Leather — Determination of ethoxylated alkylphenols —
Part 2: Indirect method

Cuir — Détermination chimique des alkylphénols éthoxylés —
Partie 2: Méthode indirecte

ICS 59.140.30

ISO/CEN PARALLEL PROCESSING

This draft has been developed within the European Committee for Standardization (CEN), and processed under the CEN-lead mode of collaboration as defined in the Vienna Agreement. This draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel five-month enquiry. Should this draft be accepted, a final draft, established on the basis of comments received, will be submitted to a parallel two-month approval vote in ISO and formal vote in CEN.

This draft International Standard is submitted to all ISO member bodies for voting, as a standard prepared by an international standardizing body in accordance with Council Resolution 42/1999. The proposer, International Union of Leather Technologists and Chemists (Commission IUC, IULTCS), has been recognized by the ISO Council as an international standardizing body for the purpose of Council Resolution 42/1999.

To expedite distribution, this document is circulated as received from the committee secretariat. ISO Central Secretariat work of editing and text composition will be undertaken at publication stage.

Pour accélérer la distribution, le présent document est distribué tel qu'il est parvenu du secrétariat du comité. Le travail de rédaction et de composition de texte sera effectué au Secrétariat central de l'ISO au stade de publication.
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 18218-2/IUC 28-2 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 Standardisation (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 organisation of professional leather societies to further the advancement of leather science and technology. IULTCS has 3 Commissions, which are responsible for establishing international methods for sampling and the testing of leather. ISO recognises IULTCS as an International Standardising 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 difficult to biodegrade 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).

In the leather industry the nonylphenol ethoxylate and octylphenol ethoxylate surfactants have been used. However, the water insoluble substances, nonylphenol and octylphenol, were not used. For this reason there have been 2 different analytical procedures prepared for analysing leather samples.

Part 1 is a fast 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.

Part 2 is a procedure for analysing the alkylphenol. The ethoxylated alkylphenol is cleaved to form the alkylphenol, which is identified using HPLC or 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 Standard 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.

Annexes A, B and C of this standard are for information only.

2 Normative references

The following referenced documents are indispensable for the application 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 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 aluminum 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$_9$ and OPEO$_{10}$ respectively. Details of the 4 analytes that can be determined are shown in Table 1.
Table 1 — Analytes determinable by this method

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Empirical formula</th>
<th>Abbreviation</th>
<th>CAS (^a) No.</th>
</tr>
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<tbody>
<tr>
<td>4-nonylphenol (mixture of isomers)</td>
<td>(C_9H_{19}-C_6H_4-OH)</td>
<td>NP</td>
<td>84852-15-3</td>
</tr>
<tr>
<td>4-tert-octylphenol</td>
<td>(C_8H_{17}-C_6H_4-OH)</td>
<td>OP</td>
<td>140-66-9</td>
</tr>
<tr>
<td>Nonylphenol ethoxylate (n (\approx 9))</td>
<td>(C_9H_{19}C_6H_4(OC_2H_4)_nOH)</td>
<td>NPEO(_9)</td>
<td>9016-45-9</td>
</tr>
<tr>
<td>Octylphenol ethoxylate (n (\approx 10))</td>
<td>(C_8H_{17}C_6H_4(OC_2H_4)_nOH)</td>
<td>OPEO(_{10})</td>
<td>9002-93-1</td>
</tr>
</tbody>
</table>

\(^a\) CAS = Chemical Abstract Service

4 Apparatus and materials

Normal laboratory apparatus and, in particular, the following:

4.1 Analytical balance, weighing to an accuracy of 0.1 mg
4.2 Ultrasonic bath, 40 kHz, with thermostat
4.3 Separating funnels, 150 ml
4.4 Rotary evaporator, with thermostat and vacuum system
4.5 Membrane filter, polyamide, 0.45 \(\mu\)m
4.6 HPLC, equipped with diode array detector (DAD) or fluorescence detector (FLD)
4.7 GC, equipped with mass selective detector (MS)

5 Chemicals

Unless otherwise stated analytical grade chemicals shall be used.

5.1 Acetonitrile for HPLC
5.2 n-Hexane
5.3 Aluminium triiodide, commercially available, or prepared according to Annex A
5.4 Sulfuric acid solution, 0.5 mol/L
5.5 Sodium thiosulfate solution, saturated
5.6 Anhydrous magnesium sulphate (\(\text{MgSO}_4\)) for analysis
5.7 Anhydrous sodium sulfate (\(\text{Na}_2\text{SO}_4\)) for analysis, treated at 800 °C for 4 hours, store dry
5.8 Sodium chloride solution, saturated
5.9 NP (in Table 1) solution for calibration, 1000 mg/L in hexane
5.10 OP (in Table 1) solution for calibration, 1000 mg/L in hexane
5.11 OPEO (in Table 1) solution for calibration, 2000 mg/L in acetonitrile
Dilute this solution with acetonitrile (5.1) if a calibration is applied.

