
**Water quality — Determination of
dissolved bromate — Method using ion
chromatography (IC) and post column
reaction (PCR)**

*Qualité de l'eau — Détermination du bromate dissous — Méthode utilisant
la chromatographie ionique (IC) et la réaction post-colonne (PCR)*





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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.

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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 11206 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Water quality — Determination of dissolved bromate — Method using ion chromatography (IC) and post column reaction (PCR)

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This 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 trained staff.

1 Scope

This International Standard specifies a method for the determination of dissolved bromate in water (e.g. drinking water, mineral water, raw water, surface water, partially treated water or swimming pool water).

Appropriate pretreatment of the sample (e.g. dilution) allows determination of bromate at concentrations $\geq 0,5 \mu\text{g/l}$.

The working range is restricted by the ion-exchange capacity of the separator column. Dilution of the sample to the bromate working range can be necessary.

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

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the 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 Interferences

Metals present in samples and eluents bind to the resin material of the separator column, resulting in a loss of performance. Metal ions can be eliminated with the aid of special cation exchangers (see 6.2 and Clause 8, Note 1).

Solid particles and organic compounds (e.g. mineral oils, detergents, and humic acids) shorten the lifetime of the precolumn and the separator column (see Clause 8, Notes 2 and 3).

Chlorite can interfere with the determination of bromate. Remove chlorite in accordance with the procedure specified in 9.4, if necessary.

NOTE Any substance that has a retention time coinciding with bromate and that produces a detector response can interfere. A high concentration of ions can have an impact on the resolution and on the analyte's retention time. Sample dilution and/or gradient elution overcomes much interference.

4 Principle

The sample is pretreated in order to remove ozone, chlorine dioxide, chlorite, metals and solids, if necessary (see Clause 8). Bromate is separated by ion chromatography (IC). An anion exchange resin is used as the stationary phase and either acids (e.g. sulfuric acid) or aqueous solutions of weak monobasic acids

and dibasic acids are used as eluents for isocratic or gradient elution (e.g. carbonate-, hydrogen carbonate-, hydroxide-eluent, e.g. manually, automatically or in situ electrochemically prepared) (5.13).

Detection of bromate [$\rho(\text{BrO}_3^-) \geq 0,5 \mu\text{g/l}$] is achieved by applying an acidic solution of potassium iodide containing a catalytic amount of molybdenum(VI), where the bromate reacts with iodide to form the tri-iodide ion in a post column reaction (PCR) step, which is measured by its UV absorption at 352 nm.

NOTE This method can be combined with ISO 10304-1^[2], ISO 10304-4^[3], and ISO 15061^[4].

The concentration of bromate is determined after a calibration according to ISO 8466-1 or ISO 8466-2 of the overall procedure.

Control experiments are necessary to check the validity of the calibration function (9.5). Replicate determinations can be necessary. Use of the method of standard addition may be required if matrix interferences are expected (9.3).

5 Reagents

Use only reagents of pro analysis grade. Weigh the reagents with an accuracy of ± 1 % of the nominal mass, unless stated otherwise. Prepare alternative volumes of solutions as described in 5.13 to 5.19, if necessary.

5.1 Water, ISO 3696, grade 1.

5.2 Sodium carbonate, Na_2CO_3 .

5.3 Sulfuric acid, $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/l}$.

5.4 Sodium hydroxide, NaOH .

5.5 Potassium hydroxide, KOH .

5.6 Sodium hydrogencarbonate, NaHCO_3 .

5.7 Ammonium heptamolybdate tetrahydrate, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$.

5.8 Potassium iodide, KI .

5.9 Nitric acid, $\rho(\text{HNO}_3) = 1,41 \text{ g/ml}$.

5.10 Iron(II) sulfate heptahydrate, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$.

5.11 Potassium bromate, KBrO_3 .

5.12 Ethylenediamine, $\text{C}_2\text{H}_8\text{N}_2$, 99 %.

5.13 Eluents.

Degas all eluents used. Take steps to avoid any renewed air pick-up during operation (e.g. by helium sparging, in-line degasser).

The choice of eluent depends on the choice of column and detector; seek advice from the column supplier. The chosen combination of separator column and eluent shall conform to the resolution requirements specified in Clause 7. Use eluents as long as the requirements in Clause 7 and in 9.3 are met.

A selection of reagents for common eluents is presented in 5.2 to 5.6. Examples for appropriate eluents are given in 5.13.2 and 5.13.3.