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FZ

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

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**Chemical assay of insect resist (IR) agent content in woolltextile
(English Translation)**

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Table of Contents

Foreword...	3
1 Scope...	4
2 Normative References...	4
3 Principle...	4
4 Instruments and Reagents...	4
5 Specimen...	5
6 Operating Procedures...	5
7 Calculation and Results Explanation...	7
8 Test Report...	8
Appendix A (Normative) Table of Fastness Coefficients.....	10
Appendix B (Normative) Table of Insect Repellent Specifications.....	11

1 Scope

This Standard specifies a method for testing the permethrin-based insect repellent content in wool textiles by liquid chromatography.

This Standard applies to the testing of permethrin-based insect repellent content in textiles containing different proportions of animal fibers and their blends.

2 Normative References

The provisions in following documents become the provisions of this Standard through reference in this Standard. For dated references, the subsequent amendments (excluding corrigendum) or revisions do not apply to this Standard, however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 6682 Water for analytical laboratory use - Specification and test methods

TWC TM27 Chemical Analysis Methods for Insect Repellent Content in Textiles

TWC TM28 Insect Repellent (IR) Adhesion Test

3 Principle

Permethrin was extracted from the insect-repellent specimen using a chemical method to prepare a solution. After dilution, the solution was analyzed by liquid chromatography under specific conditions. The test results were then compared with the test results of a set of standard strength insect-repellent solutions (which were obtained using the same chromatograph under the same conditions).

4 Instruments and Reagents

The analysis was performed according to the high-performance liquid chromatography (HPLC) method in TWC TM27. Unless otherwise specified, all reagents used in the analysis were of analytically pure and Grade III water as specified in GB/T 6682.

4.1

High-performance liquid chromatograph (HPLC), ultraviolet spectrophotometer.

4.2

Constant temperature water bath.

4.3

Ultrasonic oscillator.

4.4 15 mL stoppered graduated test tubes.

4.5

Thermoplastic paper.

4.6

Microsample centrifuge filter tubes, 0.5 μm polytetrafluoroethylene filter membrane.

4.7

Potassium hydroxide and glacial acetic acid.

4.8

Deionized water.

4.9

Methanol.

4.10

Permethrin insect repellent with known content (% , mass concentration).

5 Specimen

5.1

Specimens shall be taken from various parts of the sample.

Carpets. Plush bundles made of different yarns shall be selected.

Yarn. Shorter lengths of yarn shall be taken from different parts of the skein or bobbin.

Blankets. If non-wool yarns are present, they shall be removed.

Wool tops, loose fibers, worsted fabrics, woolen fabrics, sweaters. It shall be taken dispersedly from a large batch of specimens.

5.2

If the specimen contains multiple colors, each color shall be sampled separately to the required weight.

5.3

Before analysis, the specimen shall be placed under standard atmospheric conditions at $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and $65\% \pm 2\%$ relative humidity for 24 h before weighing.

6.1 Preparation of standard solution for trans-/cis-permethrin insect repellent

Place 10 mL of the insect repellent in a volumetric flask. Dissolve it in 1000 mL of methanol to prepare a 10 mL/L stock solution. Dilute the stock solution with methanol to prepare a set of standard solutions. The concentration of the insect repellent in the standard solutions shall be between 0.04% and 0.5% (calculated based on fiber weight).

6.2 Extraction method for trans-/cis-permethrin insect repellent

6.2.1

Accurately weigh 0.5000 g of the insect repellent specimen fiber (accurate to 0.1 mg).

6.2.2

Place the specimen of 6.2.1 into a 15 mL stoppered graduated tube; add 10 mL of methanol;

seal with thermoplastic paper, and incubate in an 80°C constant temperature water bath for 15 min.

6.2.3

Remove it and place in an ultrasonic oscillator for continuous oscillation for 90 min.

6.2.4

After removing it and allowing to cool off to room temperature; transfer the specimen solution into a centrifuge filter tube and filter through a 0.5 μm PTFE membrane (2500 $\mu\text{g/g}$ x 15 min).

6.2.5

Reserve the supernatant after filtration for later use.

6.3.1 HPLC analysis conditions

Chromatograph. ZorbaxC8 (5 μm) 25 cm x 4.6 mm (inner diameter) stainless steel column;

Eluent (mobile phase). Distilled water/methanol = 20/80; add 1 mL of 5% (mass concentration) potassium hydroxide solution and 1 mL of 20% (volume fraction) glacial acetic acid per liter of solution; filter and degas;

Flow rate. 1.3 mL/min;

Column temperature. 40°C;

Injection volume. 20 μL ;

UV detection wavelength. 230 μm ;

Sensitivity. 0.02 AnFs;

Recording paper speed. 3 mm/min.

6.3.2 Chromatographic analysis of standard solutions and extracts; calculate the amount of

insect repellent in the specimen according to the method described in 7.1.

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