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**NATIONAL STANDARD OF THE
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Thiophanate-methyl technical

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(English Translation)

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Table of Contents

Foreword
2 Normative references
3.1 Appearance
3.2 Technical Specifications
4.1 Sampling
4.2 Identification Test
4.3 Determination of the mass fraction of thiophanate-methyl
4.6 Determination of loss on drying
4.7 Product testing and acceptance
5 Marking, labeling, packaging, storage and transportation

Foreword

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

This draft standard method using the reformed modified using FAO Specifications 262/T C/S/F (1993) "thiophanate-methyl original drug." the main difference between the present technical standard with FAO Specifications 262/T C/S/F (1993) as follows.

This standard specifies --- DAP (2,3- diamino phenazine) mass fraction of not more than 5.0mg/kg, FAO Specifications predetermined DAP (2,3- two Diaminophenazine) mass fraction of not more than 0.5mg/kg;

--- This standard specifies a pH range of indicators corresponding to a predetermined acidity index FAO specifications;

--- This standard specifies the drying loss indicators, FAO specifications not control the indicators.

The date of implementation of this standard, the former Chemical Industry Standard HG2462.1-1993 "thiophanate-methyl original drug" void.

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. Jiangsu abundance of blue and Chemical

Corporation, Haili Guixi Chemical Pesticide Co., Ltd., Jiangsu dragon of Studies Limited.

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.

Determination 1601 pH value pesticides GB/T GB/T 1604 pesticide regulations for acceptance of goods GB/T 1605-2001 sampling commercial pesticides GB 3796 pesticide packaging General

3.1 Appearance

White to gray powder.

3.2 Technical Specifications

Thiophanate-methyl original drugs should also meet the requirements of Table 1.

Table 1 thiophanate-methyl original drug control programs Index

Item Index

Thiophanate-methyl mass fraction /% ≥ 95.0 HAP (2- amino-3-hydroxy-phenazine) A mass fraction/(mg/kg) ≤ 0.5 DAP (2,3- diamino phenazine) A mass fraction/(mg/kg) ≤ 5.0 the pH range of 5.0 to 8.0 Loss on drying /% ≤ 0.5 when a normal production, HAP content, DAP mass fraction was measured 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

HPLC --- This identification test can be carried out simultaneous determination of the content of thiophanate methyl and spirit. In the same chromatographic operating conditions , The peak of a retention time in the sample solution and the standard solution thiophanate-methyl retention time, the relative difference of 1.5% should be or less.

Infrared spectroscopy --- sample and thiophanate-methyl standard IR in the range

4000cm⁻¹ ~ 400cm⁻¹ absorption spectrum should ignore significant differences.
Thiophanate-methyl standard infrared spectra of Figure 1.

FIG 1 thiophanate-methyl standard IR spectrum

4.3.1 Method summary

Sample was dissolved in methanol, + water as the mobile phase, used Hypersil-ODS filler stainless steel column and UV detector (269 nm), thiophanate-methyl of the sample were separated by HPLC, external standard.

4.3.2 Reagents and solutions

Methanol. HPLC grade;

Water. The new secondary steam distilled water;

Thiophanate-methyl Sample. thiophanate-methyl known mass fraction, ≥98.0%.

4.3.3 Instruments

HPLC. a variable wavelength UV detector;

Chromatographic data processor;

Column. 200mm x 4.6mm (i.d) stainless steel column, built-Hypersil-ODS, 5μm packing;

Filter. membrane pore size of about 0.45 μm;

Microsyringe. 50μL;

Quantitative sampling tube. 10μL;

Ultrasonic cleaning.

4.3.4 HPLC operating conditions

Mobile phase. [Psi] (methanol. water) = 60:40, by membrane filtration, and degassing;

Flow rate. 1.0mL/min;

Column temperature. room temperature;

Detection wavelength. 269nm;

Injection volume. 10μL;

Retention time. thiophanate-methyl about 4.8min.

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.

Typical thiophanate-methyl original drug by HPLC is shown in Figure 2.

1 --- thiophanate-methyl.

Figure 2 HPLC thiophanate-methyl original drug

4.3.5.1 Preparation of standard solution

Thiophanate-methyl Weigh 0.1 g of standard sample (accurate to 0.0002g), a 50mL volumetric flask, dissolve and dilute to volume with methanol, Shake well. 5mL pipetted with the above solution diluted further with methanol 50mL volumetric flask to the mark.

4.3.5.2 Preparation of sample solution

Thiophanate-methyl, said sample is taken containing 0.1g (accurate to 0.0002g), a 50mL volumetric flask, dissolved in methanol and diluted to Mark and shake. A pipetted sample solution was diluted to 5mL above another in methanol 50mL volumetric flask 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, the needle until two adjacent thiophanate methyl pk area opposite After the change is less than 1.2%, measured according to the standard solution, sample solution, the sample solution, the order of standard solution.

4.3.6 Computing

The measured sample solution and the preceding two needles two doses of the standard sample solution thiophanate-methyl pk area were averaged. A sample Mass fraction of 1-yl thiophanate (%), according to equation (1).

$$1 = A2$$

Where.

A1 --- the standard solution, the average area of thiophanate-methyl pk;

A2 --- sample solution, thiophanate-methyl pk average area;

--- Sample mass fraction thiophanate methyl, expressed in%.

4.3.7 Allows poor

Results difference between the two replicates thiophanate-methyl content should not exceed 1.2%, and the arithmetic mean value as a measurement result.

Determination of DAP and 4.4 HAP mass fraction