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**Water quality — Determination  
of selected highly volatile organic  
compounds in water — Method  
using gas chromatography and mass  
spectrometry by static headspace  
technique (HS-GC-MS)**

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*Qualité de l'eau — Dosage de composés organiques hautement  
volatils sélectionnés dans l'eau — Méthode par chromatographie  
en phase gazeuse par la technique de l'espace de tête statique et  
spectrométrie de masse (HS-GC-MS)*

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html). (standards.iteh.ai)

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## Introduction

Various methods are available for the determination of highly volatile organic compounds in water. This document specifies a gas chromatographic method with mass spectrometric detection (GC-MS) for the determination of volatile organic compounds using the static headspace technique (HS).

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# Water quality — Determination of selected highly volatile organic compounds in water — Method using gas chromatography and mass spectrometry by static headspace technique (HS-GC-MS)

**WARNING** — Persons using this document should be familiar with normal laboratory practice. This document 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.

**IMPORTANT** — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

## 1 Scope

This document specifies a method for the determination of selected volatile organic compounds in water (see Table 1). This comprises among others volatile halogenated hydrocarbons as well as gasoline components (BTXE, TAME, MTBE and ETBE).

The method is applicable to the determination of volatile organic compounds (see Table 1) in drinking water, groundwater, surface water and treated waste water in mass concentrations >0,1 µg/l. The lower application range depends on the individual compound, the amount of the blank value and the matrix.

The applicability of the method to further volatile organic compounds not indicated in Table 1 is not excluded, but this is checked in individual cases.

**Table 1 — Volatile organic compounds determinable by this method**

Name (other name)	Molecular formula	CAS-RN <sup>a</sup>	EC-Number <sup>b</sup>	Molar mass g/mol
allyl chloride <sup>c</sup> (3-chloropropene)	C <sub>3</sub> H <sub>5</sub> Cl	107-05-1	203-457-6	76,53
benzene	C <sub>6</sub> H <sub>6</sub>	71-43-2	200-753-7	78,11
biphenyl	C <sub>12</sub> H <sub>10</sub>	92-52-4	202-163-5	154,21
bromodichloromethane	CHBrCl <sub>2</sub>	75-27-4	200-856-7	163,83
chlorobenzene	C <sub>6</sub> H <sub>5</sub> Cl	108-90-7	203-628-5	112,56
2-chloro-1,3-butadiene (chloroprene)	C <sub>4</sub> H <sub>5</sub> Cl	126-99-8	204-818-0	88,54
2-chlorotoluene	C <sub>7</sub> H <sub>7</sub> Cl	95-49-8	202-424-3	126,58
3-chlorotoluene	C <sub>7</sub> H <sub>7</sub> Cl	108-41-8	203-580-5	126,58
4-chlorotoluene	C <sub>7</sub> H <sub>7</sub> Cl	106-43-4	203-397-0	126,58
dibromochloromethane	CHBr <sub>2</sub> Cl	124-48-1	204-704-0	208,28
1,2-dibromoethane	C <sub>2</sub> H <sub>4</sub> Br <sub>2</sub>	106-93-4	203-444-5	187,86
1,2-dichlorobenzene	C <sub>6</sub> H <sub>4</sub> Cl <sub>2</sub>	95-50-1	202-425-9	147,00
1,3-dichlorobenzene	C <sub>6</sub> H <sub>4</sub> Cl <sub>2</sub>	541-73-1	208-792-1	147,00

<sup>a</sup> CAS-RN: Chemical Abstracts Service Registry Number.

<sup>b</sup> EC-Number: European Inventory of Existing Commercial Substances (EINECS) or European List of Notified Chemical Substances (ELINCS).

<sup>c</sup> Compounds do not have long-term stability.

<sup>d</sup> Compounds can coelute.

<sup>e</sup> Source: Hazardous Substance Data Base University Hamburg (Germany).

