SLOVENSKI STANDARD
oSIST prEN ISO 18218-1:2021

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Usnje - Določevanje etoksilatnih alkilfenolov - 1. del: Neposredna metoda (ISO/DIS 18218-1:2021)

Leather - Determination of ethoxylated alkylphenols - Part 1: Direct method (ISO/DIS 18218-1:2021)

Leder - Bestimmung von ethoxylierten Alkylphenolen - Teil 1: Direktes Verfahren (ISO/DIS 18218 1:2021)

Cuir - Détermination des alkylphénols éthoxylés - Partie 1: Méthode directe (ISO/DIS 18218-1:2021)

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Leather — Determination of ethoxylated alkylphenols —

Part 1:
Direct method

Cuir — Détermination des alkylphénols éthoxylés —

Partie 1: Méthode directe

ICS: 59.140.30

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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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

ISO 18218-1 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.

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

— a new clause 3 has been added, the following clauses have been re-numbered;

— the Note in clause 8.1 has been deleted.

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.

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 restricted the sale and use of nonylphenol and nonylphenol ethoxylate in product preparations for industries with discharges to waste water. Preparations containing concentrations equal or higher than than 0,1 % of nonylphenol ethoxylate or nonylphenol were forbidden. This Directive is included as part of the EU Regulation 1907/2006 (REACH).

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 (with Ph = phenyl, n ≥ 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].*

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 part of ISO 18218 is a method that directly determines the ethoxylated alkylphenol. It is an efficient procedure for the analysing of a larger number of leather samples. This procedure requires HPLC with triple quadrupole mass spectrometer (MSMS) to identify the nonylphenol ethoxylate and octylphenol ethoxylate.

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

Leather — Determination of ethoxylated alkylphenols —

Part 1:
Direct method

1 Scope

This document is a method for determining ethoxylated alkylphenols (nonyphenol ethoxylate [$NPEO_n$ with $1 \leq n \leq 16$] and octylphenol ethoxylate [$OPEO_n$ with $1 \leq n \leq 16$]) in leather. This direct method is especially suitable where a larger number of leather samples are to be checked for the presence of ethoxylated alkylphenols.

This method requires the use of high-performance liquid chromatography (HPLC) with triple quadrupole mass spectrometer (MSMS) to identify and quantify the ethoxylated alkylphenols.

NOTE 1 In the leather industry, the most commonly used commercial ethoxylated alkylphenol has been 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 ISO 18218-1 and ISO 18218-2[1] 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, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 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 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 https://www.electropedia.org/

4 Principle

The leather sample is extracted in methanol using an ultrasonic bath. Subsequently, an aliquot of the solution can, after filtering, be directly analysed without further cleaning of the sample using high-performance liquid chromatography (HPLC) with a triple quadrupole mass spectrometer (MSMS).

5 Apparatus and materials

Normal laboratory apparatus and, in particular, the following:

5.1 Ultrasonic bath, with controllable heating capable of maintaining a temperature of (70 ± 5) °C.

5.2 Glass container with a screw cap (22 ml has been found suitable).

5.3 Polypropylene or **polyethylene syringe**, 2 ml.

5.4 Syringe membrane filters, pore size 0,2 µm, for use with syringe (5.3).

5.5 Volumetric flasks, 10 ml and 100 ml.

5.6 Analytical balance, weighing to 1 mg.

5.7 Pipettes, various sizes, 1 ml to 5 ml.

5.8 Instrumental equipment, high-performance liquid chromatograph with gradient elution and triple quadrupole mass spectrometer (HPLC-MSMS).

6 Chemicals

If not otherwise defined, analytical reagent grade chemicals shall be used.

6.1 Methanol.

6.2 Nonylphenol ethoxylate, $NPEO_n$ with n = 9 – 10, CAS No. 68412-54-4, Sigma-Aldrich® Product No. T9284 (IGEPAL® CO-630)[1)], technical grade.

NOTE The brand name is given to improve the comparability of test results amongst laboratories. The commercial nonylphenol ethoxylate contains groups of ethoxylates of nonylphenol with linear and branched structures, so use of another reference can lead to different results. Only technical grade references are currently available from laboratory chemical suppliers.

6.3 Octylphenol ethoxylate, $OPEO_n$ with n = 9 – 10, CAS No. 9002-93-1, Sigma-Aldrich® Product No. 542334 (Triton™ X-100)[1)], technical grade.

NOTE The brand name is given to improve the comparability of test results amongst laboratories. The commercial octylphenol ethoxylate contains groups of ethoxylates of octylphenol with linear and branched structures, so use of another reference can lead to different results. Only technical grade references are currently available from laboratory chemical suppliers.

6.4 Stock solution of nonylphenol ethoxylate and octylphenol ethoxylate, ρ = 250 µg/ml.

25 mg of the respective alkylphenol ethoxylate (6.2 and 6.3) are dissolved in different 100 ml volumetric flasks (5.5) with methanol (6.1) and filled up to the mark.

6.5 Calibration solutions of nonylphenol ethoxylate and octylphenol ethoxylate

Four calibration solutions of ρ = 2,5 µg/ml, ρ = 5 µg/ml and ρ = 10 µg/ml and ρ = 50 µg/ml for each alkylphenol ethoxylate are prepared using the respective stock solution (6.4).

1) Sigma-Aldrich® Product No. T9284 (IGEPAL® CO-630) and Sigma-Aldrich® Product No. 542334 (Triton™ X-100) are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO or IDF of these products.