INTERNATIONAL STANDARD

ISO 18218-2

IULTCS/IUC 28-2

Second edition
2019-06

Leather — Determination of ethoxylated alkylphenols —

Part 2: Indirect method

Cuir — Détermination des alkylphénols éthoxylés —

Partie 2: Méthode indirecte

Reference numbers
ISO 18218-2:2019(E)
IULTCS/IUC 28-2:2019(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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 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.

This second edition cancels and replaces the first edition (ISO 18218-2:2015), which has been technically revised as follows:

— 6.14 and 6.15 have been added;

— 7.4, 7.5 and 7.6 have been technically revised;

— 8.1 has been revised by including a reference to 6.14.

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 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 shall 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 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).

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, $n \geq 1$)

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].'

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 analysis 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 document specifies 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 document specifies 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 document 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 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 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

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

$NPEO_9$ and $OPEO_{10}$, 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 no.
4-nonylphenol (mixture of isomers)	C_9H_{19}-C_6H_4-OH	NP	84852–15–3
4-tert-octylphenol	C_8H_{17}-C_6H_4-OH	OP	140–66–9
Nonylphenol ethoxylate	C_9H_{19}-C_6H_4-$(OC_2H_4)_n$OH (n≈9)	$NPEO_9$	9016–45–9
Octyphenol ethoxylate	C_8H_{17}-C_6H_4-$(OC_2H_4)_n$OH (n≈10)	$OPEO_{10}$	9002–93–1
Key CAS: chemical abstract service			

NOTE There are many CAS numbers for the nonylphenol ethoxylates and octylphenol ethoxylates. The CAS numbers in Table 1 are for the normalized structures used in the external calibration (see 8.2).

5 Apparatus and materials

Normal laboratory apparatus and, in particular, the following:

5.1 Analytical balance, weighing to an accuracy of 0,1 mg.

5.2 Ultrasonic bath, (40 ± 2) kHz, with thermostat capable of maintaining a temperature of (50 ± 5) °C.

5.3 Separating funnels, 150 ml.

5.4 Rotary evaporator, with thermostat and vacuum system.

5.5 Membrane filter, polyamide, 0,45 µm.

5.6 HPLC, equipped with diode array detector (DAD) or fluorescence detector (FLD).

5.7 GC, equipped with mass selective detector (MSD).

5.8 Filter paper, fast, quantitative.

6 Chemicals

Unless otherwise stated, analytical grade chemicals shall be used.

6.1 Acetonitrile, for HPLC.

6.2 n-Hexane.

NOTE Iso-hexane can also be used.

6.3 Aluminium triiodide, commercially available, or prepared according to Annex A.

6.4 Sulfuric acid solution, 0,5 mol/l.

6.5 Sodium thiosulfate solution, saturated at room temperature.

6.6 Anhydrous magnesium sulfate ($MgSO_4$), for analysis.

6.7 Anhydrous sodium sulfate (Na_2SO_4), as desiccant for analysis. If not already an anhydrous powder, it can be treated at 800 °C for 4 h, store dry.

NOTE Other suitable desiccants can be used.

6.8 Sodium chloride solution, saturated at room temperature.

6.9 NP (in Table 1) **solution**, for calibration, 1 000 mg/l in n-hexane.

6.10 OP (in Table 1) **solution**, for calibration, 1 000 mg/l in n-hexane.

6.11 OPEO (in Table 1) **solution**, for calibration, 2 000 mg/l in acetonitrile. Dilute this solution with acetonitrile (6.1) if a calibration is applied.

6.12 NPEO (in Table 1) **solution**, for calibration, 4 000 mg/l in acetonitrile. Dilute this solution with acetonitrile (6.1) if a calibration is applied.

6.13 4n-nonylphenol (4n-NP, CAS no. 104-40-5) **solution**, 1 000 mg/l in acetonitrile. The 4n-NP can be used as an internal standard for GC-MS analysis. Dilute this solution with acetonitrile (6.1) if the internal calibration curve is applied.

6.14 4n-nonylphenol (4n-NP, CAS no. 104-40-5) **solution**, 1 000 mg/l in n-hexane. The 4n-NP can be used as an internal standard for GC-MS analysis. Dilute this solution with in n-hexane (6.2) if the internal calibration curve is applied.

6.15 Distilled or deionised water, according to ISO 3696:1987, Grade 3.

7 Sampling and sample preparation

7.1 Preparation of leather samples

7.1.1 Sampling and preparation of samples

Sample the leather according to ISO 2418. 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.

Prepare the leather sample in accordance with ISO 4044.

7.1.2 Sample extraction

Accurately weigh to 10 mg approximately 2,5 g of the leather sample (7.1.1) into an Erlenmeyer flask, then mix with approximately 3 g of Na_2SO_4 (6.7). Add a (50,0 ± 1) ml aliquot of acetonitrile (6.1) into the flask, then close with a stopper.

