



# SLOVENSKI STANDARD SIST EN ISO 18857-1:2007

01-februar-2007

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Water quality - Determination of selected alkylphenols - Part 1: Method for non-filtered samples using liquid-liquid extraction and gas chromatography with mass selective detection (ISO 18857-1:2005)

STANDARD PREVIEW

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Wasserbeschaffenheit - Bestimmung ausgewählter Alkylphenole - Teil 1: Verfahren für nichtfiltrierte Proben mittels Flüssig-Flüssig-Extraktion und Gaschromatographie mit massenselektiver Detektion (ISO 18857-1:2005)

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Qualité de l'eau - Dosage d'alkylphénols sélectionnés - Partie 1: Méthode pour échantillons non filtrés par extraction en phase liquide-liquide et chromatographie en phase gazeuse avec détection sélective de masse (ISO 18857-1:2005)

**Ta slovenski standard je istoveten z: EN ISO 18857-1:2006**

### ICS:

13.060.50 Ú!^ã \ aãã[ ä^Á aá^ { ä } ^  
•} [ çã Examination of water for chemical substances

**SIST EN ISO 18857-1:2007**

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**EN ISO 18857-1**

August 2006

ICS 13.060.50

English Version

**Water quality - Determination of selected alkylphenols - Part 1:  
Method for non-filtered samples using liquid-liquid extraction and  
gas chromatography with mass selective detection (ISO 18857-  
1:2005)**

Qualité de l'eau - Dosage d'alkylphénols sélectionnés -  
Partie 1: Méthode pour échantillons non filtrés par  
extraction en phase liquide-liquide et chromatographie en  
phase gazeuse avec détection sélective de masse (ISO  
18857-1:2005)

Qualité de l'eau - Dosage d'alkylphénols sélectionnés -  
Partie 1 : Méthode pur échantillons non filtrés part  
extraction en phase liquide-liquide et chromatographie en  
phase gazeuse avec détection sélective de masse (ISO  
18857-1:2005)

This European Standard was approved by CEN on 3 August 2006.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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**EN ISO 18857-1:2006 (E)****Foreword**

The text of ISO 18857-1:2005 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 18857-1:2006 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 February 2007, and conflicting national standards shall be withdrawn at the latest by February 2007.

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**Endorsement notice**

The text of ISO 18857-1:2005 has been approved by CEN as EN ISO 18857-1:2006 without any modifications.

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2005-01-15

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**Water quality — Determination of  
selected alkylphenols —**

Part 1:

**Method for non-filtered samples using  
liquid-liquid extraction and gas  
chromatography with mass selective  
detection**iTeh STANDARD PREVIEW  
(standards.iteh.ai)*Qualité de l'eau — Dosage d'alkylphénols sélectionnés —**Partie 1: Méthode pour échantillons non filtrés par extraction en phase  
liquide-liquide et chromatographie en phase gazeuse avec détection  
sélective de masse*Reference number  
ISO 18857-1:2005(E)

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## ISO 18857-1:2005(E)

## 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 18857-1 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*  
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- *Part 2: Method for filtered samples using solid phase extraction and gas chromatography with mass selective detection*



# Water quality — Determination of selected alkylphenols —

## Part 1:

### Method for non-filtered samples using liquid-liquid extraction and gas chromatography with mass selective detection

**WARNING** — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard 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 method for the determination of 4-nonylphenol (mixture of isomers) and 4-(1,1,3,3-tetramethylbutyl)phenol in non-filtered samples of drinking water, ground water and surface water. The method is applicable in a concentration range from 0,005 µg/l to 0,2 µg/l for 4-(1,1,3,3-tetramethylbutyl)phenol and from 0,02 µg/l to 0,2 µg/l for 4-nonylphenol (mixture of isomers). Depending on the matrix, the method is also applicable to waste water containing the analyzed compounds in the concentration range from 0,1 µg/l to 50 µg/l. Higher concentrations can be measured after appropriate dilution of the sample.

## 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 reference document (including any amendments) applies.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

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

ISO 5667-2, *Water quality — Sampling — Part 2: Guidance on sampling techniques*

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

## 3 Principle

The compounds 4-(1,1,3,3-tetramethylbutyl)phenol and 4-nonylphenol (mixture of isomers) are extracted from the acidified water sample with toluene. The extract is cleaned, if necessary, with silica and the alkylphenols are separated by gas chromatography using capillary columns. The alkylphenols are identified by mass spectrometry and quantified using an internal standard over the total procedure. The response factor using 4-(1,1,3,3-tetramethylbutyl)phenol and a technical mixture of isomers of 4-nonylphenol is determined daily.

## ISO 18857-1:2005(E)

## 4 Interferences

The extent and the importance of interferences are sample-dependent.

There are many sources of sample contamination, including contamination of reagents during storage, contamination of equipment reused in the sequential extraction of samples and standards and carryover contamination from the septum seal on a sample bottle or vial and GC septa. Organic polymers can form alkyl phenols thus leading to elevated values. Avoid direct contact with plastics material as these can contain alkylphenols that can contaminate the sample.

## 5 Reagents

Use reagents with negligibly low concentrations of target alkyl phenols compared with the concentration to be determined and verify by blank determinations.

**5.1 Water**, grade 1, as specified in ISO 3696:1987.

**5.2 Acid**, e.g. hydrochloric acid,  $c(\text{HCl}) = 37\%$ , or sulfuric acid,  $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/l}$ .

