INTERNATIONAL STANDARD

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Water quality — Determination of dalapon, trichloroacetic acid and selected haloacetic acids — Method using gas chromatography (GC-ECD and/or GC-MS detection) after liquid-liquid extraction and derivatization

Qualité de l'eau — Dosage du dalapon, de l'acide trichloroacétique et d'acides haloacétiques sélectionnés — Méthode par chromatographie en phase gazeuse (détection CG-DCE et/ou CG-SM) après extraction liquide-liquide et dérivatisation

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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.

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 23631 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Introduction

The user should be aware the particular problems could require the specifications of additional marginal conditions.

Water quality — Determination of dalapon, trichloroacetic acid and selected haloacetic acids — Method using gas chromatography (GC-ECD and/or GC-MS detection) after liquid-liquid extraction and derivatization

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.

Diazomethane is explosive, extremely toxic and severely irritating, causing pulmonary oedema when inhaled in high concentrations. Long-term, low-level exposure may lead to sensitization, resulting in asthma-like symptoms. Also, diazomethane and several of its chemical precursors have been cited as carcinogens.

IMPORTANT — It is absolutely essential that tests conducted according to this International Standard be carried out by suitably trained staff.

1 Scope

This International Standard specifies a method for the determination of dalapon, trichloroacetic acid (TCA) and selected haloacetic acids (see Table 1) in ground water and drinking water by gas chromatography (GC-ECD and/or GC-MS detection) after liquid-liquid-extraction and derivatization using diazomethane. Depending on the matrix, the method is applicable to a concentration range from 0,5 µg/l to 10 µg/l. The validated reporting limit of TCA and dalapon is about 0,05 µg/l (see Table C.1). Detection by electron-capture detector (ECD) in general leads to lower detection limits. Detection by mass spectrometry (MS) allows analyte identification.

This method may be applicable as well to compounds not mentioned in Table 1 or to other types of water. However, it is necessary to verify the applicability of this method for these special cases.

Table 1 — Haloacetic acids determined by this method

Name	Molecular formula	Relative molecular mass	CAS registry No.
Bromochloroacetic acid	$C_2H_2BrClO_2$	173,4	5589-96-8
Dalapon [a]	$C_3H_4Cl_2O_2$	143,0	75-99-0
Dibromoacetic acid	$C_2H_2Br_2O_2$	217,8	631-64-1
Dichloroacetic acid	$C_2H_2Cl_2O_2$	128,9	79-43-6
Monobromoacetic acid	$C_2H_3BrO_2$	138,9	79-08-3
Monochloroacetic acid	$C_2H_3ClO_2$	94,5	79-11-8
Trichloroacetic acid (TCA)	$C_2HCl_3O_2$	163,4	76-03-9

[a] 2,2-Dichloropropionic acid.

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 3696:1987, *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

Dalapon, trichloroacetic acid (TCA) and selected haloacetic acids are extracted from the acidified water sample with methyl-*tert*-butyl ether (MTBE). The extract is concentrated by evaporation.

The analytes are methylated using diazomethane.

The methylated analytes are separated, identified and quantified by means of capillary gas chromatography with electron-capture detection (GC-ECD) and/or mass spectrometry (GC-MS).

4 Interferences

4.1 Interferences with the extraction procedure

Suspended particles in the water may interfere with the liquid-liquid-extraction procedure causing problems in layer separation. In this case, filter the water sample through a glass fibre filter (6.15) prior to enrichment.

4.2 Interferences with the gas chromatography and mass spectrometry procedure

Interferences may be caused e.g. by the injection system used or by inadequate separation of the analytes. Experienced operators, using the information given in the instrument manuals, may be able to minimize this type of interference. Regular checking of the chromatographic and spectrometric system is required to maintain adequate performance. Required system stability should be checked regularly by the use of a GC-standard.

Insufficiently purified solvents (5.6) as well as insufficiently purified sodium chloride (5.10) may cause severe interferences. Reagents used in the method to perform derivatization may lead to interferences in the ECD-chromatograms. Therefore, it is highly recommended that temperatures of the diazomethane building process be carefully kept in limits (see 5.19).

5 Reagents

Use solvents and reagents of sufficient purity, i.e. with negligibly low impurities compared with the concentration of analytes to be determined. As reagents use, as far as available, “residual grade” or better in order to obtain clean blanks. Check blanks regularly and establish proper charge control.

5.1 Water, complying to grade 1 as defined in ISO 3696:1987, or equivalent.