For GC-MS analysis, an internal standard shall be used. Add a 100 µl aliquot of 4n-NP solution (6.13) to the flask to achieve the final concentration of 2,0 mg/l.

Put the flask into an ultrasonic water bath (5.2) and extract at (50 ± 5) °C for (60 ± 5) min. Then cool the flask to room temperature.

Filter the extracts through a fast, quantitative filter paper (5.8) to remove leather and salt particles. Collect at least 30 ml of the filtrate for the analysis as in 7.4 and 7.5.

7.2 Preparation of leather process auxiliary samples

Accurately weigh to 10 mg approximately 0,5 g of leather auxiliary sample into a flask, carefully mixed with approximately 2 g of $MgSO_4$ (6.6). Then use acetonitrile (6.1), 3 × 7 ml (approximately), to dissolve the sample by stirring with a glass rod. Filter the extracts through a quantitative filter paper. If the extracts contain insoluble material, centrifuge the extract. Collect the extracts in a 50 ml volumetric flask and fill to 50,0 ml with acetonitrile.

For GC-MS analysis, an internal standard shall be used. Add a 100 µl aliquot of 4n-NP solution (6.13) to the flask to achieve the final concentration of 2,0 mg/l.

7.3 Blank determination

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

7.4 Determination of OP and NP

For HPLC analysis, use the sample extracts, either (7.1.2) or (7.2), directly after filtering through a polyamide membrane (5.5).

For GC-MS analysis, add 10,0 ml of the sample extract, either (7.1.2) or (7.2), to a separating funnel (5.3). Subsequently, add approximately 20 ml of water (6.15) and 1 ml of sulfuric acid solution (6.4). Extract the mixture with 2 × 20 ml (approximately) of n-hexane (6.2), separate, and collect the organic phase. After that, wash the n-hexane extracts with approximately 30 ml of water, remove the aqueous layer, and dehydrate the organic layer with approximately 5 g of Na_2SO_4 (6.7). Remove the organic solvent by rotary evaporator (5.4) at approximately 50 °C. Redissolve the residues in (10,0 ± 0,1) ml of n-hexane (6.2) and the solution is then ready for GC-MS analysis after filtering through a polyamide membrane (5.5).

If the organic phase cannot separate freely in the funnel after treating with n-hexane, add approximately 30 ml of saturated sodium chloride (6.8) to the funnel, then shake the mixture for approximately 30 s and stand until separated.

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

7.5 Determination of OPEO and NPEO

Prepare aluminium triiodide (6.3) in acetonitrile for the cleavage of NPEO and OPEO according to Annex A.

Aluminium triiodide is extremely air and water sensitive. If commercial aluminium triiodide (6.3) 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 (7.1.2 or 7.2) into the flask containing approximately 1 g of aluminium triiodide, and continue refluxing at (90 ± 2) °C for (30 ± 5) min.

Take out the flask and slowly add water (6.15) dropwise until the reaction subsides. Dilute the contents with approximately 20 ml of water (6.15) and cool to room temperature.

Add the mixture to a separating funnel (5.3), rinse the flask with approximately 20 ml of n-hexane (6.2), and transfer the organic solution to the funnel. Then add approximately 1 ml of sulfuric acid solution (6.4) and shake. Collect the organic phase and extract the aqueous phase with another 20 ml of n-hexane. Combine all the organic phase. Subsequently, add approximately 2 ml of sodium thiosulfate solution (6.5) and shake until the pink colour (from iodine) disappears. Wash the organic phase with approximately 30 ml of water (6.15), remove the aqueous layer and dehydrate the organic layer with approximately 4 g of Na_2SO_4 (6.7). Remove the organic solvent by rotary evaporator at approximately 50 °C.

If the organic phase cannot separate freely in the funnel after treating with n-hexane, add approximately 30 ml of saturated sodium chloride (6.8) to the funnel, then shake the mixture for approximately 30 s and stand until separated.

For HPLC analysis, redissolve the residues in (10,0 ± 0,1) ml of acetonitrile (6.1) and filter through a polyamide membrane (5.5).

For GC-MS analysis, redissolve the residues in (10,0 ± 0,1) ml of n-hexane (6.2) and filter through a polyamide membrane (5.5).

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

7.6 Chromatographic analysis

Detection of NP and OP can be performed using the chromatographic techniques HPLC (5.6) or GC-MS (5.7). Other validated methods may be used. The quantification is performed by means of HPLC or GC-MS. Where gas chromatography is used, an appropriate internal standard (6.13) or (6.14) shall be used.

Examples of suitable operating parameters and of the chromatographic analysis for NP and OP are listed in Annex B for HPLC and Annex C for GC-MS. The diagnostic ions for the identification and quantification are listed in Table C.1. Figures B.1 and B.2 show HPLC chromatograms and Figures C.1 and C.2 show GC-MS chromatograms.

