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IULTCS IUC 28-1

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Leather — Determination of ethoxylated alkylphenols (APEO) —

Part 1: Direct method

Cuir — Détermination des alkylphénols éthoxylés (APEO) —

Partie 1: Méthode directe

Reference numbers
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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.

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 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*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 18218-1:2015), which has been technically revised.

The main changes are as follows:

— a new Clause 3 has been added and subsequent clauses renumbered;

— Clauses 6 and 8 have been technically revised;

— the NOTE in 7.1 of the previous edition has been deleted;

— a new calculation procedure in 8.4 has been added;

— Annex A has been technically revised;

— a new Annex B has been added.

A list of all parts in the ISO 18218 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

Nonylphenol ethoxylate belongs to the non-ionic surfactants. The biodegradation of nonylphenol ethoxylate releases the persistent pollutant, the branched nonylphenol. Nonylphenol is a hormonal acting substance that is toxic for waterborne organisms and many other organisms. For this reason, the release of nonylphenol ethoxylate into the environment should be avoided.

In 2003 the European Directive 2003/53/EC[4] restricted the sale and use of nonylphenol and nonylphenol ethoxylate in product preparations for industries with discharges to wastewater. Preparations containing concentrations equal to or higher than 0,1 % of nonylphenol ethoxylate or nonylphenol were forbidden. This Directive is included as part of the EU Regulation 1907/2006 (REACH).[3]

No detailed composition of the chemical substance nonylphenol ethoxylate can be given; it is assigned the general structural formula:

(C_9 alkyl chain, branched or linear)–Ph–$[OCH_2CH_2]_n$–OH

where Ph = phenyl, $n \geq 1$.

To cover the group of ethoxylates of 4-nonylphenol, branched and linear, the European Chemical Agency (ECHA) has assigned the substance the following definition:

"4-nonylphenol, branched and linear, ethoxylated [substances with a linear and/or branched alkyl chain with a carbon number of 9 covalently bound in position 4 to phenol, ethoxylated covering UVCB and well-defined substances, polymers and homologues, which include any of the individual isomers and/or combinations thereof]."[5]

In the leather industry, nonylphenol ethoxylate and octylphenol ethoxylate surfactants have been used. However, the water-insoluble substances nonylphenol and octylphenol have not been used. For this reason, two different analytical procedures have been prepared for analysing leather samples.

This document is a method that directly determines the ethoxylated alkylphenol. It is an efficient procedure for the analysis of a larger number of leather samples. This procedure requires liquid chromatography (LC) with triple quadrupole mass spectrometer (MS/MS) to identify the nonylphenol ethoxylate and octylphenol ethoxylate.

ISO 18218-2 specifies a procedure for analysing the alkylphenol component. The ethoxylated alkylphenol is cleaved to form the alkylphenol, which is identified using LC or gas chromatography-mass spectrometry (GC-MS) equipment. This method can also be used to indirectly determine the alkylphenol ethoxylate content in leather.

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Leather — Determination of ethoxylated alkylphenols (APEO) —

Part 1: Direct method

1 Scope

This document is a method for determining ethoxylated alkylphenols (APEO) [nonyphenol ethoxylate ($NPEO_n$, where $2 \leq n \leq 16$) and octylphenol ethoxylate ($OPEO_n$, where $2 \leq n \leq 16$)] in leather. This direct method is especially suitable when a larger number of leather samples are to be checked for the presence of ethoxylated alkylphenols.

This method requires the use of liquid chromatography (LC) with a triple quadrupole mass spectrometer (MS/MS) to identify and quantify the ethoxylated alkylphenols.

NOTE 1 In the leather industry, the most commonly used ethoxylated alkylphenol is the NPEO, with an average of 9 EO. It has an optimum cloud point in water for the typical leather processing temperatures of 40 °C to 55 °C.

NOTE 2 This document and ISO 18218-2 use different solvents for the extraction of the ethoxylated alkylphenols from leather. Consequently, the two analytical methods are expected to give similar trends but not necessarily the same absolute result for the ethoxylated alkylphenol content in leather.

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, mechanical and fastness tests — Position and preparation of specimens for testing*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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 terminology databases for use in standardization at the following addresses:

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

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

4 Principle

The leather sample is extracted with methanol using an ultrasonic bath. Subsequently, an aliquot of the solution can, after filtering, be directly analysed without further cleaning of the sample using LC with a MS/MS detector.

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