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Vattenundersökningar – Bestämning av utvalda alkylfenoler – Del 2: Gaskromatografisk metod med masspektrometrisk bestämning av alkylfenoler, deras etoxylater och bisfenol A i ofiltrerade prover efter fastfas extraktion och derivatisering (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 (ISO 18857-2:2009)

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Europastandarden EN ISO 18857-2:2011 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN ISO 18857-2:2011.

The European Standard EN ISO 18857-2:2011 has the status of a Swedish Standard. This document contains the official version of EN ISO 18857-2:2011.

Förhållandet till övriga delar under samma huvudtitel - Utdrag ur Förord i ISO 18857-2:2009/ Relations to other parts under the same general title - Extract from the Foreword of ISO 18857-2:2009

ISO 18857 consists of the following parts, under the general title *Water quality — Determination of selected alkylphenols:*

- *Part 1: Method for non-filtered samples using liquid-liquid extraction and gas chromatography with mass selective detection*
- *Part 2: Gas chromatographic-mass spectrometric determination of alkylphenols, their ethoxylates and bisphenol A in non-filtered samples following solid-phase extraction and derivatisation*

Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.

Information about the content of the standard is available from the Swedish Standards Institute (SIS), telephone +46 8 555 520 00. Standards may be ordered from SIS Förlag AB, who can also provide general information about Swedish and foreign standards.

Denna standard är framtagen av kommittén för Kemiska vattenundersökningar, SIS/TK 424.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på www.sis.se - där hittar du mer information.

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 18857-2

November 2011

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English Version

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 (ISO 18857-2:2009)

Qualité de l'eau - Dosage d'alkylphénols sélectionnés - Partie 2: Dosage par chromatographie en phase gazeuse-spectrométrie de masse d'alkylphénols, de leurs éthoxylates et du bisphénol A dans des échantillons non filtrés après extraction en phase solide et dérivation (ISO 18857-2:2009)

Wasserbeschaffenheit - Bestimmung von ausgewählten Alkylphenolen - Teil 2: Gaschromatographische-massenspektrometrische Bestimmung von Alkylphenolen, deren Ethoxylaten und Bisphenol A für nichtfiltrierte Proben unter Verwendung der Festphasenextraktion und Derivatisierung (ISO 18857-2:2009)

This European Standard was approved by CEN on 15 October 2011.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

The text of ISO 18857-2:2009 has been prepared by Technical Committee ISO/TC 147 “Water quality” of the International Organization for Standardization (ISO) and has been taken over as EN ISO 18857-2:2011 by Technical Committee CEN/TC 230 “Water analysis” the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2012, and conflicting national standards shall be withdrawn at the latest by May 2012.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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Endorsement notice

The text of ISO 18857-2:2009 has been approved by CEN as a EN ISO 18857-2:2011 without any modification.

Introduction

The user should be aware that particular problems could require the specifications of additional marginal conditions.

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

WARNING — Persons using this part of ISO 18857 should be familiar with normal laboratory practice. This part of ISO 18857 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this part of ISO 18857 be carried out by suitably qualified staff.

1 Scope

This part of ISO 18857 specifies a gas chromatographic-mass spectrometric (GC-MS) determination of selected alkylphenols, their ethoxylates and bisphenol A in non-filtered samples of drinking, ground, surface, and waste waters following solid-phase extraction and derivatisation.

The lower limit of the working range depends on the matrix, on the specific compound to be analysed and on the sensitivity of the mass spectrometric detection unit. The method is applicable in a working range from 0,005 µg/l to 0,2 µg/l for 4-(1,1,3,3-tetramethylbutyl)phenol (OP), and its mono- (OP_1EO) and diethoxylate (OP_2EO), from 0,03 µg/l to 0,2 µg/l for 4-nonylphenol (mixture of isomers) (NP), and its mono- (NP_1EO) and diethoxylate (NP_2EO), and from 0,05 µg/l to 0,2 µg/l for bisphenol A (BPA).

Depending on the matrix, the method is also applicable to waste water in a working range from 0,1 µg/l to 50 µg/l for OP, OP_1EO, OP_2EO and BPA, and from 0,5 µg/l to 50 µg/l for NP, NP_1EO and NP_2EO. The working ranges are based on experimental work carried out in ruggedness testing. Water samples containing suspended matter at concentrations of more than 500 mg/l and waste water samples are extracted by passing a 100 ml sample through the cartridge.

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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes and sampling techniques*

ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of water samples*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

3 Principle

Extraction of the analytes listed in Table 1 from an acidified water sample by solid-phase extraction, solvent elution, derivatisation and determination by GC-MS detection.

