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Kakovost vode – Določevanje organsko vezanih halogenov, sposobnih adsorpcije (AOX)

Water quality -- Determination of adsorbable organically bound halogens (AOX)

Qualité de l'eau -- Dosage des halogènes adsorbables organiquement liés (AOX)

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

Qualité de l'eau — Dosage des halogènes adsorbables organiquement liés (AOX)

Reference number
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Contents

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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 3.

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

This second edition cancels and replaces the first edition (ISO 9562:1989), which has been technically revised.

Annexes A and B of this International Standard are for information only.

Introduction

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

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

1 Scope

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

The method is applicable to concentrations of inorganic chloride ions in the test sample (see clause 9) of less than 1 g/l. Samples with higher concentrations need to be diluted prior to analysis.

For samples containing suspended solids, halogens adsorbed onto the solid matter are also included. Filtration of the sample before analysis enables the determination of dissolved and particulate AOX to be carried out.

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

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3696, *Water for laboratory use — Specification and test methods*.

3 Terms and definitions

For the purposes of this International Standard, 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 in accordance with this International Standard

3.2
dissolved organic carbon
DOC
amount of organic carbon present in a water sample after filtration through a membrane filter of pore size 0,45 µm

4 Principle

The water sample is acidified with nitric acid. Organic compounds contained in the sample are adsorbed onto activated carbon, either by a shaking procedure or by column adsorption. Inorganic halides are displaced by rinsing the activated carbon with acidified sodium nitrate solution. Combustion of the loaded carbon is then carried out in an oxygen stream. Adsorption of the hydrogen halides thus formed is followed by determination of the halide ions by an argentometric titration, such as microcoulometry. The result is expressed as the mass concentration of chloride.

5 Interferences

5.1 High AOX values can result from the presence of free chlorine. Reactions of this oxidizing agent with organic substances in the sample and with the activated carbon can be prevented by the addition of sodium sulfite, immediately after sampling.

5.2 Some inorganic bromine and iodine compounds are irreversibly bound to activated carbon, causing positive bias. These interferences can be diminished by the addition of sodium sulfite.

5.3 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.

5.4 Insoluble inorganic halides can cause positive bias.

5.5 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.

5.6 For samples with high chloride concentrations (approximately 1 g/l) the shaking procedure (see 9.3.1) can result in higher interferences (positive bias, see 10.2) than the column procedure (see 9.3.2).

6 Reagents

Use only reagents of recognized analytical grade and water of grade 1 in accordance with ISO 3696.

The purity of water, reagents and gases shall be confirmed.

For successful application of the method, the AOX content must be negligibly low 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 content in the total blank (see 9.6).

6.1 Activated carbon.

For the shaking procedure, use an activated carbon of about 10 µm to 50 µm grain size. For the column adsorption, use a grain size distribution of 50 µm to 150 µm.

For the storage of activated carbon, see annex A.

NOTE Several methods can be used for the determination of the adsorption capacity. One of these methods is described in [1]. The iodine number gives an indication of the adsorption capacity of the activated carbon. The iodine number determined in accordance with the method specified in [1] should be > 1 050.

The blank value of the washed activated carbon shall be less than 15 µg of chloride equivalent per gram of activated carbon.

6.2 Nitric acid, HNO_3, ρ = 1,4 g/ml, 65 % mass fraction solution.

6.3 Hydrochloric acid, c(HCl) = 0,100 mol/l.

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

6.4 Sulfuric acid, H_2SO_4, ρ = 1,84 g/ml.

6.5 Gases for combustion, for example oxygen (O_2), or a mixture of oxygen and an inert gas.

6.6 Nitrate stock solution, $c(NaNO_3)$ = 0,2 mol/l.

Dissolve 17 g of sodium nitrate ($NaNO_3$) in water in a 1 000 ml volumetric flask, add 1,4 ml of nitric acid (see 6.2), and make up to volume with water.

6.7 Nitrate washing solution, $c(NaNO_3)$ = 0,01 mol/l.

Pipette 50 ml of the nitrate stock solution (see 6.6) in a 1 000 ml volumetric flask, and make up to volume with water.

6.8 Sodium sulfite solution, $c(Na_2SO_3)$ = 1 mol/l.

Dissolve 126 g anhydrous Na_2SO_3 in water in a 1 000 ml volumetric flask and make up to volume with water.

6.9 4-Chlorophenol, stock solution, AOX = 200 mg/l.

Dissolve 72,5 mg of 4-chlorophenol (C_6H_4ClOH) in water in a 100 ml volumetric flask and make up to volume with water.

6.10 4-Chlorophenol, working solution, AOX = 1 mg/l.

Pipette 5 ml of 4-chlorophenol, stock solution, (see 6.9) into a 1 000 ml volumetric flask, and make up to volume with water.

6.11 2-Chlorobenzoic acid, stock solution, AOX = 250 mg/l.

Dissolve 110,4 mg of 2-chlorobenzoic acid (ClC_6H_4COOH) in water in a 100 ml volumetric flask and make up to volume with water.

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

6.12 2-Chlorobenzoic acid, working solution, AOX = 1 mg/l.

Pipette 4 ml of 2-chlorobenzoic acid, stock solution (see 6.11), into a 1 000 ml volumetric flask, and make up to volume with water.

The stock solutions (see 6.9 and 6.11) may be stored for at least 1 month and the working solutions (see 6.10 and 6.12) for 1 week at 4 °C in glass bottles.

6.13 Standard solutions for checks on the overall procedure (9.5.2)

Pipette 1 ml, 5 ml, 10 ml, 20 ml, and 25 ml of the working solutions (see 6.10 or 6.12) into five separate 100 ml volumetric flasks, and make up to volume with water.

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 standard solutions shall be prepared daily.