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PEOPLE'S REPUBLIC OF CHINA**

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Tetramethrin technical material

胺菊酯原药

(English Translation)

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Foreword

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

This standard was drafted in accordance with rules GB/T 1.1-2009 given.

Please note that some of the content of this document may involve patents, the issuing authority of this document does not assume responsibility for identifying these patents.

The standard proposed by China Petroleum and Chemical Industry Federation.

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

This standard is drafted. Yangzhou Agricultural Chemical Corporation.

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1 Scope

This standard specifies the requirements of the original drug tetramethrin, Test Method for marking, labeling, packaging, storage, and acceptance period.

This standard applies to tetramethrin impurities derived from the original drug tetramethrin and production thereof.

Note: Other names, structural formulas and physicochemical parameters of the basic tetramethrin see Appendix A.

2 Normative references

The following documents for the application of this document is essential. For dated references, only applies to the version dated paper Pieces. For undated references, the latest edition (including any amendments) applies to this document.

Preparation GB/T 601 standard titration solution Chemicals GB/T 1604 pesticide regulations for acceptance of goods GB/T 1605-2001 sampling commercial pesticides GB 3796 pesticide packaging General GB/T 6682-2008 analytical laboratory use specifications and test methods (ISO 3696. 1987, MOD) GB/T 8170-2008 rounding off means about rules and determining limiting values GB/T 19138 Determination of acetone insoluble pesticide

3.1 Composition and appearance

This product should tetramethrin production and related impurities, as a white to pale yellow powder-like or small.

3.2 Technical Specifications

Tetramethrin original drug shall comply with the requirements in Table 1.

Table 1 tetramethrin original drug control project indicators

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Tetramethrin mass fraction /% ≥ 92.0 Cis-isomer/trans-isomer $(20 \pm 5)/(80 \pm 5)$ Acidity (H₂SO₄ basis) /% ≤ 0.2 Drying loss mass fraction /% ≤ 0.5 Acetone insoluble mass fraction /% ≤ 0.2 Test Method 4

4.1 General provisions

This standard reagent in the absence of water and other requirements stated, refer to three predetermined analytical reagents and GB/T 6682-2008 of water. The test standard titration solution, in the absence of other requirements specified, and prepared according to GB predetermined calibration/T 601 a. test result 4.3.3 determination performed by the

rounding value in comparison 8170-2008 GB/T .

4.2 Sampling

According to GB/T 1605-2001 of "original drug product sampling" method, is determined by random sampling method of the package, a final sample volume should not be Less than 100g.

4.3 Identification Test

Optionally one of the following methods. When it is not determined by a process should then be determined using another method.

GC --- present identification test can be conducted simultaneously with the determination of the mass fraction of tetramethrin. Under the same chromatographic conditions, the sample solution Retention time and peak tetramethrin solution of a standard sample peak retention time of the solution, the relative difference should be less than 1.5%.

Infrared spectroscopy --- tetramethrin standard sample and the infrared in the range of 4000cm⁻¹ ~ 400cm⁻¹ absorption spectrum should be no significant difference. Tetramethrin standard infrared spectra of Figure 1.

1 tetramethrin standard infrared spectrum of FIG.

4.4.1 Method summary

Sample was dissolved in ethyl acetate, diisooctyl phthalate esters as internal standard, using the DB-210 capillary column and flame ionization detection Detector, tetramethrin of sample and measured by gas chromatography.

4.4.2 Reagents and solutions

Tetramethrin standard. tetramethrin known mass fraction, $w \geq 98.0\%$;

Diisooctyl phthalate;

Ethyl acetate;

Internal standard solution. Weigh 0.8g of diisooctyl phthalate, in 100mL volumetric flask, dissolved in ethyl acetate, constant volume, Shake well.

4.4.3 Instruments

Gas chromatograph. with a hydrogen flame ionization detector;

Chromatographic data processor or chromatography workstation;

Column. 30m x 0.25mm (id) DB-210 fused Shi Ying column, film thickness 0.25μm.

4.4.4 Chromatographic operating conditions

Temperature. column compartment 240 °C, the gasification chamber 250 °C, the detector chamber 250 °C;

Gas flow rate. carrier gas (N₂) 1.6mL/min, hydrogen 30mL/min, air 400mL/min;

Retention time. about internal standard 3.5min, cis tetramethrin about 4.7min, trans tetramethrin about 5.4min.

Gas chromatography operational conditions described above, a typical system operating parameters; instruments according to different characteristics, for a given operating parameter adjusted as appropriate, in order to for best results. Typical gas chromatograph tetramethrin parent drug shown in Figure 2.

1 --- internal standard;

2 --- cis-tetramethrin;

3 --- trans tetramethrin.

Fig 2 GC tetramethrin original drug and the internal standard

4.4.5.1 Preparation of standard solution

Sample Weigh 0.08 g of tetramethrin (accurate to 0.0002g), in 15mL clean, dry vial was stoppered, was added with a pipette accurately Internal standard solution 10mL, shake.

4.4.5.2 Preparation of sample solution

Weigh a sample containing 0.08g of tetramethrin (accurate to 0.0002g), in 15mL clean, dry vial stoppered with and 4.4.5.1 with a pipette in exact 10mL was added to the standard solution, shake.

4.4.5.3 Determination

Under the above operating conditions, after the instrument is stable, the number of consecutive doses of the standard solution is injected until the two needles tetramethrin adjacent peak area relative change After less than 1.2%, measured according to the standard solution, sample solution, the sample solution, the order of standard solution.

4.4.5.4 Computing

The two needle and the sample solution was measured before and after the two doses of the standard sample solution tetramethrin and internal standard peak areas were averaged. Sample the mass fraction of tetramethrin by formula (1).

$w_1 = r_2 \times m_1 \times w_{r1} \times m_2$ (1) where.