ICS 61.060
CCS Y 78

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 46974-2025

Footwear - Chemical tests - Determination of short-chain chlorinated paraffins

鞋类 化学试验方法 短链氯化石蜡的测定

Issued on: December 31, 2025 **Implemented on: July 1, 2026**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

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Foreword

This document was drafted in accordance with the rules provided in GB/T 1.1-2020 Directives for Standardization - Part

1.Rules for the Structure and Drafting of Standardizing Documents. Please be noted that certain content of this document may involve patents. The institution issuing this document does not undertake the responsibility of identifying these patents. This document was proposed by China National Light Industry Council. This document shall be under the jurisdiction of National Technical Committee 305 on Footwear of Standardization Administration of China (SAC/TC 305). The drafting organizations of this document. SINOLIGHT Inspection & Certification Co., Ltd.; SGS-CSTC Standards Technical Services (Shanghai) Co., Ltd.; Zhejiang Red Dragonfly Shoes Co., Ltd.; Chongqing Academy of Metrology and Quality Inspection; China Light Industry Inspection and Certification (Wenling) Co., Ltd.; China Leather & Footwear Research Institute. The main drafters of this document. Chang Wenkai, Su Jian, Zhang Li, Wu Wenshun, Zhang Wei, Wu Xinxin, Wang Dingqiao, Wang Hao, Zhu Na. Footwear - Chemical Tests - Determination of Short-chain Chlorinated Paraffins WARNING

---personnel using this document shall have practical experience working in a formal laboratory. This document does not address all potential safety issues; users are responsible for taking appropriate safety and health precautions and ensuring compliance with relevant national regulations.

1 Scope

GB/T 46974-2025 is the Chinese national standard covering SCCPs in footwear - plasticisers and flame retardants listed under the Stockholm Convention, which turn up in PVC soles and coated uppers and are restricted in every market a Chinese shoemaker exports to. First edition, and it belongs with GB/T 47912-2026 on the medium-chain paraffins in textile auxiliaries. It was issued on 31 December 2025 and has been in force since 1 July 2026, as a first edition. The document is under the responsibility of the China National Light Industry Council. This page is published from the official record of the 2025 edition; the clause text of a standard this recent is not yet in circulation, and the figures, limits and tables it contains are those of the document itself, delivered in full with the English translation.

This document describes a qualitative and quantitative method for determining the content of short-chain chlorinated paraffins (C10~13, SCCPs) in footwear and footwear components using gas chromatography-negative ion chemical ionization mass spectrometry. This document is applicable to the determination of SCCPs content in various types of footwear materials.

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 45063 Footwear - Chemical Tests - General Principles on the Preparation of Samples

3 Terms and Definitions

The following terms and definitions are applicable to this document.

3.1 short-chain chlorinated paraffins; SCCPs A mixture of chlorinated straight-chain alkanes with 10 ~ 13 carbon atoms, obtained by chlorinating different n-alkanes.

4 Principle

Under specified conditions, the sample is ultrasonically extracted with toluene or n-hexane.

The extraction solution is dried under nitrogen, re-dissolved in n-hexane, purified with concentrated sulfuric acid, and qualitatively and quantitatively analyzed using gas chromatography-negative ion chemical ionization mass spectrometry (GC-NCI-MS).

5 Reagents and Materials

5.1 Toluene CAS No.. 108-88-3, analytically pure.

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

5.3 Concentrated Sulfuric Acid 98% (mass fraction), rho =

1.84 g/mL (20 C), CAS No.. 7664-93-9, analytically pure.

5.4 Internal Standard -1,2,3,4,5,6-hexachlorocyclohexane (CAS No.. 6108-10-7) or -1,2,3,4,5,6- hexachlorocyclohexane (Lindane, CAS No.. 58-89-9), chromatographically pure.

5.5 Standard Solutions

5.5.1 SCCPs, chlorine content 51.5% (51.5% Cl), CAS No.. 85535-84-8, industrial grade, rho = 100 g/mL in n-hexane.

5.5.2 SCCPs, chlorine content 55.5% (55.5% Cl), CAS No.. 85535-84-8, industrial grade, rho = 100 g/mL in n-hexane.

5.5.3 SCCPs, chlorine content 63.0% (63.0% Cl), CAS No.. 85535-84-8, industrial grade, rho = 100 g/mL in n-hexane.

5.5.4 Internal standard solution, n-hexane solution of internal standard (5.4), rho = 100 g/mL.

5.6 Polytetrafluoroethylene Filter Membrane 0.45 m.

6.1 Analytical balance, with a division value of

0.1 mg.

6.2 Volumetric flasks.

6.3 Extractor, with polytetrafluoroethylene stopper, 20 mL, 50 mL, etc.

6.4 Ultrasonic extractor, temperature adjustable, temperature controllable to (60 5) C.

6.5 Nitrogen blowing instrument, gas flow controllable.

6.6 Rotary evaporator, temperature controllable to (55 5) C.

6.7 Vortex mixer.

6.8 Horizontal oscillator, which can ensure efficient and thorough mixing of phases. NOTE. oscillation conditions with a minimum frequency of 5 s^-1 and an amplitude of 2 cm to 5 cm are suitable.

6.10 Sample vial, 2 mL.

6.11 Gas chromatograph-mass spectrometer (GC-NCI-MS) equipped with a negative ion chemical ionization source.

7 Sampling

In accordance with GB/T 45063, prepare the sample to be determined. The test sample shall consist of a single material removed from the shoe, such as leather, textiles, polymers, coating materials or other materials, and be prepared into fragments with any side length not exceeding 5 mm.

8 Test Procedures

8.1 Extraction Accurately weigh-take (1.0 0.1) g of representative sample fragments (Chapter 7) into the extractor (6.3), accurate to

0.1 mg. For non-leather samples, add

10.0 mL of toluene (5.1), and for leather samples, add

10.0 mL of n-hexane (5.2), wet the entire sample, and use a PTFE stopper to seal the extractor (6.3). If the test sample is not completely immersed in the extraction solution, then, continue adding toluene (5.1) or n-hexane (5.2), until the sample is completely wetted. In an ultrasonic extractor (6.4), ensure that the extractor (6.3) is sealed, extract at (60 5) C for (60 5) min, then, cool to room temperature.

8.2.1 For the toluene extraction solution, transfer-take

5.0 mL, and use a nitrogen blowing instrument (6.5) or a rotary evaporator (6.6) to evaporate the extraction solution to dryness (free of toluene) at (55 5) °C. Use a vortex mixer (6.7) to dissolve the residue after nitrogen blowing in

1.96 mL of n-hexane (5.2), and add 40 L of internal standard solution (5.5.4).

8.2.2 For the n-hexane extraction solution, transfer-take

1.96 mL and add 40 L of internal standard solution (5.5.4).

8.2.3 Slowly add

2.0 mL of concentrated sulfuric acid (5.3) to the extraction solution (