



SLOVENSKI STANDARD
oSIST prEN ISO 18127:2024
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Kakovost vode - Določanje organsko vezanega fluora, klora, broma in joda, sposobnega adsorpcije (AOF, AOCl, AOBr, AOI) - Metoda z zgorevanjem in ionsko kromatografijo (ISO/DIS 18127:2024)

Water quality - Determination of adsorbable organically bound fluorine, chlorine, bromine and iodine (AOF, AOCl, AOBr, AOI) - Method using combustion and subsequent ion chromatographic measurement (ISO/DIS 18127:2024)

Wasserbeschaffenheit - Bestimmung von adsorbierbarem organisch gebundenem Fluor, Chlor, Brom und Iod (AOF, AOCl, AOBr, AOI) - Verfahrens mittels Verbrennung und nachfolgender Ionenchromatographischer Messung (ISO/DIS 18127:2024)

Qualité de l'eau - Dosage du fluor, du chlore, du brome et de l'iode adsorbables liés organiquement (AOF, AOCl, AOBr, AOI) - Méthode en utilisant la combustion et la mesure ultérieure par chromatographie ionique (ISO/DIS 18127:2024)

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13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances
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ISO/DIS 18127

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

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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 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).

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For an explanation of 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 www.iso.org/iso/foreword.html.

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

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ISO/DIS 18127:2024(en)**Introduction**

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

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

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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 $\mu\text{g/l}$ AOF, expressed as F;
- ≥ 10 $\mu\text{g/l}$ AOCl, expressed as Cl;
- ≥ 1 $\mu\text{g/l}$ AOBr, expressed as Br;
- ≥ 1 $\mu\text{g/l}$ AOI, expressed as I.

The method is applicable for the determination of adsorbable organically bound fluorine, chlorine, bromine or 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 method can also be applied for samples containing suspended matter. Halogens adsorbed on the suspended solids (e.g. undissolved halides) are determined, too. Filtration of the sample prior to analyses 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.

Procedures for each separate parameter are described in normative [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 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.

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The AOCl, AOBr and AOI results according to [Annex B](#), [Annex C](#) and [Annex D](#) can also be reported as CIC-AOX_(Cl) ([Annex J](#)).

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*

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

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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

CIC-AOX

equivalent mass of chlorine, bromine and iodine representing the sum of organically bound chlorine, bromine and iodine, measured under the conditions of this method; expression of results as mass concentration of chlorine

4 Interferences

4.1 Interference during adsorption

Sample matrix characteristics (e.g. high concentrations of dissolved 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 the influence of DOC on the adsorption reaction of the sample can be provided by current measurements or previous testing of the sampling point. For samples not filtered, 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 is analysed at least the earliest 8 h after sampling.

Particulate inorganic halogen compounds with a melting point $< 1\ 000\ ^\circ\text{C}$ cause a positive bias.

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

High contents of undissolved substances can cause interference with the column method. In these cases, the use of the shaking procedure ([Annex E](#)) should be considered.

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

4.2 Interferences on 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.

Missing or too little water supply in the AOF determination can cause a negative bias.

4.3 Interferences on 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

Basic requirements for the procedure are:

- 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.

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- 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 of 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 absorbing solution

The absorbing solution is injected into an ion chromatograph with a suppressed conductivity detector where anions are separated and quantitated. 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. Determination 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 $\lambda = (215 \pm 10)$ nm for bromide and at $\lambda = (226 \pm 10)$ nm for iodide.

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 $\geq 18 \text{ M}\Omega \cdot \text{cm}$ (25 °C).

6.3 Reagents for sample stabilization and pre-treatment.

6.3.1 Nitric acid, $w(\text{HNO}_3) = 65 \%$.

6.3.2 Sodium sulfite, Na_2SO_3 .

6.3.2.1 Sodium sulfite solution, $c(\text{Na}_2\text{SO}_3) = 1 \text{ mol/l}$.

Dissolve 126 g Na_2SO_3 (6.3.2) in water (6.2) in a 1 000 ml volumetric flask and dilute to volume with water (6.2).

The solution is stable for four weeks if stored at $(5 \pm 3) \text{ }^\circ\text{C}$.

6.3.3 Iodine starch paper.

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6.3.4 Potassium iodide.

6.3.5 Starch solution, mass fraction 1 %.

6.4 Reagents and gases for adsorption, combustion and absorption.

6.4.1 Activated carbon with an iodine number > 1 050 mg/g.

Suitable natural coal-derived activated carbons have particle sizes in the range of about 50 µm to 150 µm. Synthetic materials with larger specific surfaces may also be used.

Various methods can be used to determine the adsorption capacity. One of these methods is described, e.g. in EN 12902. The iodine number gives an indication of the adsorption capacity of the activated carbon.

NOTE 1 Activated carbon qualities with grain sizes < 50 µm can lead to blockage of the adsorption column.

NOTE 2 To prevent the activated carbon from being contaminated by adsorption of halogen-containing compounds from the laboratory air during storage, the activated carbon should be stored in an airtight container (e.g. aluminium screw bottle, halogen polymer-free plastic bottle, or similar). Only the daily required amount for the intended series or samples should be taken from a separate container. The remainder of the activated carbon taken from the container each day should be discarded. Any additional activated carbon not used within the same day shall be discarded.

