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**Water quality — Determination of highly  
volatile halogenated hydrocarbons —  
Gas-chromatographic methods**

*Qualité de l'eau — Dosage des hydrocarbures halogénés hautement volatils —  
Méthodes par chromatographie en phase gazeuse*



## Contents

|                                                                               |          |
|-------------------------------------------------------------------------------|----------|
| <b>SECTION 1: General</b>                                                     | <b>1</b> |
| 1.1 Scope                                                                     | 1        |
| 1.2 Normative references                                                      | 2        |
| 1.3 Definition                                                                | 3        |
| <b>SECTION 2: Liquid/liquid extraction and analysis by gas chromatography</b> | <b>4</b> |
| 2.1 Principle                                                                 | 4        |
| 2.2 Interferences                                                             | 4        |
| 2.3 Reagents                                                                  | 4        |
| 2.4 Apparatus                                                                 | 6        |
| 2.5 Sampling and sample preparation                                           | 8        |
| 2.6 Procedure                                                                 | 8        |
| 2.7 Calibration                                                               | 11       |
| 2.8 Identification and evaluation                                             | 16       |
| 2.9 Expression of results                                                     | 18       |
| 2.10 Precision data                                                           | 19       |
| 2.11 Test report                                                              | 21       |

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Printed in Switzerland

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|---------------------------------------------------------------------------------------------------------|---------------|
| <b>SECTION 3: Static headspace method and analysis by gas chromatography</b>                            | <b>22</b>     |
| 3.1 Principle                                                                                           | 22            |
| 3.2 Interferences                                                                                       | 22            |
| 3.3 Reagents                                                                                            | 22            |
| 3.4 Apparatus                                                                                           | 22            |
| 3.5 Sampling                                                                                            | 23            |
| 3.6 Procedure                                                                                           | 24            |
| 3.7 Calibration                                                                                         | 26            |
| 3.8 Identification and evaluation                                                                       | 29            |
| 3.9 Expression of results                                                                               | 31            |
| 3.10 Precision data                                                                                     | 31            |
| 3.11 Test report                                                                                        | 33            |
| <br><b>Annex A (informative) Characteristics of highly volatile halogenated hydrocarbons</b>            | <br><b>34</b> |
| <b>Annex B (informative) Examples of gas chromatograms for highly volatile halogenated hydrocarbons</b> | <b>41</b>     |
| <b>Annex C (informative) Example of a microseparator</b>                                                | <b>45</b>     |
| <b>Annex D (informative) Sensitivity of electron-capture detector</b>                                   | <b>46</b>     |
| <b>Annex E (informative) Extraction recovery with pentane</b>                                           | <b>47</b>     |
| <b>Annex F (informative) Qualitative method for testing the quality of "penicillin type" stoppers</b>   | <b>48</b>     |
| <b>Annex G (informative) Collection of samples</b>                                                      | <b>49</b>     |

## 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.

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.

International Standard ISO 10301 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Annexes A to G of this International Standard are for information only.

## Introduction

Highly volatile halogenated hydrocarbons are used in industrial, commercial and domestic fields, and can enter a water body via waste water and may consequently contaminate drinking water. Furthermore, they can originate from the use of chlorine as an oxidizing agent in water and waste-water treatment. They also can be introduced by inappropriate handling. In addition, they can be formed by decomposition of higher molecular mass organohalogen derivatives.

In uncontaminated ground water and rain water, the concentrations of halogenated hydrocarbons are generally below 0,1 µg/l. In surface water they may be higher, depending on the origin and quality of the water. In untreated waste water the concentrations may reach saturation of the aqueous phase. In general, the solubility of these compounds in organic solvents and in fatty material exceeds their solubility in water.

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# Water quality — Determination of highly volatile halogenated hydrocarbons — Gas-chromatographic methods

## Section 1 : General

### 1.1 Scope

This International Standard specifies two methods for the determination of highly volatile halogenated hydrocarbons using gas chromatography.

Section 2 specifies a method for the determination by liquid/liquid extraction of highly volatile halogenated hydrocarbons in drinking water, ground water, swimming pool water, most rivers and lakes and many sewage and industrial effluents. Typical values of "quantification limits" are given in table 1.

