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Water quality — Determination of dissolved anions by liquid chromatography of ions —

Part 4: Determination of chlorate, chloride and chlorite in water with low contamination

Qualité de l'eau — Dosage des anions dissous par chromatographie des ions en phase liquide —

Partie 4: Dosage des ions chlorate, chlorure et chlorite dans des eaux faiblement contaminées

Reference number
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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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 230, *Water analysis,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 10304-4:1997), which has been technically revised. The main changes compared to the previous edition are as follows:

— in the introduction, all requirements concerning the application of the method have been deleted and moved to other clauses;

— in Clause 2, all the references made but withdrawn since the publication of the 1997 edition (e.g. ISO 10304-2) have been deleted and the references ISO 5667-1 and ISO 5667-3 have been moved to the Bibliography;

— in 6.8, various eluent formulations have been reduced to one example;

— in Clause 8, the calculation procedure for the peak resolution according to the USP definition [Formula (1)] has been completed with the EP definition [Formula (2)] (both calculations are equivalent);

— in 9.1, information that drinking water disinfection treatment using chlorine dioxide can cause the formation of chlorite and chlorate (paragraph 2) and helpful precautions to minimize/eliminate such formation (paragraph 3) have been added;

— in Clause 11, the option to report result concentrations in microgram per litre has been added.

A list of all parts in the ISO 10304 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Water quality — Determination of dissolved anions by liquid chromatography of ions —

Part 4: Determination of chlorate, chloride and chlorite in water with low contamination

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 the dissolved anions chlorate, chloride and chlorite in water with low contamination (e.g. drinking water, raw water or swimming pool water).

The diversity of the appropriate and suitable assemblies and the procedural steps depending on them permit a general description only.

For further information on the analytical technique, see Bibliography.

An appropriate pre-treatment of the sample (e.g. dilution) and the use of a conductivity detector (CD), UV detector (UV) or amperometric detector (AD) make the working ranges given in Table 1 feasible.

Table 1 — Working ranges of the analytical method

Anion	Working range mg/l[a]	Detection
Chlorate	0,03 to 10	CD
Chloride	0,1 to 50	CD
Chlorite[b]	0,05 to 1	CD
	0,1 to 1	UV; λ = 207 nm to 220 nm
	0,01 to 1	AD; 0,4 V to 1,0 V

[a] The working range is restricted by the ion-exchange capacity of the columns. If necessary, samples can be adjusted to this range by dilution.

[b] The minimum working range for chlorite of 0,05 mg/l was obtained using calibration checks, but the interlaboratory trials (see Table A.4) showed that it is difficult to obtain this with sufficient accuracy, and only if taking great care.

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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3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Interferences

Organic acids such as mono- and dicarboxylic acids or disinfection by-products (e.g. chloroacetic acid) can interfere.

Dissolved organics can react with the working electrode of the amperometric detector, causing a decrease in sensitivity.

The presence of fluoride, carbonate, nitrite and nitrate can cause interference with the determination of chlorate, chloride and chlorite. The respective concentrations given in Table 2 are typical for conductivity, UV and amperometric detectors.

Elevated loads of chloride and bromide can cause interference with the determination of chlorite and chlorate. Remove chloride and bromide with the aid of special exchangers (9.2).

Solid particles and organic compounds (such as mineral oils, detergents and humic acids) shorten the lifetime of the separator column. They are therefore eliminated from the sample prior to analysis (Clause 9).

Table 2 — Typical cross-sensitivity of anions

Relation of the mass concentration[a] of		Detection method
measured ion	interfering ion	
1 part chlorate	50 parts bromide	CD
1 part chlorate	500 parts nitrate	CD
1 part chloride	500 parts fluoride	CD
1 part chloride	1 000 parts chlorite	CD
1 part chloride	50 parts nitrite	CD
1 part chlorite	100 parts fluoride	CD
1 part chlorite	10 parts fluoride	UV
1 part chlorite	1 000 parts carbonate	CD
1 part chlorite	1 000 parts chloride	CD / UV / AD
1 part chlorite	100 parts nitrite	AD

[a] In case the quality requirements in Clause 8 (e.g. see Figures 2 and 3) are not achieved, the sample shall be diluted.

5 Principle

Liquid chromatographic separation of chlorate, chloride and chlorite is carried out by means of a separator column. A low-capacity anion exchanger is used as the stationary phase and usually aqueous solutions of salts of weak mono- and dibasic acids as mobile phases (eluent, 6.8).

Detection is by CD with or without suppressor device, UV or AD.

When using conductivity detectors, it is essential that the eluents have a sufficiently low conductivity. For this reason, conductivity detectors are often combined with a suppressor device (cation exchangers)

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