Table 1 (continued)

Name (other name)	Molecular formula	CAS-RN <sup>a</sup>	EC-Number <sup>b</sup>	Molar mass g/mol
1,4-dichlorobenzene	C <sub>6</sub> H <sub>4</sub> Cl <sub>2</sub>	106-46-7	203-400-5	147,00
dichlorodiisopropyl ether	C <sub>6</sub> H <sub>12</sub> Cl <sub>2</sub> O	108-60-1	203-598-3	171,06
1,1-dichloroethane	C <sub>2</sub> H <sub>4</sub> Cl <sub>2</sub>	75-34-3	200-863-5	98,96
1,2-dichloroethane	C <sub>2</sub> H <sub>4</sub> Cl <sub>2</sub>	107-06-2	203-458-1	98,96
1,1-dichloroethene	C <sub>2</sub> H <sub>2</sub> Cl <sub>2</sub>	75-35-4	200-864-0	96,94
<i>cis</i> -1,2-dichloroethene	C <sub>2</sub> H <sub>2</sub> Cl <sub>2</sub>	156-59-2	205-859-7	96,94
<i>trans</i> -1,2-dichloroethene	C <sub>2</sub> H <sub>2</sub> Cl <sub>2</sub>	156-60-5	205-860-2	96,94
dichloromethane	CH <sub>2</sub> Cl <sub>2</sub>	75-09-2	200-838-9	84,93
1,2-dichloropropane	C <sub>3</sub> H <sub>6</sub> Cl <sub>2</sub>	78-87-5	201-152-2	112,99
<i>cis</i> -1,3-dichloropropene	C <sub>3</sub> H <sub>4</sub> Cl <sub>2</sub>	10061-01-5	233-195-8	110,97
<i>trans</i> -1,3-dichloropropene	C <sub>3</sub> H <sub>4</sub> Cl <sub>2</sub>	10061-02-6	602-030-00-5 <sup>e</sup>	110,97
2,3-dichloropropene	C <sub>3</sub> H <sub>4</sub> Cl <sub>2</sub>	78-88-6	201-153-8	110,97
1,1-dimethylpropyl-methyl ether, <i>tert</i> -amyl methyl ether (TAME)	C <sub>6</sub> H <sub>14</sub> O	994-05-8	213-611-4	102,17
ethyl benzene	C <sub>8</sub> H <sub>10</sub>	100-41-4	202-849-4	106,17
ethyl <i>tert</i> -butyl ether (ETBE)	C <sub>6</sub> H <sub>14</sub> O	637-92-3	211-309-7	102,17
hexachlorobutadiene	C <sub>4</sub> Cl <sub>6</sub>	87-68-3	201-765-5	260,76
hexachloroethane	C <sub>2</sub> Cl <sub>6</sub>	67-72-1	200-666-4	236,74
isopropylbenzene (cumene)	C <sub>9</sub> H <sub>12</sub>	98-82-8	202-704-5	120,19
methyl <i>tert</i> -butyl ether (MTBE)	C <sub>5</sub> H <sub>12</sub> O	1634-04-4	216-653-1	88,15
naphthalene	C <sub>10</sub> H <sub>8</sub>	91-20-3	202-049-5	128,17
<i>n</i> -propylbenzene	C <sub>9</sub> H <sub>12</sub>	103-65-1	203-132-9	120,19
1,1,1,2-tetrachloroethane	C <sub>2</sub> H <sub>2</sub> Cl <sub>4</sub>	630-20-6	211-135-1	167,85
tetrachloroethene	C <sub>2</sub> Cl <sub>4</sub>	127-18-4	204-825-9	165,84
tetrachloromethane (carbon tetrachloride)	CCl <sub>4</sub>	56-23-5	200-262-8	153,82
toluene	C <sub>7</sub> H <sub>8</sub>	108-88-3	203-625-9	92,14
tribromomethane (bromoform)	CHBr <sub>3</sub>	75-25-2	200-854-6	252,73
1,2,3-trichlorobenzene	C <sub>6</sub> H <sub>3</sub> Cl <sub>3</sub>	87-61-6	201-757-1	181,45
1,2,4-trichlorobenzene	C <sub>6</sub> H <sub>3</sub> Cl <sub>3</sub>	120-82-1	204-428-0	181,45
1,3,5-trichlorobenzene	C <sub>6</sub> H <sub>3</sub> Cl <sub>3</sub>	108-70-3	203-608-6	181,45
1,1,1-trichloroethane	C <sub>2</sub> H <sub>3</sub> Cl <sub>3</sub>	71-55-6	200-756-3	133,40
1,1,2-trichloroethane	C <sub>2</sub> H <sub>3</sub> Cl <sub>3</sub>	79-00-5	201-166-9	133,40
trichloroethene	C <sub>2</sub> HCl <sub>3</sub>	79-01-6	201-167-4	131,39
trichloromethane (chloroform)	CHCl <sub>3</sub>	67-66-3	200-663-8	119,38
1,1,2-trichlorotrifluoroethane	C <sub>2</sub> Cl <sub>3</sub> F <sub>3</sub>	76-13-1	200-936-1	187,38
1,2,4-trimethylbenzene (pseudocumene)	C <sub>9</sub> H <sub>12</sub>	95-63-6	202-436-9	120,19