**Table 1 — Typical values of "quantification limits" for some highly volatile halogenated hydrocarbons using liquid/liquid extraction**

| Compound                           | Quantification limits<br>µg/l |
|------------------------------------|-------------------------------|
| Dichloromethane                    | 50                            |
| Chloroform                         | 0,05 - 0,3                    |
| Carbon tetrachloride               | 0,01 - 0,1                    |
| 1,1-Dichloroethane                 | 1,0 - 5                       |
| 1,2-Dichloroethane                 | 5 - 10                        |
| 1,1,1-Trichloroethane              | 0,02 - 0,1                    |
| 1,1,2,2-Tetrachloroethane          | 0,05 - 0,1                    |
| Hexachloroethane                   | 0,01 - 0,05                   |
| <i>cis</i> -1,2-Dichloroethylene   | 5 - 50                        |
| <i>trans</i> -1,2-Dichloroethylene | 1 - 10                        |
| Trichloroethylene                  | 0,05 - 0,1                    |
| Tetrachloroethylene                | 0,1                           |
| Hexachlorobutadiene                | 0,01                          |
| Tribromomethane                    | 0,1                           |
| 1,1,2-Trichlorotrifluoroethane     | 0,1                           |

Section 3 specifies a method for the determination of highly volatile halogenated hydrocarbons in drinking water, surface waters and ground water by a static head-space method. Typical values of "quantification limits" are given in table 2.

In practise, the head-space method is applicable for industrial effluents as a screening method, but in some cases it is necessary to confirm the result by the liquid-liquid extraction method.

NOTE : When applying this International Standard, the guide on analytical quality control for water analysis (see ISO/TR 13530) should be followed, especially for the calibration steps.

**Table 2 — Typical values of "quantification limits" for some highly volatile halogenated hydrocarbons using static head-space method**

| Compound                                | Quantification limits<br>µg/l |
|-----------------------------------------|-------------------------------|
| Dichloromethane                         | 50                            |
| Chloroform                              | 0,3                           |
| Carbon tetrachloride                    | 0,1                           |
| 1,1-Dichloroethane                      | 100                           |
| 1,2-Dichloroethane                      | 100                           |
| 1,1,1-Trichloroethane                   | 0,1                           |
| 1,1,2-Trichloroethane                   | 20                            |
| 1,1-Dichloroethylene                    | 10                            |
| <i>cis</i> -1,2-Dichloroethylene        | 50                            |
| <i>trans</i> -1,2-Dichloroethylene      | 25                            |
| Trichloroethylene                       | 0,2                           |
| Tetrachloroethylene                     | 0,2                           |
| 1,2-Dichloropropane                     | 50                            |
| 1,3-Dichloropropane                     | 200                           |
| <i>cis+trans</i> -1,3-Dichloropropylene | 10                            |
| Dibromomethane                          | 0,3                           |
| Tribromomethane (Bromoform)             | 5                             |
| 1,2-Dibromoethane                       | 2                             |
| Bromochloromethane                      | 1                             |
| Bromodichloromethane                    | 0,2                           |
| Dibromochloromethane                    | 0,3                           |
| 1,1,3-Trifluoroethane                   | 1                             |

## 1.2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

|                              |                                                                                   |
|------------------------------|-----------------------------------------------------------------------------------|
| ISO 5667-1:1980,             | Water quality - Sampling - Part 1 : Guidance on the design of sampling programmes |
| ISO 5667-2:1991,             | Water quality - Sampling - Part 2 : Guidance on sampling techniques               |
| ISO/TR 13530:— <sup>1)</sup> | Water quality - Guide to analytical quality control for water analysis            |

<sup>1)</sup> In preparation.

### 1.3 Definition

For the purposes of this International Standard, the following definition applies :

**1.3.1 highly volatile halogenated hydrocarbons:** Fluorinated, chlorinated, brominated and/or iodinated mainly nonaromatic hydrocarbons composed of one to six atoms of carbon.

