



SLOVENSKI STANDARD SIST EN ISO 15680:2004

01-februar-2004

Water quality - Gas-chromatographic determination of a number of monocyclic aromatic hydrocarbons, naphthalene and several chlorinated compounds using purge-and-trap and thermal desorption (ISO 15680:2003)

Wasserbeschaffenheit - Gaschromatische Bestimmung einer Anzahl monocyclischer aromatischer Kohlenwasserstoffe, Naphthalin und einiger chlorierter Substanzen mittels Purge und Trap und thermischer Desorption (ISO 15680:2003)

Qualité de l'eau - Dosage par chromatographie en phase gazeuse d'un certain nombre d'hydrocarbures aromatiques monocycliques, du naphthalene et de divers composés chlorés par dégazage, piégeage et désorption thermique (ISO 15680:2003)

STANDARD PREVIEW

(standards.itech.ai)

[SIST EN ISO 15680:2004](https://standards.itech.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-)

<https://standards.itech.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563->

Ta slovenski standard je istoveten z: EN ISO 15680:2003

Ta slovenski standard je istoveten z: EN ISO 15680:2003

ICS:

13.060.50 Ú!^ã\ ææç[å^Á ä^ { ã } ^ Examination of water for chemical substances

SIST EN ISO 15680:2004 en

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[SIST EN ISO 15680:2004](#)

<https://standards.iteh.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-ca3dc0c95d16/sist-en-iso-15680-2004>

EUROPEAN STANDARD

EN ISO 15680

NORME EUROPÉENNE

EUROPÄISCHE NORM

November 2003

ICS 13.060.50

English version

Water quality - Gas-chromatographic determination of a number of monocyclic aromatic hydrocarbons, naphthalene and several chlorinated compounds using purge-and-trap and thermal desorption (ISO 15680:2003)

Qualité de l'eau - Dosage par chromatographie en phase gazeuse d'un certain nombre d'hydrocarbures aromatiques monocycliques, du naphthalène et de divers composés chlorés par dégazage, piégeage et désorption thermique (ISO 15680:2003)

Wasserbeschaffenheit - Gaschromatische Bestimmung einer Anzahl monocyclischer aromatischer Kohlenwasserstoffe, Naphthalin und einiger chlorierter Substanzen mittels Purge und Trap und thermischer Desorption (ISO 15680:2003)

This European Standard was approved by CEN on 28 October 2003.

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 Management Centre 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 Management Centre has the same status as the official versions.

<https://standards.iteh.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-b0d56751f453/iso-15680-2003>

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.



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

Management Centre: rue de Stassart, 36 B-1050 Brussels

EN ISO 15680:2003 (E)

CORRECTED 2003-12-17

Foreword

This document (EN ISO 15680:2003) has been prepared by Technical Committee ISO/TC 147 "Water quality" in collaboration with 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 2004, and conflicting national standards shall be withdrawn at the latest by May 2004.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of ISO 15680:2003 has been approved by CEN as EN ISO 15680:2003 without any modifications.

(standards.iteh.ai)

NOTE Normative references to International Standards are listed in Annex ZA (normative).

[SIST EN ISO 15680:2004](https://standards.iteh.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-ca3dc0c95d16/sist-en-iso-15680-2004)

<https://standards.iteh.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-ca3dc0c95d16/sist-en-iso-15680-2004>

Annex ZA (normative)

Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

NOTE Where an International Publication has been modified by common modifications, indicated by (mod.), the relevant EN/HD applies.

<u>Publication</u>	<u>Year</u>	<u>Title</u>	<u>EN</u>	<u>Year</u>
ISO 3696	1987	Water for analytical laboratory use - Specification and test methods	EN ISO 3696	1995

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[SIST EN ISO 15680:2004](https://standards.iteh.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-ca3dc0c95d16/sist-en-iso-15680-2004)

<https://standards.iteh.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-ca3dc0c95d16/sist-en-iso-15680-2004>

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[SIST EN ISO 15680:2004](#)

<https://standards.iteh.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-ca3dc0c95d16/sist-en-iso-15680-2004>

