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**Water quality — Determination of  
adsorbable organically bound halogens  
(AOX)**

*Qualité de l'eau — Dosage des composés organiques halogénés  
adsorbables (AOX)*

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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 9562 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

This third edition cancels and replaces the second edition (ISO 9562:1998), which has been technically revised.

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## Introduction

Adsorbable organically bound halogens (AOX) is an analytical convention. The result is a parameter used for water quality control purposes. It represents the sum of organically bound chlorine, bromine and iodine (but not fluorine) that can be adsorbed on activated carbon under specified conditions and, if the sample is not filtered, includes that associated with suspended matter.

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

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# Water quality — Determination of adsorbable organically bound halogens (AOX)

**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.

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

## 1 Scope

This International Standard specifies a method for the direct determination of an amount of usually 10 µg/l in water of organically bound chlorine, bromine and iodine (expressed as chloride) adsorbable on activated carbon.

This method is applicable to test samples (see 9.2) with concentrations of inorganic chloride ions of less than 1 g/l. Samples with higher concentrations are diluted prior to analysis.

This method is also applicable to samples containing suspended solids where halogens are adsorbed onto the solid matter (e.g. insoluble halides). Filtration of the sample before analysis allows the separate determination of dissolved and particulate adsorbable organically bound halogens (AOX).

Filtered samples with high inorganic chloride content can be analysed by a modified method [dissolved adsorbable organically bound halogens after solid phase extraction in waters with high salt content (SPE-AOX)] (see Annex A). However, results obtained by this modified method can differ significantly from those of the required method.

## 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 1773:1997, *Laboratory glassware — Narrow-necked boiling flasks*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

## 3 Terms and definitions

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

### 3.1

#### adsorbable organically bound halogens

#### AOX

equivalent amount of chlorine, bromine, and iodine contained in organic compounds, expressed as chloride when determined according to this International Standard

**3.2**  
**dissolved organic carbon**  
**DOC**

amount of organically bound carbon present in water originating from compounds passing through a membrane filter of 0,45 µm pore size and including cyanate and thiocyanate

**4 Interferences**

**4.1** High AOX values can result from the presence of active chlorine and of some inorganic bromine and iodine compounds, irreversibly bound to activated carbon. Reactions of these oxidizing agents with organic substances in the sample and with the activated carbon can be prevented by the addition of sodium sulfite, immediately after sampling.

**4.2** Organic bromine and iodine compounds may, during combustion, decompose to elemental bromine or iodine respectively and this can yield higher oxidation states of these elements. These fractions of AOX may be incompletely determined, thus leading to negative bias.

**4.3** Samples containing living cells (for example microorganisms or algae) may give rise to high results because of their chloride content. In these cases, the sample is not analysed until at least 8 h after acidification.

**4.4** For samples with high chloride concentrations (approximately 1 g/l), the shaking procedure (9.3.2) can result in higher interferences than the column procedure (9.3.4).

**4.5** Alcohols, aromatic compounds, or carboxylic acids may give rise to negative bias (e.g. in case of DOC values > 100 mg/l).

**4.6** For samples containing suspended solids, the stirring method (9.3.3) may lead to an insufficient covering of the particles. If the particles contain substances contributing to the AOX, the shaking or column method is recommended.

**4.7** The recovery of some polar and hydrophilic compounds, such as monochloroacetic acid, is incomplete.

**5 Principle**

Acidification of the water sample with nitric acid. Adsorption of organic compounds contained in the sample onto activated carbon, either by a shaking procedure, a stirring procedure, or by column adsorption. Displacement of inorganic halides by rinsing the activated carbon with sodium nitrate solution acidified with nitric acid. Combustion of the loaded carbon in an oxygen stream. Absorption of the hydrogen halides in an acceptor solution followed by determination of the halide ions by an argentometric titration, such as microcoulometry. Expression of the result as the mass concentration of chloride.

**6 Reagents**

Use only reagents of recognized analytical grade. The purity of water, reagents and gases shall be confirmed.

The AOX content shall be negligible when compared with the lowest AOX content to be determined. The overall AOX content of water, chemicals and gases can be checked by measuring the total blank (10.2).

**6.1 Water**, Grade 1 as specified in ISO 3696:1987.

**6.2 Activated carbon**, the handling of which is given in Annex B, for one of three procedures listed in 6.2.1 to 6.2.3.