**5.3 Silica**, medium pore size 6 nm (60 Å), grain-size 0,063 mm to 0,2 mm (230 mesh to 70 mesh).

Purify about 100 g of silica in a quartz beaker (6.5) by heating to  $550\text{ }^\circ\text{C} \pm 20\text{ }^\circ\text{C}$  in a muffle furnace (6.6) for at least 10 h. Let the silica cool to about  $100\text{ }^\circ\text{C}$  and transfer it to a wide-necked glass bottle. Let it cool to room temperature in a desiccator. Add water (5.1) to the silica to produce a concentration of about 3 % mass fraction. Homogenize on a shaking device for 2 h. Store tightly sealed.

**5.4 Hexane**,  $\text{C}_6\text{H}_{14}$ .

**5.5 Toluene**,  $\text{C}_7\text{H}_8$ . <https://standards.iteh.ai/catalog/standards/sist/d37c1b1f-85de-48e7-80ad-f4006c39944c/sist-en-iso-18857-1-2007>

Other extracting solvents may be used if equivalent recoveries can be achieved (see Annex C).

**5.6 Sodium sulfate**,  $\text{Na}_2\text{SO}_4$ , anhydrous, powdered.

**5.7 Acetone**,  $\text{C}_3\text{H}_6\text{O}$ .

**5.8 4-*n*-nonylphenol(ring- $^{13}\text{C}_6$ ) solution**,  $\text{C}_9\text{H}_{19}\text{-}[^{13}\text{C}_6]\text{H}_4\text{-OH}$ , 1 ng/ $\mu\text{l}$ , used as an internal standard.

Weigh 10 mg of 4-*n*-nonylphenol(ring- $^{13}\text{C}_6$ ) in a 100 ml measuring flask and bring to volume with acetone (5.7). Dilute this solution with acetone (5.7) in the ratio 1:100.

Alternative internal standards (e.g. 4-*n*-nonylphenol) that meet the internal standard requirements are acceptable. An internal standard should have the following characteristics: It should be stable and should not interfere with the analyte. It should be as similar in structure to the analyte as possible, to ensure that it will show the same properties relative to, for example, adsorption on glass surfaces, extraction, concentration as the analyte. It should not be present in the sample matrix.

**5.9 4-nonylphenol solution**, 1 ng/ $\mu\text{l}$ , used as a calibration standard.

Weigh 10 mg of 4-nonylphenol,  $\text{C}_{15}\text{H}_{24}\text{O}$  (technical mixture of isomers), CAS No 84852-15-3, in a 100 ml measuring flask and bring to volume with toluene (5.5). Dilute this solution in the ratio 1:100 with toluene (5.5) or acetone (5.7) if a calibration over the total procedure (9.3, 9.4) is applied.

**5.10 4-(1,1,3,3-tetramethylbutyl)phenol solution**, 1 ng/μl, used as a calibration standard.

Weigh 10 mg of 4-(1,1,3,3-tetramethylbutyl)phenol, C<sub>14</sub>H<sub>22</sub>O, CAS No 140-66-9, in a 100 ml measuring flask and bring to volume with toluene (5.5). Dilute this solution in the ratio 1:100 with toluene (5.5) or acetone (5.7) if a calibration over the total procedure (9.3, 9.4) is applied.

Store solutions 5.8, 5.9 and 5.10 in the refrigerator protected from light. Check the solutions weekly prior to use.

NOTE The solutions 5.8, 5.9 and 5.10 are commercially available.

**5.11 Nitrogen**, N<sub>2</sub>, purity ≥ 99,996 %.

## 6 Apparatus

Clean all glassware by rinsing with acetone (5.7). Avoid detergents when using a labware-washing machine. Alternatively, prior to use, heat all glassware, except volumetric ware, to at least 250 °C for a minimum of 2 h.

The given volumes are chosen corresponding to the described extraction volume of 1 000 ml. Smaller volumes are possible.

Usual laboratory equipment, and the following.

**6.1 Shaking device.**

**6.2 Flat-bottomed glass bottles**, 1 000 ml, preferably of brown glass, with straight shoulders and a glass stopper or polytetrafluoroethene- (PTFE-) lined screw cap.

The sampling bottle shall allow direct extraction from the bottle.

**6.3 Quartz wool**, rinsed with hexane (5.4).

**6.4 Clean up column**, inner diameter 8 mm, length 120 mm, with glass or PTFE stopcocks.

**6.5 Quartz beaker**, 100 ml.

**6.6 Muffle furnace**, capable of maintaining a temperature of at least 600 °C.

**6.7 Evaporation device**, rotary evaporator, turbo-evaporator or vacuum concentration device.

**6.8 Separator**, for an example see Annex D, or another suitable device for phase separation.

**6.9 Drying column**, chromatographic column, length 600 mm, inner diameter 30 mm (or other convenient size), with coarse frit filter disk.

**6.10 Tapered flask**, 100 ml.

**6.11 Vials**, of brown glass, with a capacity of, for example 1,5 ml, compatible with the autosampler.

**6.12 Gas chromatograph**, temperature-programmable, with all required accessories, including gases, capillary columns, capillary injector and mass spectrometric detector.

The mass spectrometer should be capable of operating across the mass range of interest and incorporate a data system capable of quantifying ions using selected *m/z* values.