INTERNATIONAL
STANDARD

ISO
15680

First edition
2003-11-01

**Water quality — Gas-chromatographic
determination of a number of monocyclic
aromatic hydrocarbons, naphthalene and
several chlorinated compounds using
purge-and-trap and thermal desorption**

iTeh STANDARD PREVIEW
(standards.iteh.ai)

*Qualité de l'eau — Dosage par chromatographie en phase gazeuse
d'un certain nombre d'hydrocarbures aromatiques monocycliques, du
naphthalène et de divers composés chlorés par dégazage, piégeage et
désorption thermique*

[SIST EN ISO 15680:2004](#)

[https://standards.iteh.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-
ca3dc0c95d16/sist-en-iso-15680-2004](https://standards.iteh.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-ca3dc0c95d16/sist-en-iso-15680-2004)



Reference number
ISO 15680:2003(E)

© ISO 2003

ISO 15680:2003(E)**PDF disclaimer**

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW (standards.iteh.ai)

[SIST EN ISO 15680:2004](https://standards.iteh.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-ca3dc0c95d16/sist-en-iso-15680-2004)

<https://standards.iteh.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-ca3dc0c95d16/sist-en-iso-15680-2004>

© ISO 2003

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword.....	iv
1 Scope.....	1
2 Normative references	1
3 Terms and definitions.....	2
4 Principle	2
5 Interferences.....	3
6 Reagents	4
7 Apparatus.....	7
8 Sample collection, preservation and preparation	8
9 Analytical procedure.....	9
10 Calibration.....	11
11 Calculation	12
12 Expression of results.....	12
13 Precision data.....	12
14 Test report.....	12
Annex A (informative) Application of purge-and-trap concentration to the GC analysis of volatile compounds in water — Example 1: Validation study in the UK.....	14
Annex B (informative) Application of purge-and-trap concentration to the GC-analysis of volatile compounds in water — Example 2: Data provided by DIN.....	19
Annex C (informative) Application of purge-and-trap concentration to the GC-analysis of volatile compounds in water — Example 3: Validation study in the Netherlands.....	22
Annex D (normative) Criteria for the GC-MS identification of target compounds.....	25
Annex E (informative) Procedures for the cleaning of glassware and the preparation of contaminant-free water.....	28
Annex F (informative) Preparation of standard solutions of volatile organic compounds	30
Annex G (informative) Determination of the (absolute) recovery of substances analysed by purge-and-trap concentration.....	32
Bibliography	33

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 15680 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

iTeh STANDARD PREVIEW (standards.iteh.ai)

[SIST EN ISO 15680:2004](https://standards.iteh.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-ca3dc0c95d16/sist-en-iso-15680-2004)

<https://standards.iteh.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-ca3dc0c95d16/sist-en-iso-15680-2004>

Water quality — Gas-chromatographic determination of a number of monocyclic aromatic hydrocarbons, naphthalene and several chlorinated compounds using purge-and-trap and thermal desorption

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.

1 Scope

This International Standard specifies a general method for the determination of volatile organic compounds (VOCs) in water by purge-and-trap isolation and gas chromatography (GC). Annexes A, B and C provide examples of analytes that can be determined using this International Standard. They range from difluorodichloromethane (R-12) up to trichlorobenzene, including all non-polar organic compounds of intermediate volatility.

Detection is preferably carried out by mass spectrometry in the electron impact mode (EI), but other detectors may be applied as well.

The limit of detection largely depends on the detector in use and the operational parameters. Typically detection limits as low as 10 ng/l¹⁾ can be achieved. The working range typically is up to 100 µg/l.

This International Standard is applicable to drinking water, ground water, surface water, seawater and to (diluted) waste water.

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 — Specifications and test methods*

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*

1) The value given is an indication of the limit of detection. It is calculated as 3 times the standard deviation of a series of measurements of 10 replicate samples under conditions of repeatability.

ISO 15680:2003(E)**3 Terms and definitions**

For the purposes of this document, the following terms and definitions apply.

3.1
volatile organic compound
VOC

organic compound, generally non-polar, with boiling point between approximately $-30\text{ }^{\circ}\text{C}$ and $220\text{ }^{\circ}\text{C}$

3.2
target compound

selected component whose presence or absence is determined

NOTE This definition can also apply to a derivative of the original compound which is formed during an intentional derivatization procedure.