NOTE Their boiling points generally fall within the range of 20 °C to 220 °C at atmospheric pressure (see annex A).

## Section 2 : Liquid/liquid extraction and analysis by gas chromatography

### 2.1 Principle

The highly volatile halogenated hydrocarbons are extracted into an organic solvent. The solution is then analysed by gas chromatography with an electron-capture detector or any other suitable detector.

### 2.2 Interferences

Interferences can be due to the sampling procedure, vials and stoppers, solvents, gases, organic compounds in the laboratory atmosphere and contamination from the autosampler. Procedures for minimizing contamination are given in 2.5 and 2.6.

### 2.3 Reagents

All reagents shall be of sufficient purity so as to not give rise to significant interfering peaks in the gas chromatogram of the solvent extract. The purity of reagents shall be verified by a suitable procedure, for example, by blank determinations (see 2.6.4).

Reagents can become contaminated by contact with air and other materials, particularly plastics, or by degradation caused by the action of light. Store all reagents in the dark in tightly sealed all-glass containers or other suitable vessels.

#### 2.3.1 Water for the preparation of calibration solutions and blank

The quality of the water used shall be determined. For example, use the following procedure as a suitable preparation method :

Place water in a bottle with a conical shoulder, nominal capacity 2 litres, fitted with a ground-glass stopper, pretreated according to 2.4.2.

Determine the content of the highly volatile halogenated hydrocarbons in this water.

If the water is contaminated, purify as follows :

- position a glass delivery tube with a sintered glass distributor a few millimetres above the bottom of the bottle ;
- heat the water to approximately 60 °C ;
- pass a stream of clean nitrogen (approximately 150-200 ml/min) through the water for 1 h via the bubbler. Let the water cool to room temperature and stopper the bottle ;
- store the water in a glass bottle in the dark.

Subsequently verify again the absence of highly volatile halogenated hydrocarbons. If contamination is found, use a purge gas of another source and repeat the procedure.

### 2.3.2 Gases for gas chromatography

Nitrogen, ultrapure, volume concentration minimum 99,996 %, or argon-methane mixture, ultrapure. Other gases for gas chromatography shall be in accordance with the instrument manufacturer's instructions.

### 2.3.3 Extraction solvent (pentane) free from highly volatile chlorinated hydrocarbons

Analyse a sample of the extraction solvent by electron-capture gas chromatography to ensure that it does not contain material giving rise to interfering peaks in the chromatogram. If the compound of interest elutes in the same range as the extraction solvent, then use other solvents such as hexane, petroleum ether, heptane or xylene (for waste waters), providing that the validity of the result is maintained.

### 2.3.4 Sodium sulfate, anhydrous

Heat a portion of about 250 g to 300 g of  $\text{Na}_2\text{SO}_4$  at  $500\text{ }^\circ\text{C} \pm 20\text{ }^\circ\text{C}$  for  $4\text{ h} \pm 30\text{ min}$  ; cool to about  $200\text{ }^\circ\text{C}$  in a muffle furnace and then to ambient temperature in a desiccator containing magnesium perchlorate (2.3.6) or equivalent alternative.

### 2.3.5 Sodium thiosulfate

Prepare a sodium thiosulfate solution (30 g/l) by dissolving  $46\text{ g} \pm 0,2\text{ g}$  of sodium thiosulfate pentahydrate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) in  $1\text{ 000 ml} \pm 5\text{ ml}$  water (see 2.3.1).

NOTE : Alternatively, solid sodium thiosulfate may be used.

### 2.3.6 Magnesium perchlorate

### 2.3.7 Water-miscible solvent

NOTE : Methanol, acetone or dimethylformamide may be used.

### 2.3.8 Reference substances

Pure examples of the highly halogenated hydrocarbons to be determined are required.

Store these reference substances in areas separate from sample extracts and the solvent used for the extraction.

NOTE : For reference substances which are gaseous at ambient temperature, it is recommended to use commercially available solutions.

### 2.3.9 Standard stock solutions

Prepare standard stock solutions by adding with a microlitre syringe defined quantities of each reference substance (see 2.3.8) under the surface of a suitable solvent.