Several methods may be applied to determine the adsorption capacity. One of these methods is described in reference [1]. The iodine number gives an indication of the activated carbon adsorption capacity. According to the method given in reference [1], the iodine numbers shall be  $> 1\ 050$ .

The blank value of the washed activated carbon shall be less than  $15\ \mu\text{g}$  of chloride equivalent per gram of activated carbon.

**6.2.1 Activated carbon for the shaking procedure**, with a grain size of about  $10\ \mu\text{m}$  to  $50\ \mu\text{m}$ .

**6.2.2 Activated carbon fleeces for the stirring procedure**, are commercially available<sup>1)</sup>.

**6.2.3 Activated carbon for the column procedure**, with a grain size of about  $50\ \mu\text{m}$  to  $150\ \mu\text{m}$ .

**6.3 Nitric acid**,  $\text{HNO}_3$ .

**6.3.1 Nitric acid, concentrated**,  $\rho(\text{HNO}_3) = 1,4\ \text{g/ml}$ ,  $w(\text{HNO}_3) = 65\ \%$ .

**6.3.2 Nitric acid, dilute**,  $c(\text{HNO}_3) = 0,02\ \text{mol/l}$ .

**6.4 Hydrochloric acid**,  $c(\text{HCl}) = 0,010\ \text{mol/l}$ .

The molarity shall be known precisely, since the acid is used for checking the microtitration (9.5.1).

**6.5 Sulfuric acid**,  $\text{H}_2\text{SO}_4$ ,  $\rho(\text{H}_2\text{SO}_4) = 1,84\ \text{g/ml}$ .

**6.6 Gases for combustion**, for example oxygen ( $\text{O}_2$ ), or a mixture of oxygen and an inert gas.

**6.7 Nitrate**, stock solution,  $c(\text{NaNO}_3) = 0,2\ \text{mol/l}$ .

Dissolve  $17\ \text{g}$  of sodium nitrate ( $\text{NaNO}_3$ ) in water (6.1) in a  $1\ 000\ \text{ml}$  volumetric flask, add  $25\ \text{ml}$  of concentrated  $\text{HNO}_3$  (6.3.1) and make up to volume with water (6.1).

If stored in a brown glass bottle, the solution is stable for three months.

**6.8 Nitrate washing solution**,  $c(\text{NaNO}_3) = 0,01\ \text{mol/l}$ ,  $\text{pH} \approx 1,7$ .

Pipette  $50\ \text{ml}$  of the nitrate stock solution (6.7) in a  $1\ 000\ \text{ml}$  volumetric flask, and make up to volume with water (6.1).

If stored in a brown glass bottle, the solution is stable for one month.

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

Dissolve  $126\ \text{g}$  of  $\text{Na}_2\text{SO}_3$  in water (6.1) in a  $1\ 000\ \text{ml}$  volumetric flask and make up to volume with water (6.1).

The solution is stable for one month if stored at  $2\ ^\circ\text{C}$  to  $8\ ^\circ\text{C}$ .

**6.10 4-Chlorophenol, stock solution**,  $\rho_{\text{Cl}}(\text{AOX}) = 200\ \text{mg/l}$ .

Dissolve  $72,5\ \text{mg}$  of 4-chlorophenol ( $\text{C}_6\text{H}_5\text{ClO}$ ) in water (6.1) in a  $100\ \text{ml}$  volumetric flask and make up to volume with water (6.1).

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1) A suitable product available commercially can be obtained from Normenausschuss Wasserwesen (NAW) im DIN Deutsches Institut für Normung e. V., 10772 Berlin. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product. Equivalent products may be used if they can be shown to lead to the same results.

For security reasons, it is advisable to use commercially available solutions.

This stock solution may be stored one month at 2 °C to 8 °C in a glass bottle.

**6.11 4-Chlorophenol, working solution,  $\rho_{Cl}(AOX) = 1 \text{ mg/l}$ .**

Pipette 5 ml of 4-chlorophenol, stock solution (6.10), into a 1 000 ml volumetric flask, and make up to volume with water (6.1).

This working solution may be stored one week at 2 °C to 8 °C in a glass bottle.

**6.12 2-Chlorobenzoic acid, stock solution,  $\rho_{Cl}(AOX) = 250 \text{ mg/l}$ .**

Dissolve 110,4 mg of 2-chlorobenzoic acid ( $\text{ClC}_6\text{H}_4\text{COOH}$ ) in water in a 100 ml volumetric flask and make up to volume with water (6.1).

