भारतीय मानक

Indian Standard

IS 582 (Part 15/Sec 2) : 2024
ISO 18218-2 : 2019

चमड़े के रासायनिक परीक्षण की पद्धतियाँ

भाग 15 एथोक्सीलेटेड एल्काइलफेनोल्स (एपीईओ) का निर्धारण

अनुभाग 2 अप्रत्यक्ष पद्धति

Methods of Chemical Testing of Leather

Part 15 Determination of Ethoxylated Alkylphenols (APEO)

Section 2 Indirect Method

ICS 59.140.30

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Leather, Tanning Materials and Allied Products Sectional Committee, CHD 17

NATIONAL FOREWORD

This Indian Standard (Part 15/Sec 2) which is identical to ISO 18218 -2 : 2019 'Leather — Determination of ethoxylated alkylphenols — Part 2: Indirect method' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Leather, Tanning Materials and Allied Products Sectional Committee and approval of the Chemical Division Council.

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.

This standard 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 Committee responsible for formulating this standard has decided to harmonize the methods of test prescribed in IS 582 with those prescribed in ISO/IULTCS standards. Accordingly, the committee decided to retain IS 582 and publish the harmonized/adopted test methods published by ISO/IULTCS in various parts of IS 582 as this standard is widely recognized by the Indian Leather Industry.

The committee further decided to publish the adopted/harmonized standards in the following manner:

a) Wherever an existing test method prescribed in IS 582 is being replaced by the corresponding ISO/IULTCS test method, the relevant part will be published with the information in the national foreword about the method of IS 582 being superseded; and

b) When a new test method is being incorporated in IS 582, the same will be published as a new standard and as subsequent part of IS 582.

This Indian Standard is published in several parts. The other parts in this series are:

Part 1 Determination of volatile matter

Part 2 Determination of water-soluble matter, water soluble inorganic matter and water-soluble organic matter

Part 3 Determination of sulphate total ash and sulphated water-insoluble ash

Part 4 N-methyl-2-pyrrolidone (NMP) in leather

Part 5 Determination of certain azo colourants in dyed leather

Sec 1 Certain aromatic amine derived from azo colourants

Sec 2 4-aminoazobenzene

Part 6 Determination of metal content

Sec 1 Extractable metals

Sec 2 Total metal content

Part 7 Quantitative analysis of tanning agents by filter method

Part 8 Determination of the Preservative (TCMTB, PCMC, OPP, OIT) content in leather by liquid chromatography

Sec 1 Acetonitrile extraction method

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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.

Indian Standard

METHODS OF CHEMICAL TESTING OF LEATHER

PART 15 DETERMINATION OF ETHOXYLATED ALKYLPHENOLS (APEO)

SECTION 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).

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

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

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

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;

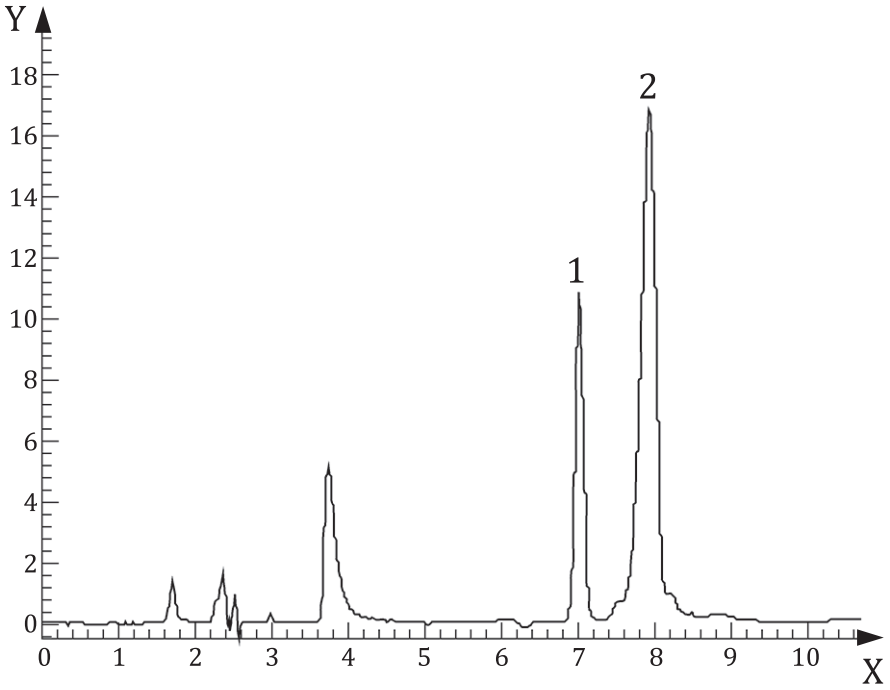
— flow rate: 1,0 mL/min;