3.3
standard compound

target compound with the highest possible purity that can be used as a reference during the analysis and free of impurities having any influence on its mass spectrum

3.4
retention-time standard

compound that is added to the sample (or to the sample extract) and to the **external standard solution** (3.6) and whose retention time is used to calculate the relative retention times of the target compounds

NOTE The retention-time standard may be identical to the internal standard(s).

3.5
relative retention time

ratio between the retention time of the target compound and the retention time of the retention-time standard

<https://standards.iteh.ai/catalog/standards/sist/9a3d3bf8-88a6-4670-b563-ca3dc0c95d16/sist-en-iso-15680-2004>

3.6
external standard solution

solution of a known concentration of the target compounds

3.7
lowest concentration for identification

lowest concentration of the target compound which, if present in the sample, still can be identified using the identification criterion that the selected diagnostic ion with the lowest intensity is still present in the mass spectrum with a signal-to-noise ratio higher than 3:1

NOTE This concentration strongly depends on the sensitivity of the instrument and on the performance characteristics of the analytical method.

3.8
diagnostic ion

ion selected from the mass spectrum of the target compound with the highest possible specificity

NOTE For the selection of diagnostic ions, see D.5.

4 Principle

A fixed volume of sample is purged with a fixed volume of an inert gas to strip out the volatile components which are subsequently trapped. This trapping can be either:

- a) on a packed adsorbent trap (preferably combined with or followed by a cryofocusing system), or
- b) directly on a capillary cold-trap.

After completion of the purge process, the trap is heated to desorb the volatile components which are swept by the GC carrier gas on to a capillary GC column. This transfer to the GC column can be done in an on-line or in an off-line set-up. To achieve narrow injection bandwidths, the use of a cryofocusing system is recommended when the trapping is done on a packed adsorbent trap as in a) or the transfer is done through an injector-splitter set at approximately 20:1 if the sensitivity of the analytical system allows this.

The components are separated by GC utilizing temperature programming, and are detected by the use of a mass spectrometer. Data are acquired in the full-scan mode or at a sufficient number of specific fragments to enable matching against those of the standards. A compound is regarded to be present when the criteria of Annex D are met. Quantification is carried out using selected characteristic fragments for each determinand.

5 Interferences

5.1 General

In principle, any purgeable compound which elutes at the same chromatographic retention time and produces a mass spectrum identical, or very similar, to any determinand under investigation will interfere. In practice, this is unlikely as the spectra of most of the determinands are characteristic. With retention-time data and the availability of the spectrum over a wide range of masses, the possibility of misidentification is quite small. Co-eluting peaks with ions with non-specific m/z values might cause interference, but quantification ions can be chosen to preclude this.

Contamination introduced during the analytical procedure is monitored by the determination of blanks (9.4).

5.2 Interferences in the sampling process

VOCs are amenable to evaporation or degassing during the sampling process, transportation, storage and preparation of the samples. This can result in measured concentrations which are too low. VOCs can also diffuse into the samples from the ambient air of the laboratory or from air in the refrigerator where samples are stored. This results in concentrations which are too high.

5.3 Interferences due to the purge gas and the GC gas

Insufficient purity of the purge gas or the GC carrier gas can cause interferences.

5.4 Interferences in the purge-and-trap process

One of the main sources of contamination during sample transportation is contaminated laboratory air in the purge vessel or sample container. Therefore, the laboratory should be free of solvents and concentrated standard solutions.

Laboratory clothing is also a potential source of contamination, particularly of highly volatile halogenated hydrocarbons.

To avoid interferences, all materials (tubing, seals, valves, etc.) should be made from stainless steel or glass. The use of plastics material should be avoided. All glassware directly in contact with the sample or purged compounds should be cleaned thoroughly (see Annex E). There is an especially high risk of entrainment after the measurement of highly polluted samples.

Purge vessels incorporating a glass frit are liable to cause cross-contamination (see also 7.1).

Purging of water samples containing surfactants can result in formation of foam which might be in direct contact with the adsorbent. If this occurs, the purge procedure shall be stopped immediately.