The dissolution of 2-chlorobenzoic acid is very slow. It is recommended to prepare this solution the day before using it.

This stock solution may be stored for one month at 2 °C to 8 °C in a glass bottle.

**6.13 2-Chlorobenzoic acid, working solution,  $\rho_{Cl}(AOX) = 1 \text{ mg/l}$ .**

Pipette 4 ml of 2-chlorobenzoic acid, stock solution (6.12), into a 1 000 ml volumetric flask, and make up to volume with water (6.1).

This working solution may be stored 1 week at 2 °C to 8 °C in a glass bottle.

**6.14 Standard solutions for the checks, on the overall procedure (9.5.2).**

Pipette, for example, 1 ml, 5 ml, 10 ml, 20 ml, and 25 ml of the working solutions (6.11 or 6.13) into five separate 100 ml volumetric flasks, and make up to volume with water (6.1).

The AOX mass concentration of these solutions is 10 µg/l, 50 µg/l, 100 µg/l, 200 µg/l and 250 µg/l respectively.

The concentration of the solutions should be chosen in such a way that the total working range is covered.

Prepare fresh standard solutions on the day of use.

**6.15 Potassium iodide (KI).**

**6.16 Starch solution, having a mass fraction of 1 %.**

## 7 Apparatus

### 7.1 Activated carbon adsorption apparatus.

#### 7.1.1 Adsorption apparatus for the shaking procedure (9.3.2).

**7.1.1.1 Filtration apparatus,** for example with filter funnel of capacity of  $V = 0,15 \text{ l}$  and filter diameter of 25 mm.

**7.1.1.2 Polycarbonate membrane filter,** for example with diameter of 25 mm and pore width of 0,45 µm or equivalent filter material.

**7.1.1.3 Conical flask,** 250 ml, in accordance with ISO 1773.

**7.1.1.4 Mechanical shaking device for the flasks**, specified in 7.1.1.3, for example with carrier plate.

**7.1.2 Adsorption apparatus for the stirring method** (9.3.3)

**7.1.2.1 Conical flask**, 100 ml, in accordance with ISO 1773.

**7.1.2.2 Attachment device for the activated carbon fleece**, e.g. coil, attached to the stopper.

**7.1.2.3 Activated carbon fleece**, for example round discs, 13 mm × 3 mm, activated carbon fibre (100 % carbon), specific surface 1 600 m<sup>2</sup>/g, mass 25 mg (for each measurement, two fleeces with a total mass of 50 mg of activated carbon are used).

**7.1.2.4 Rinsing device**, e.g. magnetic stirrer with cylindrical glass filter funnel for fixation of the activated carbon fleece.

**7.1.3 Adsorption apparatus for the column procedure** (9.3.4).

Suitable pump, e.g. piston pump with polytetrafluoroethene (PTFE) tube and with adsorption tubes, inner diameter about 3 mm, length 40 mm to 50 mm, attached vertically one behind the other. Other column dimensions are acceptable. Each tube should be filled with about 50 mg of activated carbon. Add the sample from the top of the column and transport it through the column using overpressure.

**7.1.3.1 Ceramic wool**, or equally suitable material, free from halogens, for fixation of the activated carbon in the columns.

**7.2 Apparatus for combustion and detection.**

**7.2.1 Combustion apparatus**, (consisting of a furnace, capable of being heated at least to 950 °C, equipped with a quartz tube, with an inner diameter of 2 cm to 4 cm and length of about 30 cm (see example Figure 1), of which both vertical and horizontal combustion tubes are common.

**7.2.2 Quartz container**, suitable for the quartz tube

**7.2.3 Argentometric measurement device**, for determination of halide concentrations, usually microcoulometer, suitable for the determination of 1 µg of chloride (absolute) with a coefficient of variation of 10 % (repeatability), or similar device (e.g. titration with a diluted AgNO<sub>3</sub> solution) for the determination of chloride ions.

**7.2.4 Absorber**, filled with sulfuric acid (6.5) for gas drying. The acid shall not backflush into the oven. The acid shall be exchanged if its volume has increased by more than 20 %.

**7.2.5 Syringe**, suitable for pipetting 10 µl to 100 µl of hydrochloric acid (6.4).

**7.3 Additional apparatus.**

**7.3.1 Measuring flask**, 1 000 ml, e.g. ISO 1042 - A 1 000 - C.

**7.3.2 Flat bottomed sampling bottles**, preferably brown glass, 1 000 ml.