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IULTCS IUC 30-1

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Leather — Determination of chlorinated hydrocarbons in leather —

Part 1: Chromatographic method for short-chain chlorinated paraffins (SCCPs)

Cuir — Dosage des hydrocarbures chlorés dans le cuir —

Partie 1: Méthode chromatographique pour les paraffines chlorées à chaîne courte (PCCC)

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 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 co-operation 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.

This first edition of ISO 18219-1 cancels and replaces ISO 18219:2015, which has been technically revised.

The main changes to ISO 18219:2015 are as follows:

— a modification of the ISO number and the title;

— Introduction revised and updated;

— a new Clause 3;

— technical changes to Clause 4;

— in 6.2, a new internal standard;

— in 8.3, the SPE clean-up changed to a procedure using sulfuric acid;

— new 9.2, 9.3, 9.4 and 9.5 to improve the method;

— a new Annex B with a LC-MS/MS procedure;

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— a new Annex C explaining how to interpret the chromatograms with peak shape evaluation (PSE).

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

Short-chain chlorinated paraffins (SCCPs) are a mixture of chlorinated hydrocarbons with a chain length of 10 to 13 carbon atoms and a chlorine content of 40 % to 70 %. SCCPs are classified as dangerous to the environment, since they are very toxic to aquatic organisms and can cause long-term adverse effects in the aquatic environment.

In 2002, the European Directive 2002/45/EC restricted the sale and use of SCCPs (C_{10} to C_{13}) in product preparations for the fatliquoring of leather. Preparations containing concentrations equal to or higher than 1 % of SCCPs were forbidden. This directive is included as part of the EU Regulation 1907/2006 (REACH). Within this EU Regulation, in October 2008, the SCCPs were added to the candidate list of substances of very high concern (SVHC)[2].

The EU Commission Regulation 2015/2030[3] in November 2015 prohibited alkanes C_{10} to C_{13}, chloro (SCCPs) (CAS No 85535-84-8), as constituents of articles. Articles containing SCCPs in concentrations lower than 0,15 % by mass are allowed.

In 2017, the SCCPs were listed in Annex A of the Stockholm Convention on Persistent Organic Pollutants (POP)[4].

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 SCCPs compounds from a test sample with the chromatogram results of a defined calibration standard of the most typically used mixture (59 % chlorination for SCCPs). With this gas chromatography negative ion chemical ionization mass spectrometry (GC-ECNI-MS) procedure it uses four ion traces for identifying the SCCPs.

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Leather — Determination of chlorinated hydrocarbons in leather —

Part 1:
Chromatographic method for short-chain chlorinated paraffins (SCCPs)

1 Scope

This document specifies a chromatographic method to determine the amount of short-chain chlorinated paraffins (SCCPs) C_{10} to C_{13} 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 sulfuric acid clean-up, an aliquot is then analysed using a gas chromatograph fitted to a mass selective detector with chemical ionization (GC-ECNI-MS).

A liquid chromatography system with single quad (LC-MS) or 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 SCCPs using the GC-ECNI-MS method, the presence of sulfochlorinated paraffins and equivalent chain-length chloroalkenes causes interference. The Annex B procedure with a LC-MS/MS method aims to give a better resolution and eliminate 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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