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Leather — Determination of chlorinated hydrocarbons in leather —

Part 2: Chromatographic method for middle-chain chlorinated paraffins (MCCPs)

Cuir — Dosage des hydrocarbures chlorés dans le cuir —

Partie 2: Méthode chromatographique pour les paraffines chlorées à chaîne moyenne (PCCM)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by the Chemical Test Commission of the International Union of Leather Technologists and Chemists Societies (IUC Commission, IULTCS) in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 289, *Leather*, the secretariat of which is held by UNI, in accordance with the agreement on technical cooperation between ISO and CEN (Vienna Agreement).

IULTCS, originally formed in 1897, is a world-wide organization of professional leather societies to further the advancement of leather science and technology. IULTCS has three Commissions, which are responsible for establishing international methods for the sampling and testing of leather. ISO recognizes IULTCS as an international standardizing body for the preparation of test methods for leather.

A list of all parts in the ISO 18219 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

Middle-chain chlorinated paraffins (MCCPs) are a mixture of chlorinated hydrocarbons with a carbon chain length of 14 to 17, and a chlorine content range of 40 % to 70 %. MCCPs are expected to be persistent and bioaccumulative in the environment, based on their similar chemical and physical properties to short-chain chlorinated paraffins (SCCPs).

The analysis of chlorinated paraffins is a challenge. The technical compounds are mixtures of up to 200 congeners with different chain lengths and degrees of chlorination. GC chromatograms of these complex mixtures typically show a lot of overlapping peaks that can be difficult to separate. In particular, the responses to the various chlorination degrees can vary over a large range.

In addition, the presence of sulfochlorinated paraffins and equivalent chain-length chloroalkenes in such technical compounds can cause interference.

This document describes a procedure to compare the chromatogram results for MCCPs compounds from a test sample with the chromatogram results of a defined calibration standard of the most typically used mixture (55 % chlorination for MCCPs). With this GC-ECNI-MS (gas chromatography negative ion chemical ionization mass spectrometry) procedure it uses four ion traces for identifying the MCCPs.

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Leather — Determination of chlorinated hydrocarbons in leather —

Part 2:
Chromatographic method for middle-chain chlorinated paraffins (MCCPs)

1 Scope

This document specifies a chromatographic method to determine the amount of middle-chain chlorinated paraffins (MCCPs) C_{14} to C_{17} in processed and unprocessed leathers.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2418, *Leather — Chemical, physical and mechanical and fastness tests — Sampling location*

ISO 4044, *Leather — Chemical tests — Preparation of chemical test samples*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

The test sample is extracted using n-hexane at 60 °C in an ultrasonic bath for 60 min. After a sulfuric acid clean-up, an aliquot is then analysed using a gas chromatograph coupled to a mass selective detector with chemical ionization (GC-ECNI-MS).

A liquid chromatography system single quad (LC-MS) or with triple quad mass spectrometry (LC-MS/MS), as described in Annex B, can also be used if the user has demonstrated that the accuracy of measurement is equivalent to that of the GC-ECNI-MS method.

In some cases when determining MCCPs using the GC-ECNI-MS method, the presence of sulfochlorinated paraffins and equivalent chain-length chloroalkenes can cause interference. Annex B proposes a LC-MS/MS application method that aims to give a better resolution and eliminates possible false positives determined with the GC-ECNI-MS method.

5 Apparatus and materials

Use normal laboratory apparatus and, in particular, the following.

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