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Water quality — Determination of adsorbable organically bound fluorine, chlorine, bromine and iodine (AOF, AOCl, AOBr, AOI) — Method using combustion and subsequent ion chromatographic measurement

Qualité de l'eau — Dosage des composés organiques adsorbables contenant du fluor, du chlore, du brome et de l'iode (AOF, AOCl, AOBr, AOI) — Méthode de combustion suivie d'un mesurage par chromatographie ionique

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Foreword

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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 document 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).

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Introduction

Adsorbable organically bound fluorine, chlorine, bromine and iodine are analytical convention parameters used to monitor water quality. They represent the sum of organically bound fluorine, chlorine, bromine and iodine that can be adsorbed on activated carbon under specified conditions and, if the sample has not been filtered, can also be attached to or contained in suspended substances.

In contrast to the adsorbable organically bound halogen (AOX) method according to ISO 9562, this method can be applied to determine the sum of organofluorine compounds in addition to the determination of the organically bound chlorine, bromine and iodine and detected halogen-specific separately.

The method is carried out by combustion ion chromatography (CIC).

Procedures for each separate parameter are described in Annex A, Annex B, Annex C and Annex D.

Alternatively, the adsorption of the organic substances contained in the water sample on activated carbon can also be carried out by the shaking method (see Annex E).

Samples with a high content of suspended solids can be analysed using the shaking method (see Annex E).

Samples with a high content of inorganic halides can be analysed using the solid phase extraction (SPE) method (see Annex F).

Results for samples analysed according to Annex E (shaking procedure) or Annex F (SPE procedure) can differ significantly from those of the method specified in the main part.

With some waters, interference can occur that cannot be eliminated. These waters cannot be measured with the method.

The AOCl, AOBr and AOI results according to Annex B, Annex C and Annex D can also be reported as adsorbable organically bound halogens determined by combustion ion chromatography (CIC-AOX) (see Annex J).

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Water quality — Determination of adsorbable organically bound fluorine, chlorine, bromine and iodine (AOF, AOCl, AOBr, AOI) — Method using combustion and subsequent ion chromatographic measurement

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 organically bound halogens fluorine, chlorine, bromine and iodine which are adsorbable on activated carbon. Adsorption takes place on activated carbon packed in columns.

The method is applicable for the determination of:

— ≥ 2 µg/l AOF, expressed as F;

— ≥ 10 µg/l AOCl, expressed as Cl;

— ≥ 1 µg/l AOBr, expressed as Br;

— ≥ 1 µg/l AOI, expressed as I.

The method is applicable for the determination of adsorbable organically bound fluorine, chlorine, bromine and iodine in water, e.g. in groundwater, surface water, bank filtrate, drinking water, aqueous eluates, cooling water and wastewater.

The working range is limited by the capacity of the activated carbon, the process blank and the capacity of the chromatographic separation column. Sample dilution into the working range can be required.

The range of application can be extended to lower concentrations with lower process blanks e.g. using low blank activated carbons.

The method can also be applied for samples containing suspended solids. Halogens adsorbed on the suspended solids (e.g. undissolved halides) are also determined. Filtration of the sample prior to analysis using a membrane filter (0,45 µm) allows the separate determination of dissolved adsorbable and particulate bound fractions of organically bound fluorine, chlorine, bromine or iodine.

Results from an international interlaboratory trial are presented in Annex K.

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 8466-1, *Water quality — Calibration and evaluation of analytical methods — Part 1: Linear calibration function*

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ISO 8466-2, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions*

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
adsorbable organically bound fluorine
AOF
equivalent mass of fluorine in organic halogen compounds, expressed as fluorine, measured under the conditions of this procedure

3.2
adsorbable organically bound chlorine
AOCl
equivalent mass of chlorine in organic halogen compounds, expressed as chlorine, measured under the conditions of this procedure

3.3
adsorbable organically bound bromine
AOBr
equivalent mass of bromine in organic halogen compounds, expressed as bromine, measured under the conditions of this procedure

3.4
adsorbable organically bound iodine
AOI
equivalent mass of iodine in organic halogen compounds, expressed as iodine, measured under the conditions of this procedure

3.5
adsorbable organically bound halogens
AOX
equivalent mass of the halogens chlorine, bromine and iodine in organic compounds, determined according to ISO 9562 and expressed as chloride

3.6
test sample
sample obtained from the original sample after preparation and dilution, if necessary, and fed into the adsorption process

3.7
combustion ion chromatography
CIC
technique comprising oxidative high-temperature combustion followed by absorption of formed hydrogen halides and subsequent ion chromatographic detection of the halide ions

3.8
adsorbable organically bound halogens, determined by combustion ion chromatography
CIC-AOX
equivalent mass of the halogens chlorine, bromine and iodine in organic compounds, measured under the conditions of this procedure and expressed as chlorine

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4 Interferences

4.1 Interferences during adsorption

AOF, AOCl, AOBr or AOI values can arise from the presence of inorganic halides in the sample. If the test sample contains as well as organohalogens in low concentration and inorganic halides in high concentration, the halides can add significant contribution to the result. These contributions can be dependent on the matrix and halogenide concentration. It is not possible to specify any information on the concentrations of inorganic halides at which significant contributions to the result for the AOF, AOCl, AOBr or AOI can be expected. These can depend on various factors, such as the properties e.g. characteristics of the activated carbon or contaminated laboratory air. Annex A, Annex B, Annex C and Annex D give examples of halide concentrations that give significant contributions to the result for the AOF, AOCl, AOBr or AOI.