— 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

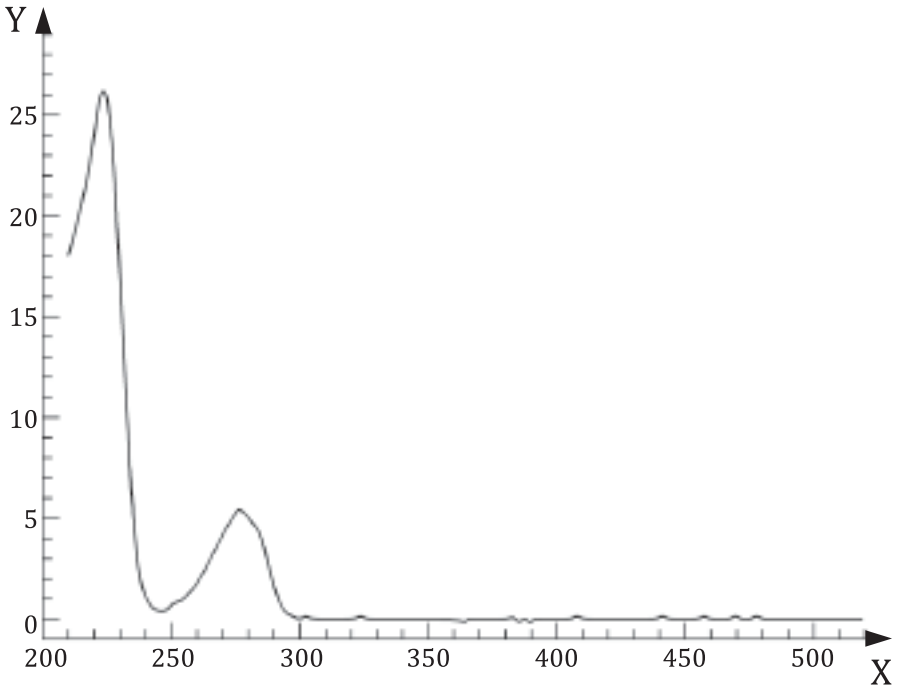
X time, min

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

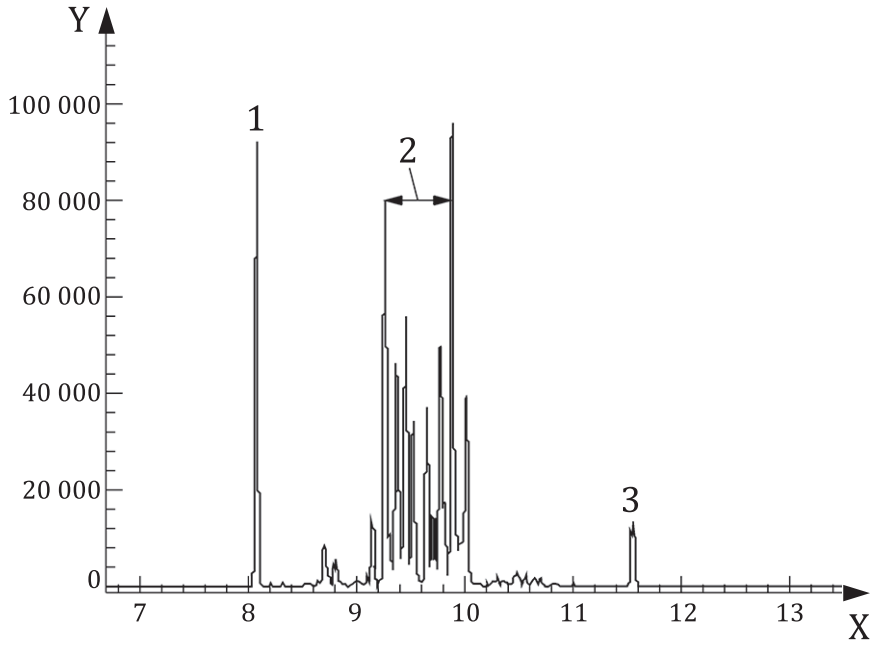
— MS source: 230 °C

— MS quadrupole: 150 °C

— Sovent delay: 5 min

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

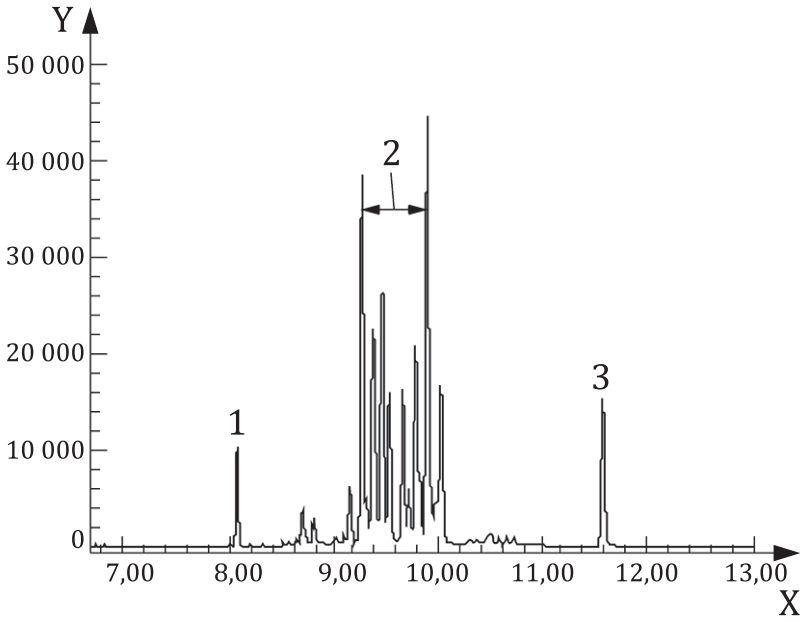
Analyte	Abbreviation	Ions
4-nonylphenol (mixture of isomers)	NP	107, 121, 135, 149
4-tert-octylphenol	OP	135, 206
4-n-nonylphenol	4n-NP	107, 220



Key

X time, 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 time, 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

[1] ISO 18218-1, *Leather — Determination of ethoxylated alkylphenols — Part 1: Direct method*

[2] ISO 18857-1, *Water quality — Determination of selected alkylphenols — Part 1: Method for non-filtered samples using liquid-liquid extraction and gas chromatography with mass selective detection*

[3] ISO 18857-2, *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*

[4] ISO 24293, *Water quality — Determination of individual isomers of nonylphenol — Method using solid phase extraction (SPE) and gas chromatography/mass spectrometry (GC-MS)*

[5] Ma H.-W., Zhang L., Huang X.-X. Determination of ethoxylated nonylphenol and octylphenol in leather by cleavage treatment combined with GC-MS. The 8th Asian International Conference on Leather Science & Technology, 12-14, November, 2010, Kolkata, India

[6] Ma H.-W., & Cheng Y. Determination of free and ethoxylated alkylphenols in leather with gas chromatography-mass spectrometry. *J. Chromatogr. A.* 2010, **1217** pp. 7914–7921

[7] Ma H.-W., & Zhang L. Determination of alkylphenols originated from alkylphenol ethoxylates by cleavage treatment combined with GC-MS. *Anal. Lett.* 2011, **44** pp. 2423–2437

[8] Ma H.-W., & Huang X.-X. APEOs in leather processing — A survey. Proceedings of 31st IULTCS Congress, September 2011, Valencia, Spain

(*Continued from second cover*)

Sec 2 Artificial perspiration extraction method

Part 9 Determination of *p*H and difference figure

Part 10 Determination of chromic oxide

Sec 1 Quantification by titration

Sec 3 Quantification by atomic absorption spectrometry

Sec 4 Quantification by inductively coupled plasma (ICP)

Part 11 Determination of chromium (VI) content

