
**Water quality — Determination of dissolved
bromate — Method by liquid
chromatography of ions**

*Qualité de l'eau — Dosage du bromate dissous — Méthode par
chromatographie des ions en phase liquide*

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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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15061 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Annexes A, B, C, D and E of this International Standard are for information only.

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Introduction

The essential minimum requirements of an ion chromatographic system applied within the scope of this International Standard for the determination of dissolved bromate are given in clause 5.

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

Further information on the analytical technique can be found in the normative references (clause 2) and the bibliography.

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Water quality — Determination of dissolved bromate — Method by liquid chromatography of ions

1 Scope

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

Appropriate pretreatment of the sample, for example by elimination of chloride, sulfate, metals, preconcentration or dilution, gives a range of applicability of 0,5 µg/l to 1 000 µg/l dissolved bromate.

The working range is restricted by the ion-exchange capacity of any preconcentration columns used and that of the separator column. Dilution of the sample to the working range may be necessary.

2 Normative references

The following normative documents contain 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 editions of the normative documents 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 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

ISO 5667-1:1980, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes*.

ISO 5667-2:1991, *Water quality — Sampling — Part 2: Guidance on sampling techniques*.

ISO 5667-3:1994, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples*.

ISO 8466-1:1990, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*.

ISO 8466-2:1993, *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

3.1 The presence of nitrate, chloride, carbonate and sulfate may affect the capacity of the concentrator column and lead to poor recovery of bromate (9.2.1).

3.2 The presence of chloride, sulfate, carbonate and hydrogen carbonate can cause interference with the determination of bromate (9.2.1). Depending on the column utilized, other ions may interfere; this should be checked.

3.3 Metals present (e.g. barium and silver ions released from sample pretreatment steps) will bind to the resin material of concentrator and separator columns, resulting in a loss of performance. Metal ions may be eliminated with the aid of a metal clean-up column or special exchangers (see Figure 1 and clause 9).

3.4 The interference of some organic acids with the determination of bromate was checked and found not to be significant to the concentrations tested (annex E).

3.5 Solid particles and organic compounds such as mineral oils, detergents and humic acids shorten the life-time of the concentrator and separator column.

4 Principle

4.1 Sample pretreatment is carried out in order to remove ozone (9.1.3) and solids, and to reduce chloride, sulfate, carbonate, hydrogen carbonate and metals present by use of cation exchangers (9.2).

4.2 Measurement of bromate is made in the range 0,5 µg/l to 1 000 µg/l, with or without preconcentration (10.3).

4.3 Liquid chromatographic separation of bromate is carried out either by means of a separator column or after elution of bromate from a concentrator column, if used. An anion exchange resin is used as the stationary phase, and usually, aqueous solutions of salts of weak mono- and dibasic acids as eluent (see 6.10 and annex A).

4.4 A conductivity detector (CD) with chemical suppression is used. A UV detector ($\lambda = 190 \text{ nm}$ to 205 nm) is suitable to confirm the CD results only.

NOTE When using conductivity detectors it is essential that the eluents have a sufficiently low conductivity. For this reason, conductivity detectors are combined with a suppressor device (cation exchanger) which reduces the conductivity of the eluent and transforms the sample species into their respective acids. UV detection measures absorbance directly.

4.5 Strongly retained ions (e.g. nitrate, phosphate, sulfate) are removed from the separator column, e.g. by flushing the separator column with a more concentrated eluent.

4.6 The concentration of bromate is determined after calibration of the overall procedure.

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5 Essential minimum requirements

a) Preconcentration

For low bromate concentrations the use of a concentrator column may be required. On-line techniques can be used (see 10.3 and annex C). Ensure that recovery is within 80 % to 120 %.

b) Resolution power of the column

It is essential that the peak resolution R shall not fall below 1,3 (clause 8, Figure 4) between bromate and the nearest peak, which is usually chloride.

c) Method of detection

Measurement of the electrical conductivity (CD) with a chemical suppressor device, and UV if confirmation is required.

d) Applicability of the method: 0,5 µg/l to 1 000 µg/l.

e) Calibration shall be carried out in accordance with ISO 8466-1 or ISO 8466-2 (10.2).

f) Guarantee of analytical quality

Control is necessary for the validity of the calibration function (10.5). Replicate determinations may be necessary. Use of the method of standard addition may be required when matrix interferences are expected (10.3).