NOTE : Suitable solvents for the preparation of standard stock solutions are acetone, pentane, hexane, dimethylbenzene or isooctane.

The containers containing the solutions shall be marked or weighed so that any evaporation losses of the solvent may be recognized. The solutions shall be stored in volumetric flasks with ground glass stoppers at a temperature of 4 °C in the dark. Prior to use, they shall be brought to ambient temperature and the level of solvent shall be adjusted, if necessary.

NOTE 1: A convenient concentration of standard stock solution is obtained by weighing 50 mg of the reference substance and dissolving it in 100 ml of the solvent. The solution is stable for about 1 year.

NOTE 2: For practical reasons, it is recommended to use mixed standard stock solutions.

### 2.3.10 Intermediate standard solutions

Prepare intermediate standard solutions by a suitable dilution of the stock solution (see 2.3.9) with the extraction solvent (see 2.3.3).

A typical value is 10 µg/ml.

Store the intermediate standard solutions at about 4 °C in the dark. These solutions are stable for 6 months.

### 2.3.11 Working standard solutions

Prepare at least five different concentrations by suitable dilutions of the intermediate standard solutions (see 2.3.10) with the extraction solvent (see 2.3.3).

Suitable concentrations are in the ng/ml range. Store these solutions at about 4 °C in the dark. These solutions are stable for at least 1 month.

## 2.4 Apparatus

**2.4.1 Gas chromatograph** fitted with an electron-capture detector or any other suitable detector and with suitable columns.

Separation of the highly volatile halogenated hydrocarbons requires high separation power. The best separations are obtained with capillary columns.

Different reference columns (specific for volatiles compounds) are available from the chromatographic equipment suppliers. The choice depends on the variety of substances to be analysed, the sampling technique, gas chromatograph configuration, etc.

As a guide, the following rules may be applied to the gas chromatograph column:

- a) nonpolar [(poly(dimethylsiloxane))] or semipolar [(poly(5 %-diphenyl-95 % dimethylsiloxane))] bonded phases ;
- b) as the relation between internal diameter and film thickness is a critical parameter, choose a phase ratio around 80 - 100 (suitable for volatile, low molecular mass compounds) ;
- c) length : generally, more than 30 m.

Annex B gives several examples of separation.

NOTE : Nonvolatile compounds, contained for example in waste water, may shorten the lifetime of the gas chromatographic column.

#### 2.4.2 Ordinary laboratory glassware

As an example of preparation, glassware to be used may be washed with detergent, rinsed with deionized water and finally with the extraction solvent or heated in an oven at 150 °C for at least 1 h and cooled at room temperature before use.

In order to minimize contamination during transport or storage, close the vessels and protect the neck of the bottle, for example with aluminium foil.

All syringes shall be thoroughly cleaned and their cleanliness verified by gas chromatography before use.

#### 2.4.3 Glass bottles, capacity about 250 ml, sealed with a solid glass stopper.

Prior to use, place the bottles upside down in a ventilated drying oven along with the stoppers and heat them for at least 1 h at 150 °C.

#### 2.4.4 Glass vials of capacity approximately 30 ml to 40 ml, with polytetrafluoroethylene (PTFE) coated septum.

#### 2.4.5 Magnetic stirrer or mechanical shaker

#### 2.4.6 Magnetic stirrer rods (length approximately 4 cm), coated with PTFE. Store the magnetic stirrer rods separately for each concentration range.

**2.4.7 Microseparator** (see as an example annex C, figure C.1).

**2.4.8 Glass wool washed with extraction solvent.**

**2.4.9 Bottles** with PTFE-coated septum, capacity approximately 2 ml, to store the extract.

## **2.5 Sampling and sample preparation**

Take samples according to ISO 5667-1 and ISO 5667-2.

Collect and store the water samples in bottles (see 2.4.3) cleaned as described in 2.4.2.

In the special case of septum-vial extraction (see 2.6.2), fill glass vials (see 2.4.4).

Collect the sample normally by immersion, by filling the bottle or the vial completely, discarding this water, refilling and stoppering so as to leave no headspace.

