
**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**

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 de bisphénol A dans
des échantillons non filtrés après extraction en phase solide et
dérivation*



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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 18857-2 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

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*

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- (OP₁EO) and diethoxylate (OP₂EO), from 0,03 µg/l to 0,2 µg/l for 4-nonylphenol (mixture of isomers) (NP), and its mono- (NP₁EO) and diethoxylate (NP₂EO), 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, OP₁EO, OP₂EO and BPA, and from 0,5 µg/l to 50 µg/l for NP, NP₁EO and NP₂EO. 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 ₁ EO	—
4-(1,1,3,3-Tetramethylbutyl)phenol diethoxylate	C ₁₈ H ₃₀ O ₃	OP ₂ EO	—
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 ₁ EO	—
4-Nonylphenol diethoxylate (mixture of isomers)	C ₁₉ H ₃₂ O ₃	NP ₂ EO	—
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, NP₁EO and NP₂EO. 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.