5.12 NPEO (in Table 1) solution for calibration, 4000 mg/L in acetonitrile

Dilute this solution with acetonitrile (5.1) if a calibration is applied.

5.13 4n-nonylphenol (4n-NP, CAS No. 104-40-5) solution, 200 mg/L in hexane

The 4n-NP can be used as an internal standard for GC-MS analysis. Dilute this solution with hexane (5.2) if the internal calibration curve is applied.

6 Sampling and sample preparation

6.1 Preparation of leather samples

6.1.1 Sampling and preparation of samples

If possible, sample according to ISO 2418. Grind the leather in accordance with ISO 4044. If sampling in accordance with ISO 2418 is not possible (e.g. leathers from finished products like shoes, garments), details about the sampling shall be given in the test report.

6.1.2 Sample extraction

Accurately weigh approximately 2.5 g of the ground leather sample into an Erlenmeyer flask, mix with approximately 3 g of Na₂SO₄. Then add 50.0 ml ± 1 ml aliquot of acetonitrile into the flask which is then closed with a stopper.

Put the flask into an ultrasonic water bath and extract at 50 °C ± 5 °C for at least 60 min. Then cool the flask to room temperature.

6.1.3 Filter the extracts through a quantitative filter paper to remove leather and salt particles. Collect at least 30 ml of the filtrate for the analysis as in 6.3.

6.2 Preparation of leather process auxiliary samples

Accurately weigh approximately 0.5 g of sample into a flask, carefully mix with approximately 2 g of MgSO₄ (5.6). Then use acetonitrile, 3 × 7 ml (approximately), to dissolve the sample by stirring with a glass rod. Filter the extracts through a quantitative filter paper. Collect the extracts in a 50 ml volumetric flask and fill to 50.0 ml with acetonitrile.

6.3 Determination of OP and NP

For HPLC analysis, use the sample extracts, either (6.1.2) or (6.2) directly after filtering through a polyamide membrane (4.5).

For GC-MS analysis, add 10.0 ml of the sample extract, either (6.1.2) or (6.2), to a separating funnel (4.3). Subsequently, add approximately 20 ml of water, and 1 ml of sulfuric acid solution (5.4). Extract the mixture two times with approximately 20 ml of n-hexane (5.2), separate and collect the organic phase. After that, wash the hexane extracts with approximately 30 ml of water and dehydrate them with approximately 5 g of Na₂SO₄ (5.7). Remove the organic solvent by rotary evaporator (4.4) at approximately 50 °C. Re-dissolve the residuals in 10.0 ml ± 0.1 ml of hexane (5.2) and the solution is ready for GC-MS analysis after filtering through a polyamide membrane (4.5).

NOTE If the organic phase cannot separate freely in the funnel after treating with hexane, add approximately 30 ml of saturated sodium chloride to the funnel, then shake the mixture and stand for separation.
The signal response for the sample extracts should be within the concentration ranges of the calibration curves. If not then the extract solutions are to be diluted accordingly with acetonitrile for HPLC analysis or n-hexane for GC-MS analysis.

### 6.4 Determination of OPEO and NPEO

Prepare aluminium triiodide (5.3) in acetonitrile for the cleavage of NPEO and OPEO according to Annex A.

**NOTE 1** Aluminium triiodide is extremely air and water sensitive. If the commercial aluminium triiodide is used, it can be dissolved in carbon disulfide at a concentration of approximately 0,1 g/ml. Pipette 10 ml of the solution into a flask and remove the solvent by heating before adding the sample extracts.

Add a 10.0 ml ± 0,1 ml aliquot of the sample extracts (deriving from 6.1.2 or 6.2) into the flask containing approximately 1 g of aluminum triiodide, and continue refluxing at 90 °C ± 2 °C for 30 min ± 5 min.

Take out the flask and slowly add water dropwiser until no boiling occurs. Dilute the contents with approximately 20 ml of water and cool to room temperature.

Add the mixture to a separating funnel, rinse the flask with approximately 20 ml of hexane (5.2) and transfer the organic solution to the funnel. Then add approximately 1 ml of sulfuric acid solution (5.4) and shake. Collect the organic phase, and extract the aqueous phase with another 20 ml of hexane. Combine all the organic phase. Subsequently, add approximately 2 ml of sodium thiosulfate solution (5.5) and shake until the pink colour disappears. Wash the organic phase with approximately 30 ml of water and dehydrate them with approximately 4 g of Na$_2$SO$_4$ (5.7). Remove the organic solvent by rotary evaporator at approximately 50 °C.

**NOTE 2** If the organic phase cannot separate freely in the funnel after treating with hexane, add approximately 30 ml saturated sodium chloride (5.8) to the funnel, then shake the mixture and stand for separation.