Loss of volatile compounds through degassing of the sample shall be avoided. Slowly fill the bottle at the sampling point until it overflows, avoiding turbulence.

The use of plastics tubing when taking samples shall be kept to a minimum in order to avoid losses or contamination of the sample.

If reaction between free halogens and organic matter in the sample, to produce trihalogenated methanes, is to be eliminated, add an excess of sodium thiosulfate (see 2.3.5) to the sampling bottle or vial after rinsing the bottle or the vial but prior to sampling.

NOTE : The quantity of sodium thiosulfate added to the sample is not critical but should be sufficient to react with all the chlorine present. Normally 0,1 ml to 0,2 ml of a 30 g/l solution (see 2.3.5) or a few crystals (3 mg to 5 mg) of solid sodium thiosulfate will be appropriate for a sample volume of about 250 ml.

If an internal standard is needed, it shall be added as soon as possible after sampling.

Avoid warming the sample during the transport.

If storage is unavoidable, cool the samples to about 4 °C and carry out the extraction within 48 h, if possible, since the extracts are much more stable than the water samples.

NOTE : If composite samples are being analysed, losses of highly volatile chlorinated hydrocarbons may occur when individual samples are mixed. Therefore, individual samples should be extracted separately and the solvent extracts combined before analysis.

## **2.6 Procedure**

Use the extraction procedures described in 2.6.1 or 2.6.2. If in the laboratory, contamination of the sample by air cannot be excluded, use septum-vial extraction (see 2.6.2).

### 2.6.1 Extraction

Take the full sample bottle (see 2.5) and discard enough water so that there is a residual sample volume of  $200 \text{ ml} \pm 10 \text{ ml}$ . Weigh the bottle and sample in order to accurately determine the sample volume. Add the extraction solvent (see 2.3.3), close and mix vigorously with the sample using a magnetic stirrer or a mechanical shaker for 5 min (see 2.4.5), to ensure that the extractant is finely dispersed in the sample in order to obtain a reproducible recovery.

NOTE : The volume of solvent used depends upon the type of sample ; suitable volumes are 10 ml for drinking water and 50 ml for waste water.

After mixing, allow the sample container to stand until the layers separate. Draw off directly the upper solvent layer using a pipette or, if the phases are not well separated, a microseparator (see 2.4.7) as follows :

Insert a glass wool plug (about 2 cm) through the central tube of the microseparator until it reaches the wider part. Rinse the tube and the plug with extraction solvent and dry both. Place the microseparator on top of the bottle containing the extracted sample and slowly add through the side tube sufficient water (see 2.3.1) so that the extract rises in the central tube past the glass wool.

It is not necessary to filter all of the organic phase through glass wool to obtain sufficient liquid for analysis.

If this treatment is not successful, separate the phases by centrifugation of a few millilitres of the turbid organic phase in a closed glass vessel (such as a centrifuge tube with a screw cap) or by freezing.

**Do not concentrate the sample extract by evaporation.**

Immediately proceed to gas chromatographic analysis (see 2.6.3). If analysis cannot be carried out immediately, store the solvent extract in an airtight stoppered bottle (see 2.4.9) at a temperature of about  $4^\circ\text{C}$ , no longer than one month.-

NOTE : For higher-boiling extraction solvents, such as hexane or 1,2-dimethylbenzene (*o*-xylene), cooling is not necessary; 1,2-dimethylbenzene extracts should be dried with anhydrous sodium sulfate (see 2.3.4) or magnesium perchlorate (see 2.3.6) (approximately 10 mg/ml) prior to gas chromatographic analysis.

The extraction shall be carried out in an environment as free as possible from volatile halogenated compounds. Often, interfering peaks will occur, especially at the beginning of the chromatogram. Contamination of the sample may be caused by sprays being used, solvents in the laboratory or refrigerants coming from chillers. Blank determinations (see 2.6.4) are necessary.

### 2.6.2 Septum-vial extraction

NOTE : Owing to the absence of a headspace, mixing with this technique is less efficient than using a bottle as in 2.6.1. Recoveries may be a little lower but are acceptable and reproducible.

