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

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2, 4-D butyl technical

24-滴丁酯原药

(English Translation)

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Foreword

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

The FAO standards and specifications 1.3/T C/S/F (1992) "2,4-Desterstechnical" of non-equivalent.

Appendix A of this standard is an informative annex.

Since the implementation of this standard, the original chemical industry standard HG2319-1992 "2,4- butylate original drug" void.

The standard proposed by China Petroleum and Chemical Industry Association.

This standard by the National Standardization Technical Committee on Pesticides (SAC/TC133) centralized.

1 Scope

This standard specifies the 2,4-D butyl ester original drug requirements, test methods and marking, labeling, packaging, storage and transportation.

This standard applies to the impurities from the production of 2,4-butyl and the composition of the produced 2,4-butyl ester 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 1604 Goods pesticide regulations for acceptance GB/T 1605-2001 Sampling Method commercial pesticides GB 3796 pesticide packaging General

3.1 Appearance

Colorless to brown oily liquid.

3.2 Technical Specifications

2,4-D butyl ester original drug should also meet the requirements of Table 1.

Table 1 2,4-D butyl ester original drug control project indicators

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Butyl 2,4 mass fraction /% ≥ 92.0 Free phenol content (2,4-DCP meter) /% ≤ 1.0 Acidity (2,4 meter) /% ≤ 1.5 Test Method 4

4.1 Sampling

According to GB/T 1605-2001 "on the original drug product sampling" approach. Determined by sampling a random number table method packages, the final sample size not less than 100g.

4.2 Identification Test

Gas Chromatography --- The identification test can be carried out simultaneously with the determination of the mass fraction of 2,4-D butyl ester. In the same chromatographic operating conditions , The retention time of the standard solution and the sample solution to a peak of 2,4-D butyl retention time, the relative difference should be 1.5% Fewer.

Infrared spectroscopy --- sample and 2,4-butyl standards in the infrared range $4000\text{cm}^{-1} \sim 400\text{cm}^{-1}$ absorption spectrum should Ignorance Significant differences. 2,4-D butyl standard infrared spectrum is shown in Figure 1.

1 of butyl 2,4-standard infrared spectrum

4.3.1 Method summary

Sample was dissolved in chloroform, di-isobutyl phthalate as internal standard, the use of polyethylene terephthalate and tetrachlorophthalic Apiezon L to fill a stainless steel column and flame ionization detector, the sample of 2,4-butyl ester by gas chromatography separation and determination. also may Determined using capillary gas chromatography, chromatographic operating conditions, see Appendix A.

4.3.2 Reagents and solutions

Chloroform;

2,4-D butyl standard. a known mass fraction $\geq 98.0\%$;

Internal standard. di-iso-butyl phthalate, should not interfere with the analysis of impurities;

Internal standard solution. Weigh isobutyl phthalate 3.5g, set 250mL flask, add appropriate amount of chloroform was dissolved and diluted to Mark and shake.

4.3.3 Instruments

Gas chromatograph. with a hydrogen flame ionization detector;

Chromatography or chromatography workstation processor;

Column. 2m x 3mm (i.d.) Stainless steel (glass) columns, built-in 1.7% tetrachlorophthalic polyethylene terephthalate and 0.3% Apiezon L/ChromosorbW-AWDMCS180 ~ 250 μ m filler;

Micro injector. 10 μ L.

4.3.4 GC operating conditions

Temperature ($^{\circ}$ C). 170 column temperature, the gasification chamber 260, the detector chamber 260;

Gas flow rate (mL/min). carrier gas (N₂) 20, hydrogen gas 30, air 300;

Injection volume. 0.6 μ L;

Retention time (min). 2,4- 11.0 butylate, internal standard 6.7.

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 2,4-D butyl ester original drug and the internal standard gas chromatography is shown in Figure 2.

1 --- internal standard;

2 --- 2,4-D ester.

Figure 2 GC-butyl 2,4-drug and internal standard

4.3.5.1 Preparation of standard solution

Weigh 2,4-butyl standard 0.1g (accurate to 0.0002g), placed in a glass bottle stuffed with a pipette 5mL internal standard solution Liquid, shake.

4.3.5.2 Preparation of sample solution

Weigh containing 2,4-D butyl ester 0.1g (accurate to 0.0002g) sample, placed in a glass vial stopper, and 4.3.5.1 with the same Pipette internal standard solution 5mL, shake.

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 needles 2,4-butyl and the internal standard peak adjacent After the area is less than 1.2% relative change, according to the solution, the sample solution, the sample solution and standard sequence of the standard solution was measured.

4.3.5.4 Calculation

The measured before and after the two-needle sample solution and two doses of the standard sample solution were 2,4-D butyl ester ratio and the internal standard peak area average. Sample mass fraction of 2,4-D butyl ester 1 (%), according to equation (1).

Where.

--- Standard samples of 2,4-butyl ester content, expressed as a percentage.

4.3.6 Allowable difference

Difference butyl 2,4 mass fraction of two parallel determination results shall not be greater than 1.2%, the arithmetic mean value as a measurement result.

4.4.1 Method summary

The sample was dissolved in ethanol, ammonia, 4-amino-antipyrine and potassium ferricyanide solution color, absorbance was measured at 520nm place.

Isolated from the calibration curve of standard sample absorbance at the same volume, calculated free phenol content.