For HPLC analysis, re-dissolve the residual in 10.0 ml ± 0,1 ml of acetonitrile (5.1) and filter through a polyamide membrane (4.5).

For GC-MS analysis, re-dissolve the residuals in 10.0 ml ± 0,1 ml of hexane (5.2) and filter through a polyamide membrane (4.5).

The signal response for the sample extracts should be within the concentration ranges of the calibration curves. If not then the extract solutions are to be diluted accordingly with acetonitrile for HPLC analysis or n-hexane for GC-MS analysis.

### 6.5 Chromatographic analysis

Detection of NP and OP can be performed using the chromatographic techniques (4.6 or 4.7). Other validated methods may be used. The quantification is performed by means of HPLC or GC-MS. Where gas chromatography is used, appropriate internal standards (5.13) shall be employed.

Operating parameters and examples of the chromatographic analysis for NP and OP are listed in Annex B and Annex C.

### 6.6 Blank determination

Treat the blank in exactly the same way as the sample, but replace the sample by the appropriate amount of acetonitrile.

### 6.7 Evaluation

For NP and OP, the amounts are usually calculated by means of a software program based on their peak areas and calibration curves. For NPEO and OPEO, the amounts are calculated based on the peak areas of the yielded NP and OP, as well as their calibration curves.
7 Calibration without internal standard

7.1 Calibration for OP and NP

The calibration curves for NP and OP are prepared by directly measuring five levels of increasing concentrations of NP and OP standards in the range 1 mg/l to 20 mg/l.

7.2 Calibration for OPEO and NPEO

For the calibration curves of NPEO and OPEO, 10.0 ml of acetonitrile spiked with NPEO<sub>9</sub> and OPEO<sub>10</sub> (listed in Table 1) standards are prepared in the range 2 mg/l to 50 mg/l. The solutions are treated as specified in Clause 6.2. Calibration curves are made by plotting five pairs of the given amounts of NPEO<sub>9</sub> and OPEO<sub>10</sub> and the signal response of the yielded NP and OP.

8 Calculation

8.1 Calculation of OP and NP

Calculate the concentration of OP and NP in the sample in accordance with Equation (1).

\[ w_{AP} = \rho_{AP} \times \frac{A_i \times V}{A_c \times m_E} \]  

where
\[ w_{AP} \] mass portion of NP or OP in the specimen, expressed in mg/kg
\[ \rho_{AP} \] concentration of NP or OP in the calibration solution in mg/l
\[ A_i \] area response of NP or OP in the specimen solution in area units
\[ A_c \] area response of NP or OP in the calibration solution in area units
\[ V \] volume the specimen is made up, in ml
\[ m_E \] weight of the leather specimen or leather process auxiliary, in g

8.2 Calculation of OPEO and NPEO

Calculate the concentration of NPEO and OPEO in the sample in accordance with Equation (2).

\[ w_{APEO} = \rho_{APEO} \times \frac{(A_2 - A_1) \times V'}{A'_c \times m_E} \]  

where
\[ w_{APEO} \] mass portion of NPEO or OPEO in the specimen, expressed in mg/kg
\[ \rho_{APEO} \] concentration of NPEO<sub>9</sub> or OPEO<sub>10</sub> in the calibration solution in mg/L
\[ A_2 \] area response of the yielded NP or OP in the specimen solution in area units
\[ A_1 \] see Equation (1)
NOTE It is noted that the NP and OP is stable during the cleavage process. Thus, the area response \( A_1 \) of NP and OP in the sample extracts contributed to the area response \( A_2 \) of the isolated total NP + OP from the cleavage reaction, because the sample extracts are directly submitted for cleavage without removing the free NP and OP. Accordingly, \( (A_2 - A_1) \) is used when calculating the contents of NP and OP yielded from the NPEO and OPEO.

### Test report

The test report shall include at least the following information:

a) reference to this document (i.e. ISO 18218-2),

b) either the type, origin and designation of the analyzed leather sample and the sampling method used, or the name and origin of the process auxiliary,

c) the analytical procedure and instrument used,

d) the analytical results for the OP, NP, OPEO and NPEO contents, as well as the sum of the four results,

e) any deviations from the analytical procedure, particularly any additional steps performed,

f) the date of the test.
Annex A
(informative)

Preparation of aluminium triiodide

A.1 Reagent
A.1.1 Acetonitrile for HPLC
A.1.2 Aluminium, with purity more than 99.9%
A.1.3 Iodine, with purity more than 99.8%

A.2 Apparatus
A.2.1 Analytical balance weighing to an accuracy of 0.01 g
A.2.2 Distillation flask with flat bottom, 100 ml
A.2.3 Oil bath with thermostatic float, ±1 °C
A.2.4 Condenser tube, allihn or Graham, matching the distillation flask neck (A.2.2)

A.3 Procedure

1) Weight 3.2 g iodine (A.1.3) and 0.4 g aluminium (A.1.2) into a 100 ml flask (A.2.2), pipette 10 ml of acetonitrile (A.1.1) to the flask and shake the flask slightly to mix the contents.

