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Leather - Determination of ethoxylated alkylphenols -  
Part 2: Indirect method (ISO 18218-2:2015)

## EESTI STANDARDI EESSÕNA

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English Version

## Leather - Determination of ethoxylated alkylphenols - Part 2: Indirect method (ISO 18218-2:2015)

Cuir - Détermination chimique des alkylphénols éthoxylés -  
Partie 2: Méthode indirecte (ISO 18218-2:2015)

Leder - Bestimmung von ethoxylierten Alkylphenolen - Teil  
2: Indirektes Verfahren (ISO 18218-2:2015)

This European Standard was approved by CEN on 28 February 2015.

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

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## European foreword

This document (EN ISO 18218-2:2015) has been prepared by Technical Committee CEN/TC 289 “Leather”, the secretariat of which is held by UNI, in collaboration with Technical Committee IULTCS “International Union of Leather Technologists and Chemists Societies”.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2015, and conflicting national standards shall be withdrawn at the latest by December 2015.

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### Endorsement notice

The text of ISO 18218-2:2015 has been approved by CEN as EN ISO 18218-2:2015 without any modification.

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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](http://www.iso.org/directives)).

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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 18218-2 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 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.

ISO 18218 consists of the following parts, under the general title *Leather — Determination of ethoxylated alkylphenols*:

- *Part 1: Direct method*
- *Part 2: Indirect method*

## Introduction

Nonylphenol ethoxylate belongs to the non-ionic surfactants. The biodegradation of nonylphenol ethoxylate releases the persistent pollutant 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 has to 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 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:



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.

ISO 18218-1 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.

This part of ISO 18218 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 and process auxiliaries.

# Leather — Determination of ethoxylated alkylphenols —

## Part 2: Indirect method

### 1 Scope

This part of ISO 18218 is a method for determining alkylphenols (nonylphenol and octylphenol) and alkylphenol ethoxylates (nonylphenol ethoxylate and octylphenol ethoxylate) in leather and process auxiliaries. The analysis is based on high-performance liquid chromatography (HPLC) or gas chromatography-mass spectrometry (GC-MS).

The analysis of the alkylphenol ethoxylate is made by cleaving the alkylphenol ethoxylate and measuring the released alkylphenol.

NOTE ISO 18218-1 and this part of ISO 18218 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 3696:1987, *Water for analytical laboratory use — Specification and test methods*

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

### 3 Principle

Leather samples are extracted with acetonitrile using an ultrasonic bath and the nonylphenol (NP) and/or octylphenol (OP) in the extract is quantitatively determined by HPLC or GC-MS.

The leather process auxiliaries are dissolved in acetonitrile and the NP and/or OP in the solution is quantitatively determined by HPLC or GC-MS.

The nonylphenol ethoxylate (NPEO) and octylphenol ethoxylate (OPEO) in the extract or solution are first converted into NP and OP, using aluminium triiodide as cleavage agent, and the NP and OP are determined by HPLC or GC-MS. The contents of NPEO and OPEO are then calculated by normalizing to NPEO<sub>9</sub> and OPEO<sub>10</sub> respectively. Examples of the four analytes used for the determination are shown in [Table 1](#).

**Table 1 — Analytes determinable by this method**

Analyte	Empirical formula	Abbreviation	CAS <sup>a</sup> no.
4-nonylphenol (mixture of isomers)	C <sub>9</sub> H <sub>19</sub> -C <sub>6</sub> H <sub>4</sub> -OH	NP	84852-15-3
4-tert-octylphenol	C <sub>8</sub> H <sub>17</sub> -C <sub>6</sub> H <sub>4</sub> -OH	OP	140-66-9
Nonylphenol ethoxylate	C <sub>9</sub> H <sub>19</sub> -C <sub>6</sub> H <sub>4</sub> -(OC <sub>2</sub> H <sub>4</sub> ) <sub>n</sub> OH (n≈9)	NPEO <sub>9</sub>	9016-45-9

<sup>a</sup> CAS = Chemical Abstract Service.