Take the vial (see 2.4.4) filled according to 2.5 and insert a hypodermic syringe needle, through the septum, a distance of approximately 1 cm into the sample. Fill a 5 ml syringe with extraction solvent (see 2.3.3) and adjust the volume in the syringe to 2,5 ml, taking care to exclude any air bubbles. Insert the syringe needle, with syringe containing solvent now attached, through the septum, as far as possible into the vial. Invert the syringe plus vial (vial now above syringe) and inject the 2,5 ml of extraction solvent (see 2.3.3) into the vial. 2,5 ml of sample will be displaced via the open syringe needle. Withdraw both needles and shake the vial vigorously for 5 min.

Allow the layers to separate.

Then proceed to gas chromatographic analysis (see 2.6.3) or keep the extract with the water present for analysis. These extracts have proved stable for one month when stored in the vials, in the dark, at a temperature of about 4 °C.

NOTE : This method relies on the volume of the vials being reproducible. Nevertheless each vial should be checked and its volume noted for use in recovery checks.

### 2.6.3 Gas chromatography

Set up the gas chromatograph (see 2.4.1), fitted with an electron-capture detector or any other suitable detector and equipped with a suitable column, according to the manufacturer's instructions.

Normally, better resolution will be obtained with capillary columns.

The electron-capture detector is sensitive to halogenated hydrocarbons, but is not specific in its response. Its sensitivity to different halogenated hydrocarbons varies considerably (see annex D, table D.1) and the linearity of response to the compounds of interest and the useable working range of concentrations shall be determined by investigation using a series of calibration standard solutions of known concentrations.

Eliminate any contamination of the electron-capture detector which may cause a high or unsteady baseline according to the instructions of the manufacturer. Such contamination may be caused by contaminated gas (especially oxygen contamination) or a dirty detector due to a high column bleeding rate.

Should it be found necessary to clean the detector, the detector response and linearity shall be reconfirmed after cleaning and before further samples are analysed.

Inject an aliquot of the clear extract (see 2.6.1) into the injection port of the gas chromatograph or, in case of septum-vial extraction (see 2.6.2), withdraw aliquots of the solvent layer for gas chromatographic analysis through the septum using an appropriate microsyringe.

Compare the gas chromatogram obtained to those of the standard solutions (see 2.7).

Evaluate the gas chromatogram qualitatively and quantitatively (see 2.8). The requirements concerning the measurements, the calibration, evaluation and calculation techniques to be used are described in 2.7 and 2.8.

#### 2.6.4 Blank determination

Prior to analysis and at regular intervals, carry out full blank determinations using water (see 2.3.1).

Take the blank through the complete analytical procedure, starting with sampling and including all steps until the evaluation of the gas chromatogram.

If the blank value is too high (> 10 % of the measured value for any of the compounds of interest), find the cause through step-by-step examination of the procedure. Decrease the blank value through suitable measures (for example, extraction of the "clean water" with the extraction solvent prior to analysis, elimination of contamination in the room air, checking of the gas chromatograph and integration parameters).

In the case of high blank values caused by laboratory air contamination, follow the method given in 2.6.2.

#### 2.7 Calibration

Initially, determine the recovery ; obtain this recovery with the two following calibration steps :

a) calibration by direct injection of solvent standard solutions (see 2.7.1).

This gives information on the linear working range of the detector, retention times and relative responses of the determinands.

b) calibration of the overall procedure (see 2.7.2) using spiked water samples, and extraction.

The data obtained from 2.7 a) are compared with those from 2.7 b) in order to calculate the recovery (see 2.7.3) of each determinand.

Carry out the daily recalibration (see 2.7.4) with solvent standard solutions according to a) or with spiked water extracts according to b).

The use of an internal standard is recommended (see 3.7.3.2) ; it allows some correction of differing extraction recovery and errors in the injected volume, for example :

- 1-bromo-2-dichloroethane ;
- 1,2-dibromoethane ;
- *trans*-1,2-dichloroethylene ;
- bromotrichloromethane ;
- 1,2-dibromo-1,1-dichloroethane.