<sup>a</sup> CAS-RN: Chemical Abstracts Service Registry Number.

<sup>b</sup> EC-Number: European Inventory of Existing Commercial Substances (EINECS) or European List of Notified Chemical Substances (ELINCS).

<sup>c</sup> Compounds do not have long-term stability.

<sup>d</sup> Compounds can coelute.

<sup>e</sup> Source: Hazardous Substance Data Base University Hamburg (Germany).



Table 1 (continued)

Name (other name)	Molecular formula	CAS-RN <sup>a</sup>	EC-Number <sup>b</sup>	Molar mass g/mol
1,3,5-trimethylbenzene (mesitylene)	C <sub>9</sub> H <sub>12</sub>	108-67-8	203-604-4	120,19
vinyl benzene (styrene)	C <sub>8</sub> H <sub>8</sub>	100-42-5	202-851-5	104,15
vinyl chloride (chloroethene) <sup>c</sup>	C <sub>2</sub> H <sub>3</sub> Cl	75-01-4	200-831-0	62,49
<i>o</i> -xylene	C <sub>8</sub> H <sub>10</sub>	95-47-6	202-422-2	106,17
<i>m</i> -xylened	C <sub>8</sub> H <sub>10</sub>	108-38-3	203-576-3	106,17
<i>p</i> -xylened	C <sub>8</sub> H <sub>10</sub>	106-42-3	203-396-5	106,17
<sup>a</sup> CAS-RN: Chemical Abstracts Service Registry Number. <sup>b</sup> EC-Number: European Inventory of Existing Commercial Substances (EINECS) or European List of Notified Chemical Substances (ELINCS). <sup>c</sup> Compounds do not have long-term stability. <sup>d</sup> Compounds can coelute. <sup>e</sup> Source: Hazardous Substance Data Base University Hamburg (Germany).				

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 5667-4, *Water quality — Sampling — Part 4: Guidance on sampling from lakes, natural and man-made*

ISO 5667-5, *Water quality — Sampling — Part 5: Guidance on sampling of drinking water from treatment works and piped distribution systems*

ISO 5667-6, *Water quality — Sampling — Part 6: Guidance on sampling of rivers and streams*

ISO 5667-10, *Water quality — Sampling — Part 10: Guidance on sampling of waste waters*

ISO 5667-11, *Water quality — Sampling — Part 11: Guidance on sampling of groundwaters*

## 3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

## 4 Principle

An exact volume of an unfiltered water sample is sealed gastight in a headspace vial and heated. After an equilibrium has become established between the volatile organic compounds dissolved in the water and those located in the gas phase above the water level, an exact gas volume is taken from the gas phase and determined by gas chromatography with mass spectrometric detection.

## 5 Interferences

### 5.1 General

If a sample consists of several liquid phases, another method shall be applied.

