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GB/T 38403.3-2025

Leather and fur - Determination of the preservative content - Part 3: Gas chromatography-mass spectrometry method (methanol extraction)

皮革和毛皮 防霉剂的测定 第3部分：气相色谱-质谱法（甲醇萃取）

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Foreword

This document is in accordance with the provisions of GB/T 1.1-2020 "Guidelines for standardization work Part 1: Structure and drafting rules for standardization documents" Drafting.

This document is Part 3 of GB/T 38403 "Determination of antifungal agents for leather and fur". GB/T 38403 has been published as follows part.

- Chemical tests for leather and fur - Determination of mildew inhibitors (TCMTB, PCMC, OPP, OIT). Liquid chromatography method (GB/T 38403-2019);

- Part 2: Liquid chromatography (artificial sweat extraction);

- Part 3: Gas chromatography-mass spectrometry (methanol extraction).

Please note that some of the contents of this document may involve patents. The issuing organization of this document does not assume the responsibility for identifying patents.

This document was proposed by China Light Industry Federation.

This document is under the jurisdiction of the National Technical Committee for Standardization of Leather Industry (SAC/TC252).

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Introduction

Leather and fur are prone to mold due to the presence of collagen, protein, oil and other ingredients. Adding mold inhibitors is to prevent leather and fur products from mold.

The most effective means to prevent leather from getting moldy, so antifungal agents are common additives in leather and fur.

It will bring different degrees of negative impact on humans and the environment, especially when the content of mildew inhibitors in leather and fur is high, it is very easy to cause human body The detection of the content of mildew inhibitors in leather has become a major concern for leather (fur) products (such as clothing, shoes, bags, sofas, etc.) companies.

Therefore, the determination of the content of mildew inhibitors in leather and fur has attracted more and more attention from the society.

In order to provide a basis for the determination of the mildew inhibitor content in leather and fur, it is planned to consist of three parts.

- Part 1: Liquid chromatography (acetonitrile extraction). The purpose is to establish a liquid chromatography method for the determination of leather and wool after acetonitrile extraction.

Test method for antifungal agent content in leather.

- Part 2: Liquid chromatography (artificial sweat extraction). The purpose is to establish the determination of Test method for mildew inhibitor content in leather and fur.

- Part 3: Gas chromatography-mass spectrometry (methanol extraction). The purpose is to establish the gas chromatography-mass spectrometry method after methanol extraction.

Test method for determining the content of mildew inhibitors in leather and fur (not applicable to the determination of TCMTB).

Among the mildew inhibitors used in leather and fur processing, TCMTB, 4-chloro-3-methylphenol (PCMC), o-phenyl Products with phenol (OPP) and N-octylisothiazolinone (OIT) as active ingredients are the most widely used. Some foreign leather buyers have Requirements are put forward for the content of four substances in leather. Regarding the determination of the content of four antifungal agents, ISO 13365-1 2020 and ISO 13365-2 2020 specifies the test methods for the determination of mildew inhibitor content in leather by acetonitrile extraction and artificial sweat extraction.

Liquid chromatography was used for determination. China has completed the conversion of these two international standards.

Mass spectrometry has higher detection sensitivity, and the detection limit can meet the testing requirements of various products. It is also simple to operate, with high reliability and practicality.

Based on this, China independently developed the third part of the standard, which stipulates that the skin This method is a test method for the content of mildew inhibitors in leather and fur. However, since TCMTB may decompose under high temperature conditions, this method is not suitable for leather.

The determination of TCMTB content in fur is not applicable.

This document is Part 3 of GB/T 38403; GB/T 38403-2019 "Chemical Tests for Antifungal Agents for Leather and Fur Determination of (TCMTB, PCMC, OPP, OIT). Liquid chromatography method" is proposed to be the first part of GB/T 38403 when it is revised, and the standard The standard name is "Determination of antifungal agents on leather and fur - Part 1: Liquid chromatography (acetonitrile extraction)".

Determination of antifungal agents for leather and fur Part 3: Gas chromatography-mass spectrometry (methanol extraction)

1 Scope

This document describes a test method for the determination of mildew inhibitors in leather and fur by gas chromatography-mass spectrometry (GC-MS).

This document applies to 4-chloro-3-methylphenol (PCMC), o-phenylphenol (OPP), N-octylphenol (CPC), and The method is suitable for the determination of the content of three kinds of mildew inhibitors, such as isothiazolinone (OIT), and is also applicable to semi-finished leathers such as wet blue leather and wet white leather.

2 Normative references

GB/T 6682

GB/T 39364

3 Terms and definitions

There are no terms or definitions that require definition in this document.

4 Principle

The sample was extracted with ultrasonic-assisted methanol, and the extract was concentrated and fixed to volume, and then analyzed by gas chromatograph equipped with a mass selective detector.

The samples were determined by GC-MSD and confirmed by selected ion monitoring and quantified by external standard method.

5 Reagents and Materials

5.1 Unless otherwise specified, all reagents used are of analytical grade and the test water should at least meet the requirements of Grade III water in GB/T 6682.

5.2 Methanol, chromatographic grade.

5.3 Acetone, chromatographic grade.

5.4 Anhydrous sodium sulfate (Na2SO4). calcine at 650°C for 4h, cool and store in a desiccator for later use.

5.5 PCMC, OPP, OIT standard products, see Appendix A for related information.

5.6 Standard stock solution. Weigh appropriate amounts of the three standards (5.5) and prepare a standard stock solution with a mass concentration of 100 mg/L using acetone (5.3).