, without the FAO specifications of the three indicators.

The date of implementation of this standard, the former Chemical Industry Standard HG2611-1994 "Methomyl original drug" abolished.

The standard proposed by China Petroleum and Chemical Industry Association.

This standard by the National Pesticide Standardization Technical Committee (SAC/TC133).

This standard is drafted. Shenyang Chemical Research Institute.

Participated in the drafting of this standard. Haili Guixi Chemical Pesticide Co., Ltd.,

Shandong Pesticide Research Institute, Jiangsu dragon Chemical Co., Ltd.,
Yancheng Limin Chemical Co., Ltd.

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.

GB/T 1600 Pesticide Method Moisture

Determination 1601 pH value pesticides GB/T

GB/T 1604 pesticide regulations for acceptance

GB/T 1605-2001 sampling commercial pesticides

GB 3796 pesticide packaging General

GB/T 19138 Determination of acetone insoluble pesticide

3.1 Appearance

White to off-white crystalline powder.

3.2 Technical Specifications

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

Table 1 Methomyl original drug control project indicators

Item Index

Methomyl content /% ≥ 98.0

Acetone insolubles a /% ≤ 0.2

Moisture /% ≤ 0.3

The pH range of 4.0 to 8.0

When a normal production, the acetone insoluble matter was detected at least once every three months.

Test Method 4

4.1 Sampling

Carried out in GB/T 1605-2001 of "original drug product sampling" approach. Determined by sampling a random number table method packages, the amount of the final sample

Not less than 100g.

4.2 Identification Test

The present liquid chromatography --- identification test can be carried out simultaneously with the determination of the mass fraction of methomyl. Chromatography under the same operating conditions, the test

A solution sample peak retention time of the standard solution Methomyl retention time, the relative difference should be less than 1.5%.

Infrared spectroscopy --- Methomyl sample and standard IR in the range of 4000cm⁻¹ ~ 400cm⁻¹ absorption spectrum should be no significant difference

different. Methomyl standard infrared spectra of Figure 1.

Figure 1 methomyl standard infrared spectrum

4.3.1 Method summary

Sample was dissolved in methanol, methanol + water as the mobile phase, used as a filler SpherisorbC8 stainless steel column and UV detector

(235nm), the sample methomyl reverse phase high performance liquid chromatography and determination.

4.3.2 Reagents and solutions

Methanol. HPLC grade;

Water. The new secondary steam distilled water;

Methomyl Standard. Known methomyl content, $\geq 99.0\%$.

4.3.3 Instruments

HPLC. a variable wavelength UV detector;

Chromatographic data processor or chromatography workstation;

Column. 200mm x 4.6mm (. I.d) stainless steel column, SpherisorbC8, 10 μ m filler contents (or with equivalent effect

C8, C18 bonded phase);

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) = 40:60, 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. 235nm;

Injection Volume. 5 μ L;

Retention time. Methomyl about 4.1min.

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.

Typical methomyl original drug by HPLC is shown in Figure 2.

1 --- methomyl.

FIG 2 FIG HPLC original drug methomyl

4.3.5.1 Preparation of standard solution

Weigh 0.10 g methomyl standard (accurate to 0.0002g), a 50mL volumetric flask, diluted with methanol to the mark. accurate

The solution was pipetted 10mL, 50mL volumetric flask, diluted with methanol to the mark.

4.3.5.2 Preparation of sample solution

Methomyl containing weighed about 0.10g sample of the original drug (accurate to 0.0002g), a 50mL volumetric flask, dilute to the mark with methanol

Degree, shake. The solution was accurately pipetted 10mL, 50mL volumetric flask, diluted with methanol to the mark.