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**Textiles — Method for the detection and determination of  
alkylphenolethoxylates (APEO)**

*Textiles — Méthode de détection et de détermination des alkylphénols éthoxylés (APEO)*

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## Foreword

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ISO 18254 was prepared by the European Committee for Standardization (CEN) in collaboration with ISO Technical Committee ISO/TC 38, Textiles, Subcommittee SC, and by Technical Committee CEN/TC 248, Textiles and textile products in collaboration accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

## Introduction

Alkylphenol ethoxylates (APEOs) are high-value products commonly used in industrial and consumer detergents and cleaners, some plastics and many industrial applications. Their “down the drain” uses may result in their presence in wastewater streams and receiving water bodies. Human exposure to APEO can occur through diverse sources such as environmental, food, or skin contact. Considering their toxicity on several animal species, minimization of exposure to APEO is recognized as important to the preservation of human health.

Nonylphenol ethoxylates belong to the non-ionic surfactant category and are of particular concern. The biodegradation of nonylphenol ethoxylate releases the branched nonylphenol, which is difficult to biodegrade. Nonylphenol is a substance having endocrine disruptive properties that can have serious effects on aquatic and many other organisms. For this reason, the release of nonylphenol ethoxylate into the environment should be avoided.

Chemical products containing nonylphenol and/or nonylphenol ethoxylates in concentrations equal to or greater than 0,1% are restricted within the EU for specific uses, among others, the processing of leather and textiles, industrial, and institutional cleaning.

This restriction is part of the entry 46 of Annex XVII of the REACH regulation EU-1907/2006, which repealed the former Directive 2003/53/EC.

The current restriction is due to be widened to apply to textile products that can be washed in water. A limit value of 0,01% (100 ppm) is expected.

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# Textiles — Method for the detection and determination of alkylphenol ethoxylates (APEO)

~~SAFETY PRECAUTIONS — It is the user's responsibility to use safe and proper techniques in handling materials in this test method. Consult manufacturers for specific details such as material safety, data sheets, and other recommendations. Good laboratory practice should be followed. Users should comply with any national and local safety regulations.~~

## 1 Scope

The ~~International Standard describes~~ analyses ~~described in this standard~~ that are used to detect extractable alkylphenol ethoxylates (nonylphenol ethoxylates and octylphenol ethoxylates) in textile products. This ~~standard requires the use of~~ International Standard provides a method that uses Liquid Chromatograph (LC) ~~with~~ Mass Spectrometry (MS) system to detect and quantify alkylphenol ethoxylates of defined ethoxylate chain length.

## 2 Principle

The textile sample is cut into small pieces, transferred to a vial, and extracted with methanol using ultrasound. The extract is filtered and not subjected to any additional cleaning. Subsequently, the methanol extract is analysed by Liquid Chromatography (LC) ~~with~~ Mass Spectrometry (MS).

## ~~3 Safety precautions~~

~~SAFETY PRECAUTIONS — It is the user's responsibility to use safe and proper techniques in handling materials in this test method. Consult manufacturers for specific details such as material safety data sheets and other recommendations. Good laboratory practice should be followed. Users should comply with any national and local safety regulations.~~

## 4 Reagents

### 4.1 General

During the analysis, unless otherwise stated, only reagents of recognized analytical grade shall be used.

NOTE OPEO and NPEO are available currently as technical grade.

~~3.1 Solvents shall be~~ of quality for HPLC analysis.

~~4.2 Octylphenol ethoxylates~~, (Triton<sup>®1</sup> X-100), (OPEOs) CAS no. 9002-93-1, Sigma-Aldrich<sup>®</sup> Part number T9284 (see ~~note~~ Note in 3.3).

<sup>1</sup> Triton<sup>®</sup> is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

**43.3 Nonylphenol ethoxylates.** (IGEPAL®<sup>2</sup> CO-630), (NPEOs) CAS no. 68412-54-4, Sigma-Aldrich® Part number 542334 (see ~~note~~Note).