Sample matrix characteristics [e.g. high concentrations of dissolved organic carbon (DOC)] can cause interference with the adsorption of the organically bound halogens and result in negative bias. These effects can be solved by sample dilution or spiking experiments, if necessary. Information on whether the DOC content of the sample influences the adsorption of AOF, AOCl, AOBr or AOI from the sample can be provided by current measurements or previous testing of the sampling point. For unfiltered samples, the total organic carbon (TOC) can also be used as information.

Samples containing living cells (e.g. microorganisms or algae) can cause a positive bias due to their halide content on AOCl, AOBr and AOI (chloride, bromide and iodide). In this case, the acidified sample should not be analysed until at least 8 h after sampling.

Particulate inorganic halogen compounds with a melting point < 1 000 °C can cause a positive bias. This can be avoided by filtering the sample.

High contents of undissolved substances can cause interference with the column method. In these cases, the use of activated carbon according to 9.4 or the shaking procedure (see Annex E) should be considered.

The recovery of some polar and hydrophilic compounds (e.g. trifluoroacetate or monochloroacetate) or volatile compounds is incomplete.

Contamination of the laboratory air due to chemicals or other sources can cause positive bias.

4.2 Interferences during combustion

Alkali metals present can cause premature devitrification of a quartz glass combustion tube.

NOTE The use of a ceramic inner tube can increase the lifetime of the combustion tube significantly.

A deficiency or insufficient quantity of water added for hydropyrolysis during AOF determination can lead to a negative bias.

4.3 Interferences during ion chromatography

Any substance that generates a detector signal similar to that of the analyte ion can cause interference. Additionally, a high concentration of ions can influence the peak resolution and the retention time of the analyte. Gradient elution can minimize many of these interferences.

5 Principle

To determine the organically bound halogens, the substances contained in the water sample are adsorbed onto activated carbon. Enriched halides are displaced from the activated carbon by washing with a nitrate wash solution. The loaded activated carbon is combusted in an oxygen stream. The resulting hydrogen halides are sorbed in an absorption solution. The halide ions are then determined by ion chromatography. The basic requirements for the procedure are:

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— Samples for the determination of AOF are treated differently from samples for the determination of AOCl, AOBr and AOI.

— Samples for the determination of AOF are not acidified. The adsorption takes place under unchanged pH conditions. Washing is performed with a neutral washing solution.

— Samples for the determination of AOCl, AOBr and AOI are adjusted to a pH < 2 with nitric acid. The adsorption and washing step are carried out in a nitric acid environment.

— The aqueous sample is passed through columns of activated carbon which adsorbs the organically bound halogens.

— The inorganic halides are removed by washing with a nitrate solution.

— The activated carbon is combusted in an oxygen stream at 950 °C or higher. For the determination of AOF, the loaded activated carbon is combusted with addition of water (hydropyrolysis).

— The resulting change in the volume in the absorption solution is taken into account when calculating the concentration (e.g. by determining the recovery of an internal standard).

— The hydropyrolytic combustion does not interfere with the determination of AOCl, AOBr and AOI.

— The resulting hydrogen halide ions from the organically bound halogens are collected in an absorption solution.

The absorption solution is injected into an ion chromatograph with a suppressed conductivity detector where anions are separated and quantified. The ion chromatographic separation is carried out on an anion exchange resin as stationary phase. Aqueous solutions of salts of weak monobasic and dibasic acids are used as eluents for isocratic or gradient elution. Quantification is carried out by means of conductivity detection (CD). The conductivity of the eluents is reduced by a suppressor device (cation exchanger). UV or tandem UV detection may be used.

NOTE A UV detector is not required to perform this analysis, but it can be used for bromide and iodide if higher sensitivity is required or in the case of matrix interference with the CD. UV detection (UVD) can be used in combination with the CD. The UV detector measures the absorption of the irradiated UV radiation. The measuring wavelength is at λ = (215 ± 10) nm for bromide and at λ = (226 ± 10) nm for iodide.

The AOCl, AOBr and AOI results according to Annex B, Annex C and Annex D can also be reported as adsorbable organically bound halogens, determined by combustion ion chromatography CIC-AOX (CIC-$AOX_{(Cl)}$).

The calculation shall be as described in Annex J.

6 Reagents

6.1 General

Use only reagents with the purity grade “for analyses”, if available. Unless otherwise stated, reagents shall be weighed to an accuracy of ±1 % of nominal mass. Commercially available solutions of equivalent quality may be used. If necessary, alternative volumes and concentrations to the solutions described in this clause may be prepared and used.

All reagents shall be checked for their contribution to the blank, if applicable.

6.2 Water, with a specific electrical resistance of ≥18 MΩ·cm (at 25 °C).

6.3 Reagents for sample stabilization and pre-treatment.

6.3.1 Nitric acid, $w(HNO_3)$ = 65 %.

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