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

*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*



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.

© 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*.

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.

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

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.