
**Textiles — Method for determination
of alkylphenols (AP)**

Textiles — Méthode de détermination de la teneur en alkylphénols (AP)

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Foreword

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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).

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This document was prepared by Technical Committee ISO/TC 38, *Textiles*.

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.

Textiles — Method for determination of alkylphenols (AP)

WARNING — This document calls for the use of substances/procedures that may be injurious to the health/environment if appropriate conditions are not observed. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety/environment at any stage.

1 Scope

This document specifies the method for the determination of extractable alkylphenols (AP) without derivatization step in textile and textile products.

2 Normative references

There are no normative references in this document.

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

The test specimen is cut into small pieces, transferred to a sample vial and treated with methanol in ultrasonic water bath. The extract is filtered and collected. Subsequently, the collected extract is analysed by gas chromatograph with mass selective detector (GC-MS), liquid chromatograph with mass selective detector (LC-MS) or liquid chromatograph with fluorescence detector (LC-FLD).

5 Reagents

Unless otherwise specified, analytical grade chemicals shall be used.

- 5.1 **4-n-Octylphenol**, CAS No.1806-26-4.
- 5.2 **4-tert-Octylphenol**, CAS No.140-66-9.
- 5.3 **4-n-Nonylphenol**, CAS No.104-40-5.
- 5.4 **4-Nonylphenol**, CAS No. 84852-15-3.
- 5.5 **Methanol**, (HPLC grade).
- 5.6 **Acetone**.
- 5.7 **Acetonitrile**, (HPLC grade).

5.8 Water, (HPLC grade).

6 Apparatus

6.1 General

Clean all glassware by rinsing with acetone ([5.6](#)) prior to use.

6.2 Apparatus and auxiliaries for preparing the sample

6.2.1 Standard laboratory equipment and the following.

6.2.2 Analytical balance with resolution of 0,01 g (for test specimen preparation).

6.2.3 Analytical balance with resolution of 0,001 g (for standard preparation).

6.2.4 Glass vial with screw cap (e.g. 40 ml).

6.2.5 Ultrasonic water bath [to be set up at $(70 \pm 5) ^\circ\text{C}$].

6.2.6 Disposable syringe and membrane filter (with pore size of 0,45 μm or less).

6.2.7 Glass vial with septum cap (for chromatographic instrument).

6.3 Chromatographic equipment

6.3.1 Gas chromatograph with mass selective detector (GC-MS), or

6.3.2 Liquid chromatograph with mass selective detector (LC-MS), or

6.3.3 Liquid chromatograph with fluorescence detector (LC-FLD).

6.3.4 Capillary column for gas chromatograph, (5 %-phenyl)-methylpolysiloxane phase, non-polar, bonded and cross-linked, low bleed.

6.3.5 C18 reverse phase column for liquid chromatograph.

7 Procedure

7.1 Standard preparation

Octylphenols and nonylphenols are weighed accurately with analytical balance ([6.2.3](#)) and dissolved with methanol ([5.5](#)) containing 1 000 mg/l for stock solution. The stock solution can be preserved in the dark at less than 4°C.

7.2 Sample preparation

The test specimen is cut into pieces with 5 mm × 5 mm and pieces are mixed homogeneously. The specimen is weighed 1 g to the nearest 0,01 g with analytical balance ([6.2.2](#)) and put into the 40 ml glass vial ([6.2.4](#)) for extraction.

7.3 Sample extraction

10 ml of methanol (5.5) are added to glass vial (6.2.4) for extraction. If sample is not sufficiently immersed in the extraction solvent, add more methanol and record the final volume for calculation of each AP described in 8.3. Then the sample vial is sonicated for extraction in ultrasonic water bath (6.2.5) at 70 °C for 60 min. Subsequently, the extract is cooled down to room temperature, approximately 1 ml to 2 ml of extract is filtered into sample vial (6.2.7) using disposable syringe equipped with membrane filter (6.2.6). The sample vial is closed with cap immediately for further analysis.

In general, samples are examined without concentration, but the instrumental equipment of the laboratories may vary, the concentration procedure of extract is applicable for lower detection on limit.

7.4 Sample analysis

Qualitative and quantitative analysis of alkylphenols is performed by using GC-MS, LC-MS or LC-FLD.