### 5.2 Interferences in the laboratory

Some of the volatile organic compounds listed in [Table 1](#) are frequently used as solvents in laboratories. Solvent vapours in the laboratory air can lead to overestimates during the analysis. Regular blank value examinations are therefore indispensable (see [9.3.1](#)).

### 5.3 Interferences by the matrix

Matrix effects that lead to different recoveries and different response factors in samples in comparison to calibration standards can be reduced by adding specific amounts of salt. An increase in the sensitivity can also be attained by adding salt. The use of sodium sulfate or sodium chloride has proven effective. However, interferences can occur depending on the salt used.

### 5.4 Interferences in the headspace

Some compounds can decompose while the equilibrium forms at e.g. 80 °C. For example, 1,1,2,2-tetrachloroethane decomposes to trichloroethene. If 1,1,2,2-tetrachloroethane is present in the sample, overestimates of trichloroethene can result.

NOTE The decomposition of 1,1,2,2-tetrachloroethane can be eliminated by acidifying the water in the headspace vial with H<sub>2</sub>SO<sub>4</sub> to pH <2 and using Na<sub>2</sub>SO<sub>4</sub> as salt. But remember that the acid has an immense influence on the life span of the column and the injector.

### 5.5 Interferences during gas chromatography and mass spectrometry

To rectify interferences that are typically caused by the injection system or by inadequate separation, enlist experts and observe the manufacturer information in the apparatus manuals. The performance and stability of the analysis system shall be checked regularly (e.g. by measuring reference solutions of known composition).

Performance data from an interlaboratory trial held in 2013 are provided in [Annex D](#).

## 6 Reagents

### 6.1 General

Unless otherwise indicated, reagents to be used are of purity grade “for analysis” or “for residue analysis”.

**6.2 Water**, complying with the requirements of ISO 3696, grade 1 or equivalent without any interfering blank values.

**6.3 Operating gases for gas chromatograph and mass spectrometer**, of high purity and according to the required specification of the manufacturer of the instrumentation, e.g. helium, minimum purity 99,996 %.

**6.4 Salts**, e.g. sodium sulfate, Na<sub>2</sub>SO<sub>4</sub>, sodium chloride, NaCl.

**6.5 Solvents**, for the preparation of stock solutions and as solubilizers in aqueous reference solutions, e.g. methanol, CH<sub>3</sub>OH, or dimethylformamide (DMF), C<sub>3</sub>H<sub>7</sub>NO.

**6.6 Reference compounds**, every compound to be analysed shall be of defined purity.

**6.7 Internal standard**, for examples of suitable internal standards, see [Annex B](#).

## **6.8 Examples of stock and standard solutions**

### **6.8.1 Stock solution**

The stock solution shall be produced by corresponding dilution of the individual compounds in e.g. methanol ([6.5](#)) or from certified standard mixtures. The concentration of every individual compound is e.g. 100 µg/ml in methanol ([6.5](#)).

Keep the stock solutions at a temperature not exceeding 6 °C and protect them from light.

They are stable for at least 12 months.

### **6.8.2 Intermediate dilutions (spiking solutions)**

Intermediate dilutions (spiking solutions) for the preparation of aqueous multi-component solutions for calibration over the total process ([6.8.4](#)) are produced by dilution of the stock solution ([6.8.1](#)) with a solvent ([6.5](#)). For example, use a microlitre syringe ([7.7](#)) to add between 5 µl and 500 µl of each of the required stock solution ([6.8.1](#)) in a 10 ml volumetric flask ([7.9](#)) filled about half-full with methanol and then fill up to the mark.

The concentrations in methanol are then between 0,05 µg/ml and 5 µg/ml.

Keep the intermediate dilutions at a temperature not exceeding 6 °C and protect them from light.

They are stable for at least 6 months.

### **6.8.3 Solution of the internal standards**

Prepare the solution by corresponding dilutions of the internal standards ([6.7](#)) with methanol ([6.5](#)), each component e.g. 1 µg/ml in methanol.