Table 1 — Analytes determinable by GC-MS following solid-phase extraction and derivatisation

Analyte	Empirical formula	Abbreviation	CAS[a] No.
4-(1,1,3,3-Tetramethylbutyl)phenol	$C_{14}H_{22}O$	OP	140-66-9
4-(1,1,3,3-Tetramethylbutyl)phenol monoethoxylate	$C_{16}H_{26}O_2$	OP_1EO	—
4-(1,1,3,3-Tetramethylbutyl)phenol diethoxylate	$C_{18}H_{30}O_3$	OP_2EO	—
4-Nonylphenol (mixture of isomers)	$C_{15}H_{24}O$	NP	84852-15-3[b]
4-Nonylphenol monoethoxylate (mixture of isomers)	$C_{17}H_{28}O_2$	NP_1EO	—
4-Nonylphenol diethoxylate (mixture of isomers)	$C_{19}H_{32}O_3$	NP_2EO	—
Bisphenol A	$C_{15}H_{16}O_2$	BPA	80-05-7

[a] CAS: Chemical Abstracts Service.

[b] The commercially produced nonylphenols are predominantly 4-nonylphenol with a varied and undefined degree of branching in the alkyl groups. This mixture of isomers falls under the CAS number 84852-15-3, but CAS numbers 104-40-5 (4-nonylphenol, straight chain) and 25154-52-3 (nonylphenol, straight chain) have also been incorrectly used to denote this isomer mixture.

4 Interferences

4.1 Sampling and extraction

Sampling containers shall consist of materials that do not change the sample when in contact with it. Avoid contact with plastics and other organic materials during sampling, sample storage or extraction.

Commercially available adsorbent materials are often of varying quality. Considerable batch-to-batch differences in quality and selectivity of this material are possible. The recovery of single substances can vary with the concentration. Therefore, check the recovery regularly at different concentrations and whenever new batches are used. Perform calibration and analysis with material from the same batch.

4.2 Gas chromatography-mass spectrometry

Substances with retention times or which produce masses similar to the analytes to be determined can interfere with the determination.

These interferences may lead to incompletely resolved signals and to additional signals in the chromatographic pattern of NP, NP_1EO and NP_2EO. They may, depending on their magnitude, affect accuracy and precision of the analytical results, since all three analytes are determined from the sum of a cluster of eight to ten chromatographic peaks (Table 3 and Annex C). It is important that the interfering peaks are not included in the calculations.

The presence of interfering compounds can, if necessary, be detected by recording full mass spectra (range of mass fragments to monitor m/z = 50 to m/z = 350).

Matrix interferences can be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences varies considerably, depending on the nature of the sample. In drinking water and ground water, matrix interferences usually do not occur.

5 Reagents

The reagents shall not have blank values that would interfere with the GC-MS analysis.

Use solvents and reagents of sufficient purity, i.e. with negligibly low impurities compared with the concentration of analytes to be determined. As reagents, use, as far as available, “residual grade” or better in order to obtain clean blanks. Check blanks regularly and establish proper charge control.

5.1 Water, as specified in ISO 3696, grade 1, or equivalent.

5.2 Acid, e.g. hydrochloric acid, w(HCl) = 37 % mass fraction, or sulfuric acid, $c(H_2SO_4)$ = 1 mol/l.

5.3 Acetone, C_3H_6O.

5.4 Internal standard solutions.

Examples of suitable internal standards are given in Table 2.

Store solutions 5.4.1 and 5.4.2 in a refrigerator protected from light. Check the solutions weekly prior to use.

Table 2 — Internal standards

No.	Name	Abbreviation	CAS No.
1	4-(1,1,3,3-Tetramethylbutyl)phenol (ring-$^{13}C_6$)	OP-$^{13}C_6$	—
2	4-(1,1,3,3-Tetramethylbutyl)phenol monoethoxylate (ring-$^{13}C_6$)	OP_1EO-$^{13}C_6$	—
3	4-(1,1,3,3-Tetramethylbutyl)phenol diethoxylate (ring-$^{13}C_6$)	OP_2EO-$^{13}C_6$	—
4	4-(3,6-Dimethyl-3-heptyl)phenol (ring-$^{13}C_6$)	363 NP-$^{13}C_6$	—
5	4-(3,6-Dimethyl-3-heptyl)phenol monoethoxylate (ring-$^{13}C_6$)	363 NP_1EO-$^{13}C_6$	—
6	4-(3,6-Dimethyl-3-heptyl)phenol diethoxylate (ring-$^{13}C_6$)	363 NP_2EO-$^{13}C_6$	—
7	Bisphenol A-d16	BPA-d16	96210-87-6

5.4.1 Internal standard stock solution.

Use commercially available internal standard solutions or prepare a solution as follows.

Weigh 10 mg of each internal standard (Table 2) separately in a 100 ml one-mark volumetric flask and make up to the mark with acetone (5.3) to give a concentration of each internal standard of 100 ng/µl.

5.4.2 Internal standard working solution.

Dilute the solution (5.4.1) with acetone (5.3) 1→100 to give a final concentration of each internal standard of 1 ng/µl.