Guidelines for suitable chromatographic conditions are given in Annex A for GC-MS/MS and in Annex B for LC-MS/MS and LC-FLD.

In case of positive results using GC-MS method, the results shall be confirmed using LC techniques.

NOTE False-positive results using GC-MS method could be caused by decomposition of tris(nonylphenyl) phosphate (TNPP) due to high injection temperature.

8 Calculation and calibration

8.1 Calibration curve

Prepare a calibration curve of the response against the known standard concentration. From the calibration curve, the concentration of the AP (alkylphenols; octylphenols and nonylphenols) in mg/l (C_S) is determined.

NOTE Concentration ranges for the calibration standards are subject to change upon the need of each laboratory and equipment used.

For quantification, the calibration curve shall have a correlation coefficient greater than 0,995 (R^2 greater than 0,990).

8.2 External calibration

The concentration of each AP in sample extract solution is calculated in mg/l according to Formula (1):

$$C_S = \frac{A_S}{A_C} \times C_C \quad (1)$$

where

C_S is the concentration of each AP in sample extract solution, in mg/l;

C_C is the concentration of each AP in calibration solution, in mg/l;

A_S is the area response of each AP in sample extract solution;

A_C is the area response of each AP in calibration solution.

8.3 Calculation of each AP in sample

The AP level is calculated as mass portion, X_i , in mg/kg of the specimen according to [Formula \(2\)](#):

$$X_i = \frac{C_S \times V \times DF}{M} \quad (2)$$

where

X_i is the concentration of each AP in sample, in mg/kg;

C_S is the concentration of each AP and calculated according to [Formula \(1\)](#), in mg/l;

V is the final volume of extract solution, in ml;

M is the mass of test specimen, in g;

DF is the dilution factor.

8.4 Reliability of the method

The reliability of the method is given in [Annex C](#).

9 Test report

The test report shall include the following information:

- a) a reference to this document, i.e. ISO 21084:2019;
- b) identification of sample and the date of analysis;
- c) detection method and quantification method;
- d) the result of each AP (nonylphenols and octylphenols are reported individually);
- e) details of any deviation from the procedure specified and of all circumstances that may have influenced the results.

Annex A (informative)

Examples of chromatographic condition — GC-MS/MS

A.1 Preliminary remark

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

A.2 Gas chromatographic conditions

A.2.1 GC-MS/MS condition

Capillary column: HP-5MS, length 35 m, inside diameter 0,25 mm, film thickness 0,25 µm

Injector mode: Splitless

Injector temperature: 250 °C

Injection volume: 1 µl

Carrier gas: Helium (rate: 1,0 ml/min)

Collision gas: Nitrogen (rate: 1,5 ml/min)

Oven program: 80 °C (0 min), 80 °C to 230 °C (7 °C/min), 230 °C (2 min)

Detection: Tandem mass spectrometer (MS/MS)

Acquisition mode: MRM mode, see [Table A.1](#) for MRM condition.

Table A.1 — MRM condition in GC-MS/MS

Compounds	m/z [Q1]	m/z [Q3]	Collision energy (eV)
4-tert-Octylphenol	135	107	10
	135	77	30
4-n-Octylphenol	107	77	20
	107	51	40
4-n-Nonylphenol	107	77	20
	107	51	40
4-Nonylphenol	107	77	20
	121	77	20
	135	77	30
	149	107	10
	163	107	20
	191	107	20

See examples in [Figure A.1](#) and [Figure A.2](#).

