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

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**Leather and fur - Determination of chlorinated hydrocarbons -
Part 1: Short-chain chlorinated paraffins (SCCPs)
(English Translation)**

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1 Scope

This document describes a test method for determining the content of short-chain chlorinated paraffins (SCCPs) (C10~C13) in leather and furs.

This document is applicable to the determination of SCCPs content in various types of leather, furs, and their products.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through 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 39364 Leather - Chemical, physical, mechanical and fastness tests - Sampling location (GB/T 39364-2020, ISO 2418.2017, MOD)

QB/T 1267 Fur - Chemical, physical, mechanical and fastness tests - Sampling location

QB/T 1272 Fur - Preparation of chemical test samples

QB/T 2716 Leather - Preparation of chemical test samples

3 Terms and Definitions

This document contains no terms or definitions that require clarification.

4 Principle

Under specified conditions, the test sample is subjected to ultrasonic extraction using n-hexane.

After purification of the extract with sulfuric acid, it is analyzed and determined using gas chromatography-electron capture negative ionization mass spectrometry (GC-ECNI-MS), with quantification performed using the internal standard method.

If the laboratory demonstrates that the accuracy of liquid chromatography-mass spectrometry (LC-MS) is equivalent to that of GC-ECNI-MS, it may also be used; refer to Appendix C.

NOTE: In certain instances, when determining SCCPs using GC-ECNI-MS, the presence of sulfonated chlorinated paraffins and chlorinated olefins of equivalent chain length may interfere with the test results. The LC-MS analytical method presented in Appendix C is designed to provide an improved solution for eliminating potential false positives associated with GC-ECNI-MS.

5 Reagents and Materials

5.1

Unless otherwise specified, all reagents used shall be of analytical grade.

5.2

n-Hexane, chromatographic grade.

5.3

Internal standard solution. Lindane, CAS No.. 58-89-9, 1 000 µg/mL; solvent. n-hexane.

5.4

SCCPs (C10~C13, CAS No.. 85535-84-8) standard solutions with varying chlorine contents,

100 µg/mL, specifically as follows.

--- SCCPs (C10~C13) with 55.5% chlorine content;

--- SCCPs (C10~C13) with 63.0% chlorine content.

5.5

SCCPs standard solution (50 µg/mL, 59% chlorine content). Transfer 533 µL of the SCCPs (C10~C13) standard solution with 55.5% chlorine content (5.4) and 467 µL of the SCCPs

(C10~C13) standard solution with 63% chlorine content (5.4) into a 2 mL volumetric flask (6.5);

add 20 μ L of the internal standard solution (5.3), and dilute to volume using n-hexane (5.2).

Standard solutions of other suitable concentrations may also be used, provided that the concentration of the internal standard is 10 μ g/mL.

5.6

Concentrated sulfuric acid, $\rho=1.84$ g/mL (20°C).

6 Instruments and Equipment

6.1

Analytical balance, with a readability of 0.1 mg.

6.2

Stoppered, sealed container, 20 mL, for use in the extraction with n-hexane.

6.3

Ultrasonic generator, with temperature control capability up to (60 $\pm$ 5)°C.

NOTE: An ultrasonic generator with an operating frequency of 40 kHz is suitable.

6.4

Pipettes or pipettors, of various capacities.

6.5

Volumetric flasks, 2 mL.

6.6 Gas chromatograph equipped with a mass spectrometric detector with an electron capture

negative ionization source (GC-ECNI-MS).

6.7 Oscillator or stirrer, or other suitable mixing equipment, for effective mixing of the phases.

7.1 Sampling

Sampling of leather shall be conducted in accordance with the provisions of GB/T 39364.

Sampling of fur shall be conducted in accordance with the provisions of QB/T 1267; during the sampling process, care shall be taken to avoid loss of the fur pile and to maintain its integrity.

If sampling cannot be performed from the standard designated area (e.g., when sampling

directly from footwear or garments), samples shall be taken from any available area within the usable material; the test samples shall be representative, and this deviation shall be noted in the test report.

Any residual adhesive remaining on the sample shall be removed using physical methods.

7.2 Preparation of Test Samples

Preparation of leather test samples shall be conducted in accordance with the provisions of QB/T 2716.

Preparation of fur test samples shall be conducted in accordance with the provisions of QB/T

1272; the test samples shall be tested together with the fur pile attached.

8.1 Extraction

Weigh (0.5 ± 0.001) g of the test sample using an analytical balance (6.1) into a stoppered, sealed container (6.2). Add 9.9 mL of n-hexane (5.2) and 100 μ L of the internal standard solution (5.3), then seal the container. Place the test sample in an ultrasonic generator (6.3) and extract it at $(60 \pm 5)^{\circ}\text{C}$ for (60 ± 2) min. Allow the test sample to cool to room temperature (below 27°C).

During extraction, ensure that the test sample is completely submerged; if complete submersion cannot be achieved, the volumes of n-hexane and internal standard solution may be increased proportionally.

8.2 Purification

Transfer 5.0 mL of the n-hexane extract into a stoppered, sealed container (6.2). Add 1.0 mL of concentrated sulfuric acid (5.6) and shake the mixture on an oscillator (6.7) for 10 min. Once the two phases have separated (which may be facilitated by centrifugation), transfer the upper n-hexane phase to a separate container. Add 1.0 mL of concentrated sulfuric acid (5.6) to perform a further purification step. Repeat this procedure until the lower acid layer becomes clear or turns white; this process must not be repeated more than five times. Take 1 mL of the upper n-hexane phase for GC-ECNI-MS (6.6) analysis.

8.3 Chromatographic Analysis

Since the test results depend on the instrumentation used, universal parameters for chromatographic analysis cannot be specified. Appendix D provides examples of chromatographic conditions that have been proven effective.