7.7 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.

8 Calibration

8.1 Calibration for OP and NP

The external 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. For example, standards of 1 mg/l, 5 mg/l, 10 mg/l, 15 mg/l and 20 mg/l.

For GC-MS, each standard contains a 100 µl aliquot of the 4n-NP internal standard solution (6.14) with a constant 4n-NP concentration of 2,0 mg/l. For internal calibration curves, plots are made by measuring five levels of increasing concentration of NP and OP standards in the range 1 mg/l to 20 mg/l with a constant 4n-NP concentration as the internal standard.

8.2 Calibration for OPEO and NPEO

For the external calibration curves of NPEO and OPEO, 10,0 ml of acetonitrile spiked with $NPEO_9$ and $OPEO_{10}$ (listed in Table 1) standards are prepared in the range 2 mg/l to 50 mg/l. For example, standards of 2 mg/l, 10 mg/l, 20 mg/l, 30 mg/l and 50 mg/l.

For GC-MS, each standard contains a 100 µl aliquot of the 4n-NP internal standard solution (6.13) with a constant 4n-NP concentration of 2,0 mg/l.

The solutions are treated as specified in 7.2 to 7.5. The external calibration curves are made by plotting five pairs of the given amounts of $NPEO_9$ and $OPEO_{10}$ and the signal response of the yielded NP and OP. The internal calibration curves are made by measuring the five levels of increasing concentrations of $NPEO_9$ and $OPEO_{10}$ and the signal response of the yielded NP and OP with a constant 4n-NP concentration of 2,0 mg/l as internal standard.

9 Calculation

9.1 Calculation of OP and NP

Calculate the concentration of OP and NP by using the external standard according to Formula (1).

$$w_{AP} = \frac{A_{AP1}}{\rho} \times \frac{V}{m_E} \quad (1)$$

If the internal standard was used, the calculation is according to Formula (2).

$$w_{AP} = \frac{A_{AP1}}{A_{ISTD}} \times \frac{1}{\rho} \times \frac{V}{m_E} \quad (2)$$

where

w_{AP} is the mass portion of NP or OP in the specimen, in mg/kg;

ρ is the slope of the calibration curve;

A_{AP1} is the area response of NP or OP in the specimen solution;

A_{ISTD} is the area of the internal standard in the specimen solution;

V is the volume the specimen is made up, in ml;

m_E is the mass of the leather specimen or leather process auxiliary, in g.

9.2 Calculation of OPEO and NPEO

Calculate the concentration of NPEO and OPEO by using the external standard according to Formula (3).

$$w_{APEO} = \frac{(A_{AP2} - A_{AP1})}{\rho'} \times \frac{V'}{m_E} \quad (3)$$

If the internal standard was used, the calculation is according to Formula (4).

$$w_{APEO} = \frac{(A_{AP2} - A_{AP1})}{A'_{ISTD}} \times \frac{1}{\rho'} \times \frac{V'}{m_E} \quad (4)$$

where

w_{APEO} is the mass portion of NPEO or OPEO in the specimen, in mg/kg;

A_{AP2} is the area of NP or OP in the specimen solution of the cleaved sample NPEO and OPEO;

A_{AP1} is the area of NP or OP in the specimen solution of the uncleaved sample [see Formula (1)];

ρ' is the slope of the calibration curve;

V' is the volume the specimen is made up, in ml;

m_E is the mass of the leather specimen or leather process auxiliary, in g [see Formula (1)];

A'_{ISTD} is the area of the internal standard in the specimen solution.

NOTE The NP and OP is stable during the cleavage process. Thus, the area response (A_{AP1}) of NP and OP in the sample extracts contributed to the area response (A_{AP2}) 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, ($A_{AP2} - A_{AP1}$) is used when calculating the contents of NP and OP yielded from the NPEO and OPEO.

10 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 analysed 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
(normative)

Preparation of aluminium triiodide

A.1 Reagents

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 or other suitable heating mantel with thermostat control, ± 1 °C.

A.2.4 Condenser tube, matching the distillation flask neck (A.2.2).

A.3 Procedure

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

2) Weigh 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.

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

4) 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.