NOTE The mentioned brand names in 43.2 and 43.3 are given to improve the comparability of the test results amongst laboratories. Using another batch or another supplier could lead to different results.

**43.4 Methanol.**

**43.5 Acetonitrile (ACN).**

**43.6 HPLC grade water.**

**43.7 30% Formic acid (v/v), volume fraction of 30 %.**

**43.8 Ammonium acetate.**

**43.9 10 mM Ammoniumammonium acetate, pH 3,6.**

**3.9.1** 0,771-g of ammonium acetate is dissolved in 900-ml of water (43.6).

**3.9.2** 10-ml of ACN (43.5) ~~are~~is added and mixed well.

**3.9.3** The pH is adjusted to 3,6 with 30% (v/v) ~~volume fraction of 30 %~~ formic acid and made up to the mark with water (43.6) in a ~~111~~ volumetric flask.

**3.9.4** The buffered solution should be filtered before use.

## 54 Apparatus

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### 5.14.1 Apparatus and auxiliaries for preparing the sample

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**54.1.1 Standard laboratory equipment,** ~~and.~~

**54.1.2 Analytical balance,** resolution at 0,01-g ~~(for test specimen preparation).~~

**54.1.3 Analytical balance,** resolution at 0,001-g ~~(for standard preparation).~~

**54.1.4 Glass container with screw top** ~~(40ml, 40 ml has been found suitable).~~

**54.1.5 Ultrasonic water bath,** to be set up at 70 °C ~~+/- ± 5 °C.~~

**54.1.6 Membrane filter,** with 0,45-~~µm~~µm pore size.

**54.1.7 Glass vial,** with septum cap (HPLC vial).

**54.1.8 pH meter,** resolution of 0,1-pH.

<sup>2</sup> IGEPAL® is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.



## 5.24.2 Chromatographic equipment

~~54.2.1 High-performance liquid chromatography~~ **Liquid Chromatography (LC) with Mass Spectrometry (MS) and Electro Spray Ionization (ESI)**.

~~54.2.2 Reverse phase column with guard column.~~

## 65 Procedure

### 6.15.1 Standard preparation

Stock solutions of ~~(Aa)~~ OPEO and ~~(Bb)~~ NPEO are prepared in methanol containing ~~1000~~ **1 000** mg/L OPEO (**4.3.2**) and NPEO (**4.3.3**).

### 6.25.2 Sample preparation

Cut the textile sample into pieces of approximately 5-mm ~~x~~ 5-mm and mix them homogeneously.

Prepare approximately 1-g of the cut textile, weigh it to the nearest 10-mg, and then place it into the glass container (extraction vessel).

Pipette 20-ml of methanol into the glass container (extraction vessel).

Place the glass container (extraction vessel) into an ultrasonic bath at 70°C for (60 ~~+/-~~ **±** 5) min.

Afterwards, let the extract cool down to room temperature.

Filter about 1-ml of the extraction solution into a HPLC vial using a disposable syringe equipped with a membrane filter.

### 6.35.3 Analysis ISO/FDIS 18254 https://standards.iteh.ai/catalog/standards/sist/3b9457bb-f2a8-4626-9786-437f96ba3f16/iso-fdis-18254

The detection and quantification of defined alkylphenol ethoxylates is conducted using ~~Liquid Chromatography (LC) / with Mass Spectrometry system (/MS)~~ with gradient elution and ~~Electro Spray Ionization (ESI)~~ mass spectrometer.

Congeners with 2 to 16 ethoxylate groups shall be used for quantification.

Guidelines for suitable chromatographic conditions are given in ~~the Annex-A~~ for LC/MS and in **Annex-B** for LC/MS/MS.

## 76 Calculation of results

### 7.16.1 Determination of the R value for each congener of APEO

Calibrate the mass fraction (R) for each APEO congener and calculate the concentration of each APEO isomer as follows:

$$\text{A Mass fraction in each APEO (n), } R (\%) = \frac{AO}{AOT} \times 100 \quad (1)$$

where

AO is the area response of each APEO congener;

AOT is the total sum of APEO area responses (from APEO 2 to APEO 16).

Where,