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

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**Import and export textiles. Determination of the residues of
endosulfan and profenofos. GC-MS method**

进出口纺织品硫丹、丙溴磷残留量的测定 气相色谱-串联质谱法

(English Translation)

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Foreword

Appendix A and Appendix B of this standard are informative annexes.

This standard is proposed and managed by the National Certification and Accreditation Administration.

This standard was drafted. Tianjin Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China.

Drafters of this standard. Guo Wei, Yu Tao, Yan Wei.

This standard is the first industry standard for entry-exit inspection and quarantine.

Determination of endosulfan and profenofos residues in import and export textiles Gas chromatography-tandem mass spectrometry

1 Scope

This standard specifies the method for the determination of alpha-endosulfan, beta-endosulfan and profenofos residues in textiles by gas chromatography.

This standard applies to textile materials and their products.

2 Principle

The sample was extracted with petroleum ether, concentrated, dissolved in n-hexane and made up to volume, using a gas chromatograph (GC/ECD) equipped with an electron capture detector.

The measurement was performed by external standard method and confirmed by ion

chromatography with gas chromatography-mass spectrometry (GC/MSD).

3 Reagents and materials

The reagents used were of analytical grade unless otherwise stated.

3.1

Acetone. heavy distillation.

3.2

Petroleum ether. boiling range 60 ° C ~ 90 ° C, heavy distillation.

3.3

n-hexane. heavy distillation.

3.4

Endosulfan, profenofos standard. purity $\geq 99\%$.

3.5

Standard working solution. accurately weigh the appropriate amount of alpha-endosulfan, beta-endosulfan and profenofos standard, dissolved in n-hexane and prepared 100 $\mu\text{g/mL}$ of standard stock solution. Dilute into a series of standard working solutions of different concentrations with n-hexane as needed. Standard reserve both the solution and the standard working solution were stored at 4 ° C. The storage period of the standard stock solution was half a year, and the standard working solution was stored for 1 W.

4 Instruments and equipment

4.1

Gas Chromatograph. equipped with an electron capture detector (ECD).

4.2

Gas Chromatography-Mass Spectrometer. equipped with a mass spectrometer (MSD).

4.3

Soxhlet extractor. 250mL.

4.4

Rotate the evaporator.

4.5

Constant temperature water bath.

4.6

Microinjector. 10 μ L.

5.1 Extraction

Take 50g representative sample, cut into 5mm x 5mm or less, mix, weigh 5.0g (accurate to 0.01g) sample from the mixed sample (for parallel test) into the filter paper tube, then placed in the Soxhlet extractor, add 100mL petroleum ether in the extraction bottle, at 80 ° C ~ 90 ° C the mixture was refluxed for 6 h in a water bath (reflow rate 8 times/h to 10 times/h). After cooling, the extract was rotary-concentrated to near dryness in a 40 ° C $\pm$ 2 ° C water bath.

Dissolve in n-hexane and dilute to 5.00 mL, and after appropriate filtration, confirm by gas chromatography or gas chromatography-mass spectrometry.

5.2.1 Gas Chromatography (GC-ECD) conditions

Since the test results are related to the instrument and conditions used, it is not possible to give a general parameter for chromatographic analysis. Adopt the following parameters it has been proven to be suitable for testing.