Annex B
(informative)

Example of HPLC chromatograms

B.1 HPLC conditions

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_{18} reverse phase;

— mobile phase: 70 % methanol/30 % water;

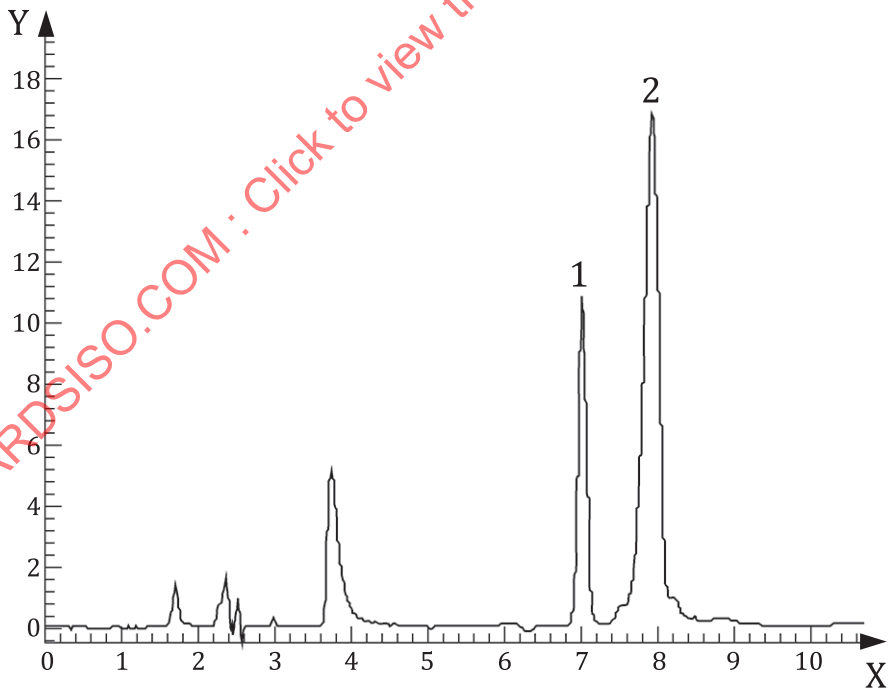
— flow rate: 1,0 mL/min;

— 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.



Key

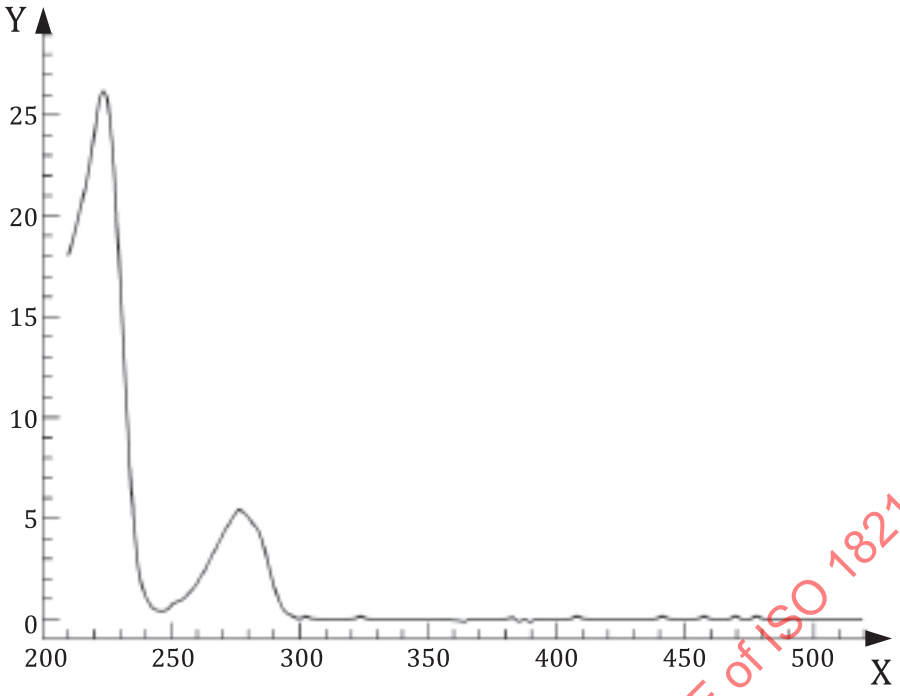
X time, min

Y absorbance unit, mAU

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)



Key

X wavelength, nm

Y absorbance unit, mAU

Figure B.2 — HPLC/DAD — UV-VIS spectrum of alkylphenols

Annex C
(informative)

Example of GC-MS chromatograms

C.1 Gas chromatographic conditions

— 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[a], Varian™ VF-5ms[a], Agilent™ HP-5ms or DB-5ms[a], Restek™ Rtx-5ms[a]. Column length: 30 m; inner diameter: 0,25 mm; thickness film: 0,5 µm

[a] Zebron™ ZB-5ms, Varian™ VF-5ms, Agilent™ HP-5ms or DB-5ms and Restek™ Rtx-5ms 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 of these products.

C.2 MS conditions

— Type: Quadrupole (electron impact mode)

— Mode: SIM (see Table C.1)

— Mass range: 40 amu to 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

Analyte	Abbreviation	Ions
4-nonylphenol (mixture of isomers)	NP	107, 121, 135, 149
4-tert-octylphenol	OP	135, 206
4-n-nonylphenol	4n-NP	107, 220