6 Reagents

Use only reagents of recognized analytical grade. Carry out weighing of the reagents with an accuracy of $\pm 1\%$ of the nominal mass, unless stated otherwise.

6.1 Water, complying with grade 1 as defined in ISO 3696.

6.2 Sodium hydrogen carbonate, NaHCO_3 .

6.3 Sodium carbonate, Na_2CO_3 .

6.4 Disodium tetraborate decahydrate, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$.

6.5 Boric acid, H_3BO_3 .

6.6 Potassium bromate, KBrO_3 .

6.7 Nitric acid, $c(\text{HNO}_3) = 0,1 \text{ mol/l}$.

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

6.9 Ethylenediamine, $\text{C}_2\text{H}_8\text{N}_2$.

6.10 Eluents.

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Degas all water used for eluent preparation. Take steps to avoid any renewed air pick-up during operation (e.g. by helium sparging). In order to minimize the growth of bacteria or algae, store the eluents in the dark and renew every 3 d.

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See annex A for examples of eluents.

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Two different types of eluent are used.

6.10.1 Eluent of Type 1, of a lower concentration level (for examples see clause A.1) to be applicable for the separation of bromate

and

6.10.2 Eluent of Type 2, of a higher concentration level (for examples see clause A.2) to be applicable to remove strongly retained ions (e.g. nitrate, phosphate) from the concentrator and separator column.

The choice of eluent is dependent 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 stated in clause 8.

A selection of reagents for common eluents is presented in 6.2 to 6.5.

6.11 Bromate stock standard solution, $\rho(\text{BrO}_3^-) = 1\,000 \text{ mg/l}$

Dry approximately 1,5 g of potassium bromate (6.6) for at least 1 h at $105\text{ °C} \pm 5\text{ °C}$. Store the dried solid in a desiccator.

Dissolve $1,306 \text{ g} \pm 0,001 \text{ g}$ of the dried potassium bromate in approximately 800 ml of water (6.1) in a 1 000 ml volumetric flask, and dilute to volume with water (6.1). Store the solution at 2 °C to 6 °C in polyethylene or glass bottles and renew it every 12 months.

Alternatively, use commercially available stock solutions of the required concentration.

6.12 Bromate standard solutions.

6.12.1 General

Depending upon the concentrations expected, prepare the following standard solutions of different bromate concentrations from the stock standard solution (6.11). Note the possible risk of changes in concentration caused by interaction with the vessel material increases with decreasing bromate concentration. Store the standard solutions in polyethylene or glass bottles.

6.12.2 Bromate Standard Solution I

The mass concentration of this solution is $\rho(\text{BrO}_3^-) = 100 \text{ mg/l}$.

Pipette 10,0 ml of stock standard solution (6.11) into a 100 ml volumetric flask, and dilute to volume with water (6.1).

Store the solution at 2 °C to 6 °C in polyethylene or glass bottles and renew every 6 months.

6.12.3 Bromate Standard Solution II

The mass concentration of this solution is $\rho(\text{BrO}_3^-) = 1 \text{ mg/l}$.

Pipette 1,0 ml of Standard Solution I (6.12.2) into a 100 ml volumetric flask, dilute to volume with water (6.1).

Store the solution at 2 °C to 6 °C in polyethylene or glass bottles and renew every 3 months.

6.13 Bromate calibration solutions. (standards.iteh.ai)

Depending on the bromate concentration expected in the sample, use the Bromate Standard Solution I or II (6.12.2 or 6.12.3) to prepare five to ten calibration solutions distributed over the expected working range as evenly as possible.

For example, proceed as follows for the range 0,5 µg/l to 5,0 µg/l BrO_3^- :

Pipette, into a series of 100 ml volumetric flasks, the following volumes: 50 µl, 100 µl, 150 µl, 200 µl, 250 µl, 300 µl, 350 µl, 400 µl, 450 µl or 500 µl of Bromate Standard Solution II (6.12.3) and dilute to volume with water (6.1).