NOTE 3 The halogen blank values of the activated carbon can be determined by analysing e.g. 100 mg of the activated carbon analyses moistened with 100 µl water (6.2). The result serves as an indication of relevant impurities. Ultimately, the analytically decisive factor is the method blank.

The applicability of an activated carbon batch shall be checked on the recoveries of fluorine, chlorine, bromine or iodine from model substances via the overall procedure given in [Annex A](#), [Annex B](#), [Annex C](#) and [Annex D](#). The compounds listed in [Annex A](#), [Annex B](#), [Annex C](#) and [Annex D](#) shall be used as test reagents.

6.4.2 Sodium nitrate, NaNO₃.

6.4.2.1 Neutral nitrate stock solution I, $c(\text{NaNO}_3) = 2 \text{ mol/l}$ for the AOF determination.

Dissolve 170 g sodium nitrate (6.4.2) in water (6.2) in a 1 000 ml volumetric flask and dilute to volume with water (6.2).

The solution is stable for six months if stored in an amber glass bottle.

6.4.2.2 Neutral nitrate wash solution II, $c(\text{NaNO}_3) = 0,01 \text{ mol/l}$ for the AOF determination.

Pipette 5 ml of neutral nitrate stock solution (6.5.2.1) in a 1 000 ml volumetric flask and dilute to volume with water (6.2).

The solution is stable for four weeks if stored in an amber glass bottle.

6.4.2.3 Nitric acid nitrate stock solution I, $c(\text{NaNO}_3) = 2 \text{ mol/l}$ for the AOCl, AOBr and AOI determination

Dissolve 170 g sodium nitrate (6.5.2) in water (6.2) in a 1 000 ml volumetric flask, add 25 ml nitric acid (6.4.1) and dilute to volume with water (6.2).

The solution is stable for six months if stored in an amber glass bottle.

6.4.2.4 Nitric acid nitrate wash solution II, $c(\text{NaNO}_3) = 0,01 \text{ mol/l}$, $\text{pH} \approx 1,7$ for the AOCl, AOBr and AOI determination.

Pipette 5 ml of nitric acid stock solution (6.4.2.3) in a 1 000 ml volumetric flask and dilute to volume with nitric acid dilution water (6.4.6).

The solution is stable for four weeks if stored in an amber glass bottle.

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6.4.3 Ethanol, C₂H₅OH, absolute.

6.4.4 Standard solutions for the test of the overall procedure

Organic single and multi-component stock solutions with appropriate and required specification may be used if commercially available. These solutions are stable for six months. The manufacturer specifications shall be observed. Alternatively, stock solutions may be prepared by weighing. See [Annex A](#), [Annex B](#), [Annex C](#) and [Annex D](#).

6.4.5 Dilution water and blank solution for the determination of the total AOF blank value 100 ml water ([6.2](#)).

6.4.6 Nitric acid dilution water and blank solution for the determination of the total AOCl, AOBr and AOI blanks

100 ml water ([6.2](#)), acidified with 0,2 ml nitric acid ([6.3.1](#)).

6.4.7 Oxygen, or a mixture of oxygen and inert gas (e.g. argon)

NOTE 1 Gas purities for oxygen $\geq 99,995\%$ and argon $\geq 99,998\%$ have proven to be sufficient.

NOTE 2 The use of argon is recommended when using adsorption columns packed with cellulose wool. Adsorption columns packed with ceramic wool or quartz wool can possibly be combusted without the use of an inert gas. For a residue-free conversion of e.g. cellulose wool packed adsorption columns, an inert gas (e.g. argon) should be used. Otherwise, incomplete combustion, resulting in incorrect results can occur.

6.4.8 Absorption solution for combustion gases

Water ([6.2](#)). Alternatively, solutions according to [C.5.5](#) and [D.5.5](#) may be prepared for absorption.

The use of an internal standard in the absorption solution is necessary if the volume of the absorption solution cannot be determined prior to sample injection into the ion chromatography. For example, methanesulfonic acid or phosphate solutions can be used as internal standards ([6.5.5](#)).

NOTE The addition of reducing agents, e.g. hydrogen peroxide in an alkaline medium, can be necessary for the determination of bromine and iodine ([C.5.5](#) and [D.5.5](#)).

6.5 Reagents for the ion chromatography

6.5.1 Eluents

Carbonate, hydrogen carbonate, hydroxide salts, e.g. manually, automatically or electrochemically generated in situ, may be used as eluents. The choice of eluent depends on the selected separation column and detector; follow the column manufacturer's instructions. The selected combination of separation column and eluent shall fulfil the requirement for the peak resolution according to [7.8](#). The eluents may be used as long as the requirements according to [7.8](#) are fulfilled. An example of a suitable eluent is described in [6.5.2.2](#).

Degas the eluents, if necessary. A renewed gas uptake shall be prevented during operation (e.g. by helium overlay or inline degasser).

6.5.2 Sodium carbonate, Na₂CO₃

6.5.2.1 Sodium carbonate concentrate, $c(\text{Na}_2\text{CO}_3) = 0,09 \text{ mol/l}$.

Dissolve 9,54 g sodium carbonate ([6.5.2](#)) in water ([6.2](#)) in a 1 000 ml volumetric flask and dilute to volume with water ([6.2](#)).

The solution is stable for six months if stored at $(5 \pm 3) \text{ }^\circ\text{C}$.