Sec 1 Colorimetric method

Sec 2 Chromatographic method

Part 12 Determination of nitrogen content and hide substance by titrimetric method

Part 13 Determination of total silicon content by reduced molybdosilicate spectrometric method

Part 14 Determination of matter soluble in dichloromethane and free fatty acid content

Part 15 Determination of ethoxylated alkylphenols

Sec 1 Direct method

The text of ISO standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions and terminologies are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'; and

b) Comma (,) has been used as a decimal marker in the International Standard, while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards, which are to be substituted in their respective places, are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 2418 Leather — Chemical, physical mechanical and fastness tests — Position and preparation of specimens for testing	IS 5868 (Part 2) : XXXX Leather — Method of sampling: Part 2 Position and preparation of specimens for testing for chemical physical mechanical and fastness tests (*second revision*) (*under preparation)*	Identical
ISO 4044 Leather — Chemical tests — Preparation of chemical test samples	IS 16256 : 2022 Leather — Chemical tests — Preparation of chemical test samples	Identical

The Committee has reviewed the provisions of the following International Standards referred in this adopted standard and has decided that they are acceptable for use in conjunction with this standard.

International Standard	*Title*
ISO 3696	Water for analytical laboratory use — Specification and test methods

In this adopted standard, reference appears to certain International Standards where the standard atmospheric conditions to be observed are stipulated which are not applicable to tropical/subtropical countries. The applicable standard atmospheric conditions for Indian conditions are 27 °C ± 2 °C and (65 ± 5) percent, relative humidity and shall be observed while using this standard.

In reporting the result of a test or analysis made in accordance with this standard, if the final value,observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'.

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.: CHD 17 (24674).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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