The concentrations of BrO_3^- in these calibration solutions are: 0,5 µg/l, 1,0 µg/l, 1,5 µg/l, 2,0 µg/l, 2,5 µg/l, 3,0 µg/l, 3,5 µg/l, 4,0 µg/l, 4,5 µg/l and 5,0 µg/l respectively.

Prepare the calibration solutions on the day of use.

6.14 Regeneration solutions.

The choice is dependent on the type of metal clean-up columns or suppressor devices. Therefore, follow the column manufacturer's instructions for the exact composition of the regeneration solutions (for examples of compositions see annex B).

6.15 Blank solution.

Fill a 100 ml volumetric flask with water (6.1).

7 Apparatus

Usual laboratory apparatus, and, in particular:

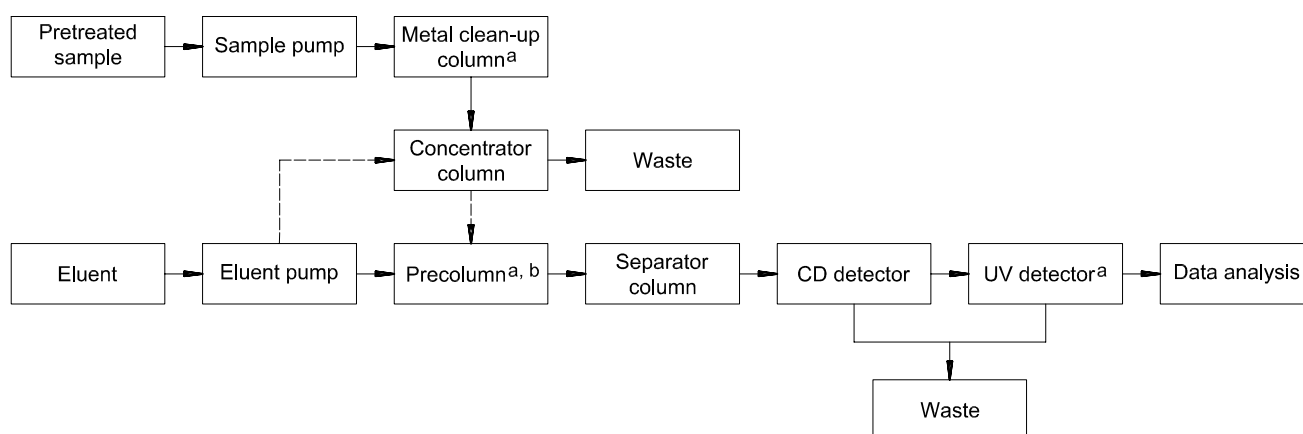
7.1 Ion chromatographic system, complying with the quality requirements of clause 8, i.e. resolution. In general, it shall consist of the following components (see Figure 1):

- eluent reservoirs, and a degassing unit for two eluents;
- pump, suitable for step gradient technique;
- sample delivery device (e.g. sample pump) including a sample injection system incorporating a sample loop of appropriate volume (e.g. 0,05 ml to 2 ml) or autosampler device;
- column-switching valves (e.g. 6-port-valve) including a device for timing and controlling valves and pump;
- concentrator column (may be required for low concentrations);
- separator column with the specified separating performance (see clause 8);
- conductivity detector with an anion suppressor device assembly;
- UV detector (e.g. spectrophotometer: 190 nm to 400 nm);
- recording device (e.g. recorder, integrator with printer, PC with software for data acquisition and evaluation).

NOTE If a preconcentration step is required, see annex C for an example of a possible system configuration.

7.2 Cartridges.

- cation exchanger in the Ag-form (cartridge);
- cation exchanger in the Ba-form (cartridge);
- cation exchanger in the H-form (cartridge);
- optional: metal clean-up column for on-line use;
- cartridges with non-polar phases to be used for sample preparation (e.g. polyvinylpyrrolidone).



Key

- Optional.
- To be recommended for direct injection, when not using a concentrator column (see 10.3, note 1).

Figure 1 — Schematic representation of an ion chromatographic system, including an on-line preconcentration system