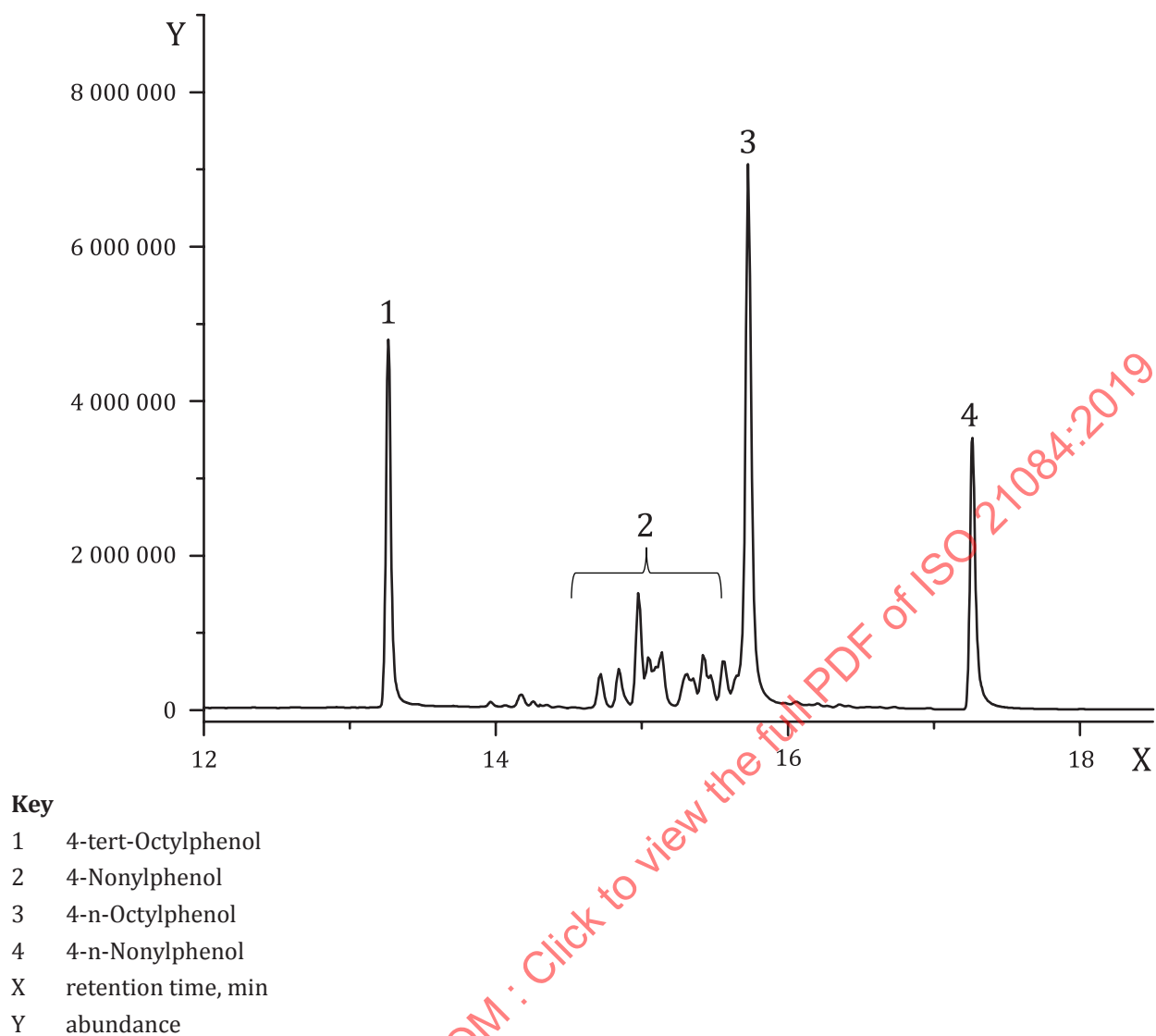


Figure A.1 — Total ion chromatogram (TIC) of alkylphenols in GC-MS/MS

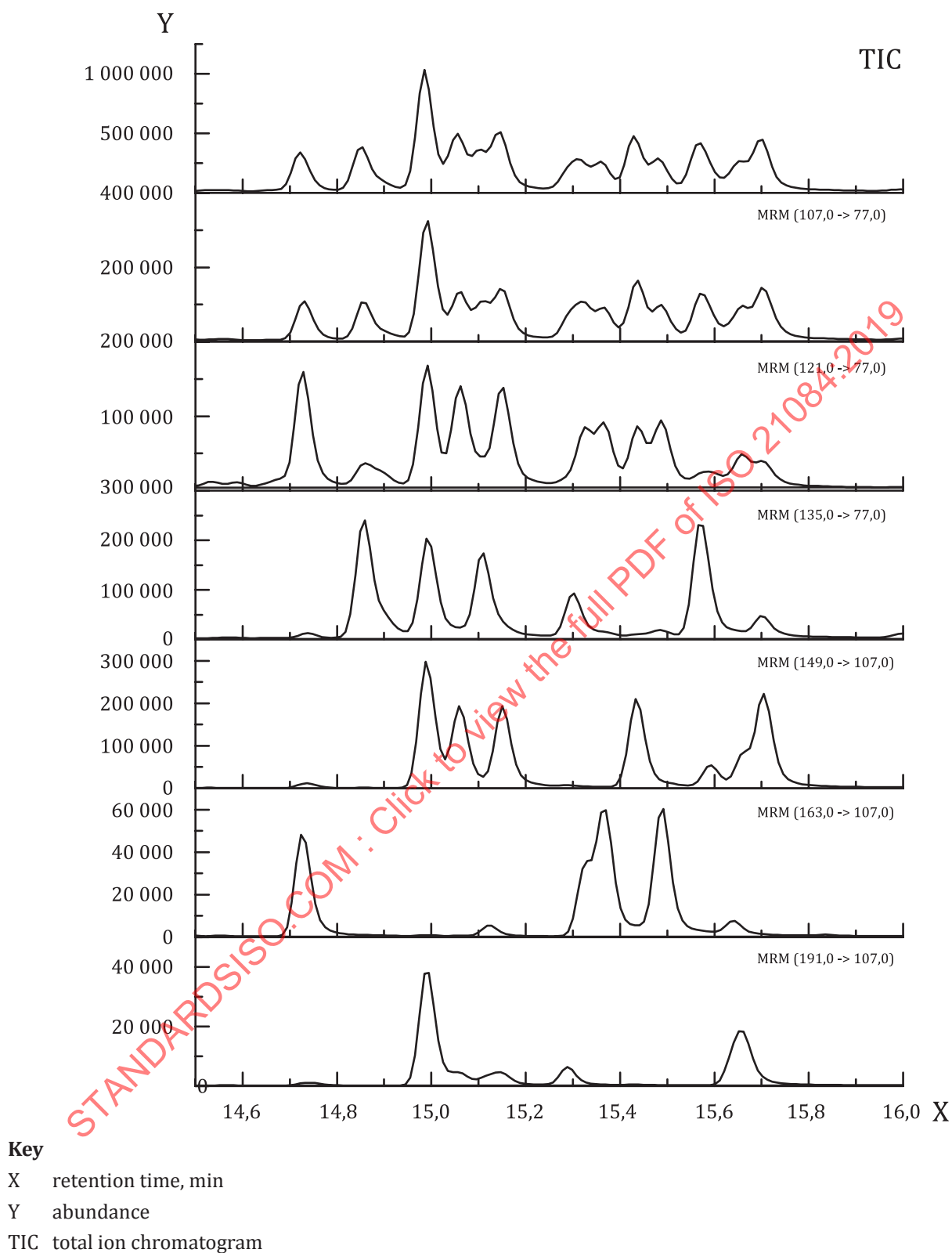


Figure A.2 — MRM chromatogram of 4-nonylphenol (technical mixture) in GC-MS/MS

Annex B (informative)

Examples of chromatographic condition — LC-MS/MS and LC-FLD

B.1 Liquid chromatographic conditions

Mobile phase 1: Acetonitrile (ACN)

Mobile phase 2: Water

Column: ZORBAX Eclipse plus C18 ^{®1)} (2,1 × 100 mm, 1,8 µm, pore size 95 Å)

Column temperature: 40 °C

Flow rate: 0,3 ml/min

Injection volume: 5 µl

Mobile phase condition: 0 min to 12 min 65 % ACN, 12 min to 18 min 98 % ACN

B.2 Detector conditions

B.2.1 Tandem mass spectrometer (MS/MS)

Ionization mode: API-ES

Polarity: Negative

Desolvation temperature: 350 °C

Desolvation gas flow (N₂): 11 l/min

Nebulizer: 50 psi

Sheath gas flow rate: 12 l/min

Sheath gas temperature: 350 °C

Capillary voltage: 3 500 V

Nozzle voltage: 1 000 V

Acquisition mode: MRM mode, see [Table B.1](#) for MRM condition.

Table B.1 — MRM condition in LC-MS/MS

Compounds	m/z[Q1]	m/z[Q3]	Collision energy (eV)
4-tert-Octylphenol	205	133	22
4-Nonylphenol	219	133	30
4-n-Octylphenol	205	106	20
4-n-Nonylphenol	219	106	20

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