2) Put the flask in an oil bath (A.2.3) and fit a condenser tube (A.2.4).

3) Heat the flask at 90 °C under reflux condition until the iodine colour disappears (approximately 2 h), yielding aluminium triiodide (white precipitate), which is ready for use.

NOTE All the glassware, such as flask and condenser tube, shall be water-free and rinsed with acetonitrile (A.1.1) prior to use.
Annex B
(informative)

Example of HPLC chromatograms

B.1 HPLC conditions for Figure B.1

As the instrumental equipment of the laboratories may vary, no generally applicable instructions can be provided for chromatographic analysis. The following parameters have been successfully tested and used.

- Stationary phase: C\textsubscript{18} reverse phase
- Mobile phase: 70 % methanol / 30 % water
- Flow rate: 1,0 mL/min
- Column temperature: 35 °C
- Injection volume: 10,0 μL
- Detection: DAD or FLD, spectrograph
- Quantification: for DAD at 225 nm, for FLD with Ex = 230 nm and Em = 296 nm

![Chromatogram of NP and OP in acetonitrile (HPLC-DAD)](image)

Key

<table>
<thead>
<tr>
<th>X</th>
<th>min</th>
</tr>
</thead>
<tbody>
<tr>
<td>Y</td>
<td>absorbance unit mAU</td>
</tr>
</tbody>
</table>

1 4-tert-octylphenol (OP), 7.015 min
2 4-nonylphenol (NP), 7.931 min

Figure B.1 — Chromatogram of NP and OP in acetonitrile (HPLC-DAD)
Figure B.2 — HPLC/DAD – UV-VIS Spectrum of alkylphenols

Key

X  n m

Y  absorbance unit mAU
Annex C
(informative)

Example of GC-MS chromatograms

C.1 Gas-Chromatographic conditions for Figure C.1 and Figure C.2

— Injection: Splitless
— Injector temperature: 250 °C
— Injection volume: 1 μL
— Transfer line temperature: 280 °C
— Carrier gas: Helium
— Flow rate: 1 mL/min
— Temperature programme: 80 °C for 1 min; 20 °C/min to 180 °C for 2 min, 5° C/min to 195 °C for 1 min, 20° C/min to 280 °C for 10 min
— GC column: Capillary gas chromatographic column in glass 5 % phenyl 95 % dimethyl polysiloxane optimised for MS (e.g., Zebron ZB-5ms; Varian VF-5ms; Agilent HP-5ms or DB-5ms; Restek Rtx-5ms). Column length: 30 m; Inner diameter: 0,25 mm; thickness film: 0,5 μm

C.2 MS conditions for Figure C.1 and Figure C.2

— Type: quadrupole (electron impact mode)
— Mode: SIM (see Table C.1)
— Mass range: 40-300 amu
— MS source: 230 °C
— MS quadrupole: 150 °C
— Sovent delay: 5 min

Table C.1 — Diagnostic ions selected for the identification and quantification

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Abbreviation</th>
<th>Ions</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-nonylphenol (mixture of isomers)</td>
<td>NP</td>
<td>107, 121, 135, 149</td>
</tr>
<tr>
<td>4-tert-octylphenol</td>
<td>OP</td>
<td>135, 206</td>
</tr>
<tr>
<td>4-n-nonylphenol</td>
<td>4n-NP</td>
<td>107, 220</td>
</tr>
</tbody>
</table>
Key
X min
Y abundance
1 4-tert-octylphenol (OP), 8.09 min
2 4-nonylphenol (NP), mixture of isomers, from 9.1 min to 10.1 min
3 4-n-nonylphenol (4n-NP), internal standard, 11.56 min

Figure C.1 — Chromatogram of NP and OP standard (GC-MS/SIM)

Key
X min
Y abundance
1 4-tert-octylphenol (OP), 8.09 min
2 4-nonylphenol (NP), mixture of isomers, from 9.1 min to 10.1 min
3 4-n-nonylphenol (4n-NP), internal standard, 11.56 min

Figure C.2 — Chromatogram of yielded NP and OP isolated from the cleavage resultants (GC-MS/SIM)
### Bibliography


2. ISO 18857-2: 2009 Water quality - Determination of selected alkylphenols - Part 2: Gas chromatographic-mass spectrometric determination of alkylphenols, their ethoxylates and bisphenol A in non-filtered samples following solid-phase extraction and derivatisation.

3. ISO 24293: 2009 Water quality - Determination of individual isomers of nonylphenol -- Method using solid phase extraction (SPE) and gas chromatography/mass spectrometry (GC/MS).


