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

GB/T 18414-2025

Replacing GB/T 18414.2-2006

**Textiles - Determination of chlorinated phenols and
ortho-phenylphenol**

纺织品 含氯苯酚邻苯基苯酚的测定

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Table of Contents

1 Scope
2 Normative References
3 Terms and Definitions
4 Principle
5 Reagents and Materials
6 Instruments and Equipment
6.2 Analytical balance. respectively with an accuracy of
7 Specimen Preparation
8 Test Steps
8.4 Gas Chromatography-Mass Spectrometry
9 Quantitation Limits, Recovery Rates and Precision...
Appendix B

1 Scope

GB/T 18414-2025 is the Chinese national standard on textiles - determination of chlorinated phenols and ortho-phenylphenol, in the field of textile and leather technology. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 28 February 2025 by the State Administration for Market Regulation; Standardization Administration of China, and takes effect on 1 March 2027. Classification: ICS 59.080.01, CCS W04. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This document describes the determination method for 19 chlorinated phenols and ortho-phenylphenol in textiles by gas chromatograph-mass spectrometer (GC-MS). This document applies to all kinds of textile products.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references

without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

3 Terms and Definitions

This document does not have terms or definitions that need to be defined.

4 Principle

After the specimen is ultrasonically extracted by potassium hydroxide / methanol solution, it is derivatized by acetic anhydride and extracted by n-hexane, and then, determined by gas chromatography-mass spectrometry using the internal standard method.

5 Reagents and Materials

Unless otherwise specified, all reagents used are of analytical grade.

5.1 n-Hexane. CAS No.. 110-54-3, chromatographically pure.

5.2 Water. Grade-3 water specified in GB/T 6682.

5.3 Acetic anhydride. CAS No.. 108-24-7.

5.4 Anhydrous sodium sulfate. CAS No.. 7757-82-6.

5.5 Chlorinated phenols and ortho-phenylphenol standard substances / standard samples. purity 97% (mass fraction), in accordance with the provisions of Appendix A. 5.6

2-Chlorophenol-d4 (internal standard 1). CAS No.. 93951-73-6, purity 97% (mass fraction).

5.7 2,4-Dichlorophenol-d3 (internal standard 2). CAS No.. 93951-74-7, purity 99% (mass fraction). 5.8 2,3,4,6-Tetrachlorophenol-13C6 (internal standard 3). CAS No.. 1246820-81-4,

purity 98% (mass fraction).

5.9 Pentachlorophenol-13C6 (internal standard 4). CAS No.. 85380-74-1, purity 98% (mass fraction).

5.10 Potassium hydroxide solution. weigh-take an appropriate amount of potassium hydroxide and use water to prepare it into 2 mol/L potassium hydroxide solution.

5.11 Standard stock solution. respectively weigh-take appropriate amounts of chlorinated phenols and ortho-phenylphenol standard substances / standard samples (5.5) (accurate to 0.1 mg) and use acetonitrile (chromatographically pure) to prepare them into 1,000 mg/L stock solution. NOTE. the standard stock solution is stored in an environment of 0 C ~ 4 C and has a validity period of 6 months.

5.12 Mixed standard working solution. pipette an appropriate amount of the standard stock

solution (5.11) of chlorinated phenols and ortho-phenylphenol and use potassium hydroxide solution (5.10) to prepare it into 10 mg/L mixed working solution. NOTE. the mixed standard working solution is stored in an environment of 0 C ~ 4 C and has a validity period of 1 month.

5.13 Internal standard stock solution. weigh-take an appropriate amount of the internal standard (5.6 ~ 5.9) (accurate to

0.1 mg) and use potassium hydroxide solution (5.10) to prepare it into 100 mg/L internal standard stock solution. NOTE. the internal standard stock solution is stored in an environment of 0 C ~ 4 C and has a validity period of 6 months.

5.14 Extraction solution. respectively pipette an appropriate amount of the internal standard stock solution (5.13), add an appropriate amount of methanol (chromatographically pure), and use potassium hydroxide solution (5.10) to prepare it into potassium hydroxide / methanol (volume ratio 1.1) extraction solution containing 10 g/L internal standard.

6 Instruments and Equipment

6.1 Gas chromatograph-mass spectrometer (GC-MS). equipped with electron impact ionization source (EI).

6.2 Analytical balance. respectively with an accuracy of

0.1 mg and

0.01 g.

6.3 Ultrasonic water bath generator. controllable temperature (70 5) C, operating frequency (40 5) kHz.

6.4 Vortex mixer.

6.5 Mechanical oscillator. the oscillation frequency is about 200 times/min (one reciprocating motion is 1 time), and the horizontal amplitude is 2 cm ~ 5 cm.

6.6 Pipette. 1 mL ~ 10 mL, 10 L ~ 100 L.

6.7 Extraction bottle. about 50 mL, made of hard glass, with a stopper.

7 Specimen Preparation

Select a sufficient amount of representative sample, cut it into pieces with a maximum size not exceeding 5 mm, evenly mix them, and use an analytical balance (6.2) to accurately weigh-take

1.0 g (accurate to

0.01

g) from them as the specimen for later use.

8 Test Steps

8.1 Extraction Place the specimen prepared in 7 in an extraction bottle (6.7), add 20 mL of extraction solution (5.14), then, seal the stopper, and in an ultrasonic generator (6.3) in a (70 ± 5) °C water bath, extract it for 2 h 5 min.

8.2 Acetylation After the extraction is completed, take out the above-mentioned extraction bottle, cool it to room temperature, and add 2 mL of acetic anhydride (5.3). Use a vortex mixer (6.4) to vortex and oscillate it for 1 min, then, add

2.0 mL of n-hexane (5.1), use a mechanical oscillator (6.5) to mechanically oscillate it for 30 min at room temperature, and let it stand for stratification. Add an appropriate amount of anhydrous sodium sulfate (5.4), take part of the supernatant liquid and filter it through an organic phase needle filter (5.15) into a sampling bottle for GC- MS determination (8.4).

8.3 Acetylation of Standard Working Solution Use a pipette (6.6) to pipette a certain volume of the mixed standard working solution (5.12) into an extraction bottle (6.7), add 20 mL of extraction solution (5.14), and in accordance with the steps of 8.2, handle it. Prepare at least 5 concentrations of acetylated standard working solutions (such as.

0.02 mg/L,

0.05 mg/L,

0.2 mg/L,

0.5 mg/L and

8.4 Gas Chromatography-Mass Spectrometry

8.4.1 Analysis conditions Appendix B gives an example of gas chromatography-mass spectrometry (GC-MS) analysis test parameters.

8.4.2 Qualitative analysis Under the analysis conditions of 8.4.1, by comparing the retention time of the specimen solution with that of the standard working solution (the retention time deviation tolerance is

0.1 min), in addition, in the mass spectrum of the sample after background subtraction, the reference qualitative ions shall all appear, and the abundance ratio of the qualitative ions shall be consistent with the relative abundance ratio of the standard working solution with an equivalent concentration (see Appendix B), then, it can be determined that the corresponding analyte is present in the sample. The total ion chromatogram of GC-MS analysis of acetylated chlorinated phenols and ortho- phenylphenol standard substances /