### **6.8.4 Aqueous multi-component solution for the calibration**

The aqueous multi-component solutions (reference solutions) for the calibration can be produced as follows.

- Pour a defined volume of e.g. 10 ml water ([6.2](#)) in an e.g. 20 ml headspace vial ([7.8](#)) and add a defined volume of e.g. 10 µl of the relevant spiking solution ([6.8.2](#)) directly into the water.
- Then add a defined volume of e.g. 10 µl of the internal standard solution ([6.8.3](#)) directly into the water ([6.2](#)).
- Close the headspace vial ([7.8](#)) with the closing cap directly after the spiking and shake.

If salt is to be added, this is done before adding the water. The addition of salt ([6.4](#)) close to saturation (e.g. 3 g NaCl or 4 g Na<sub>2</sub>SO<sub>4</sub> in 10 ml, respectively) is recommended to minimize matrix effects.

Alternatively, the spiking solution ([6.8.2](#)) and internal standard solution ([6.8.3](#)) can also be added via the septum. This will not result in losses; nevertheless, the quality of the septa used should be ensured by blank samples.

It is recommended that the spiking volume be kept constant.

The aqueous multi-component solutions for the calibration can also be prepared in volumetric flasks ([7.9](#)). Ensure that no losses result due to pipetting and homogenization. Stir the solution for at least 10 s. Take care of a thorough mixing but avoid the formation of a turbulence funnel.

## 6.9 Sodium thiosulfate pentahydrate, $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ .

NOTE Ascorbic acid can also be used for the neutralizing of the chlorine as far as validated.

## 7 Apparatus

### 7.1 General

Equipment or glass parts that come into contact with the water sample shall be free of the compounds to be analysed and free of residues that can cause interfering blank values. This can be attained by cleaning the glass equipment.

**7.2 Sample bottles**, e.g. narrow-necked flat-bottomed bottles with glass stoppers, preferably brown glass bottles, nominal volume e.g. 250 ml or less.

**7.3 Dry cabinet.**

**7.4 Gas chromatograph (GC)**, with mass spectrometer (MS) and headspace (HS) sampler.

**7.5 Capillary columns**, with e.g. (medium-)polar stationary phase, e.g. inner diameter  $\leq 0,32$  mm, length about 30 m to 60 m, film thickness 1  $\mu\text{m}$  to 3  $\mu\text{m}$  (phase ratio  $> 300$ ).

**7.6 Magnetic stirring rod**, polytetrafluorethylene (PTFE)-encased.

**7.7 Microlitre syringes**, various nominal volumes e.g. 10  $\mu\text{l}$ , 100  $\mu\text{l}$ , 250  $\mu\text{l}$ , 1 000  $\mu\text{l}$ .

**7.8 Headspace vials (HS vial)**, e.g. 20 ml for automatic sampler.

**7.9 Volumetric flasks**, nominal volume e.g. 10 ml, 50 ml, 100 ml, e.g. volumetric flasks ISO 1042 - A10 - C.

**7.10 Pipettes.**

## 8 Sampling

Take and handle the samples, taking into account the specifications given in ISO 5667-3, ISO 5667-4, ISO 5667-5, ISO 5667-6, ISO 5667-10 and ISO 5667-11.

Completely fill the sample bottle (7.2) with the sample.

Ensure a laminar flow during the sampling. Turbulent flows can lead to losses of the substances to be analysed.

Add sodium thiosulfate pentahydrate (6.9) to water samples likely to contain chlorine, thus obtaining a concentration of approximately 80 mg/l to 100 mg/l.

Pre-rinsing of the sample bottle (7.2) with sample material is to be avoided, as this can possibly result in an increased concentration of suspended matter components and also the loss of stabilization reagents in the sample.

Glass equipment or equipment made from stainless steel or e.g. scoops made from glass shall be used. Plastic equipment is to be avoided as blank values and analyte losses due to adsorption effects can occur as a result of this.

It shall be ensured that no interfering compounds get into the sample and no losses occur in the compounds to be determined (see Clause 5).