INTERNATIONAL STANDARD

ISO 15061

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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

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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.

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.

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 (λ = 190 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.

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 ± 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, $Na_2B_4O_7 \cdot 10\ H_2O$.

6.5 Boric acid, H_3BO_3.

6.6 Potassium bromate, $KBrO_3$.

6.7 Nitric acid, $c(HNO_3)$ = 0,1 mol/l.

6.8 Sulfuric acid, $\rho(H_2SO_4)$ = 1,84 g/ml.

6.9 Ethylenediamine, $C_2H_8N_2$.

6.10 Eluents.

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.

See annex A for examples of eluents.

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, $\rho(BrO_3^-)$ = 1 000 mg/l

Dry approximately 1,5 g of potassium bromate (6.6) for at least 1 h at 105 °C ± 5 °C. Store the dried solid in a desiccator.

Dissolve 1,306 g ± 0,001 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 $\rho(BrO_3^-)$ = 100 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 $\rho(BrO_3^-)$ = 1 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.

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):

a) eluent reservoirs, and a degassing unit for two eluents;

b) pump, suitable for step gradient technique;

c) 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;

d) column-switching valves (e.g. 6-port-valve) including a device for timing and controlling valves and pump;

e) concentrator column (may be required for low concentrations);

f) separator column with the specified separating performance (see clause 8);

g) conductivity detector with an anion suppressor device assembly;

h) UV detector (e.g. spectrophotometer: 190 nm to 400 nm);

i) 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.

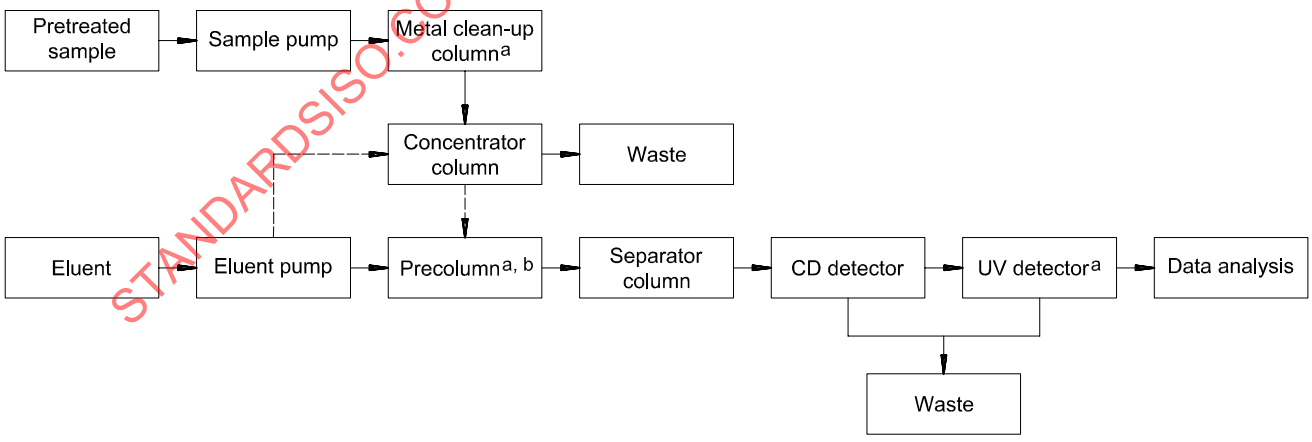
— cation exchanger in the Ag-form (cartridge);

— 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

a Optional.

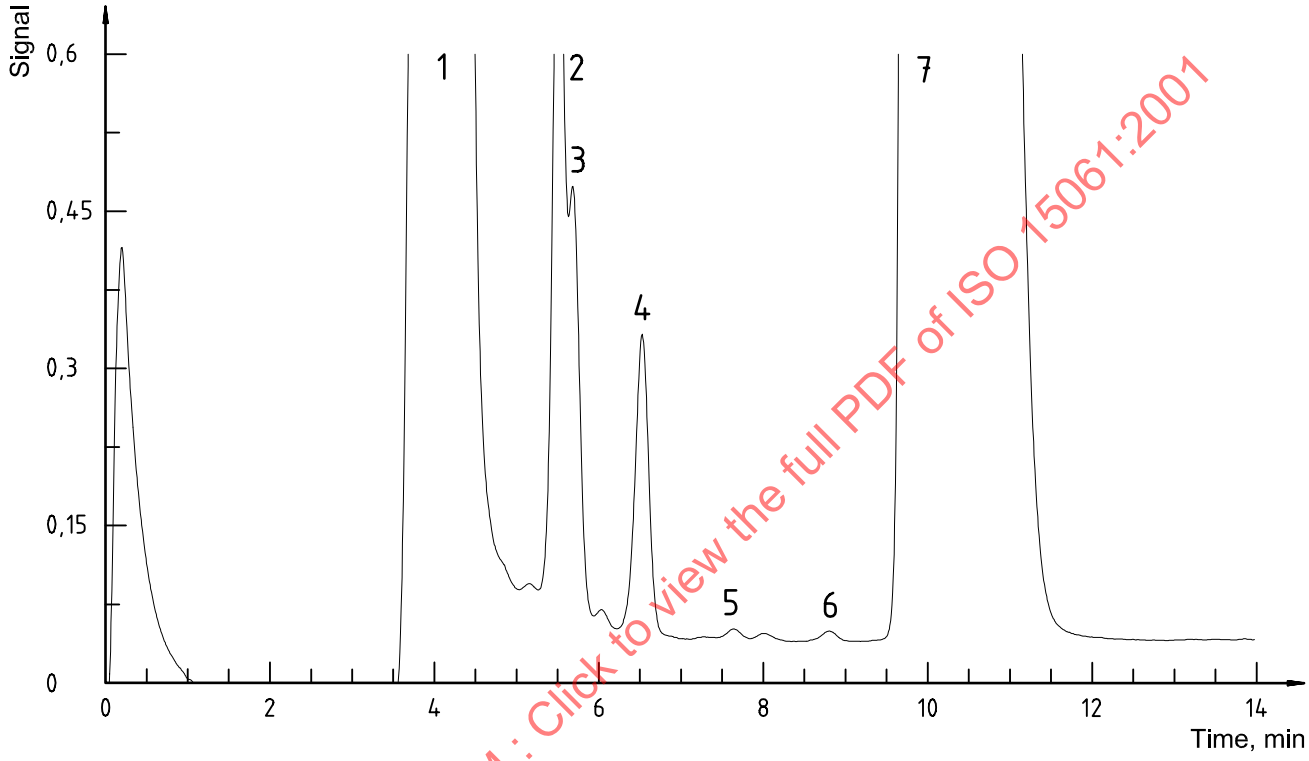
b To be recommended for direct injection, when not using a concentrator column (see 10.3, note 1).

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

8 Quality requirements for the separator column

Separation conditions shall be such that possible interfering anions do not interfere with bromate. Figures 2 and 3 give examples for different types of water matrix checked.

In chromatograms of samples and standard solutions of bromate, the peak resolution R between bromate and its nearest peak, usually chloride, shall not fall below 1,3 [see equation (1) and Figure 4].



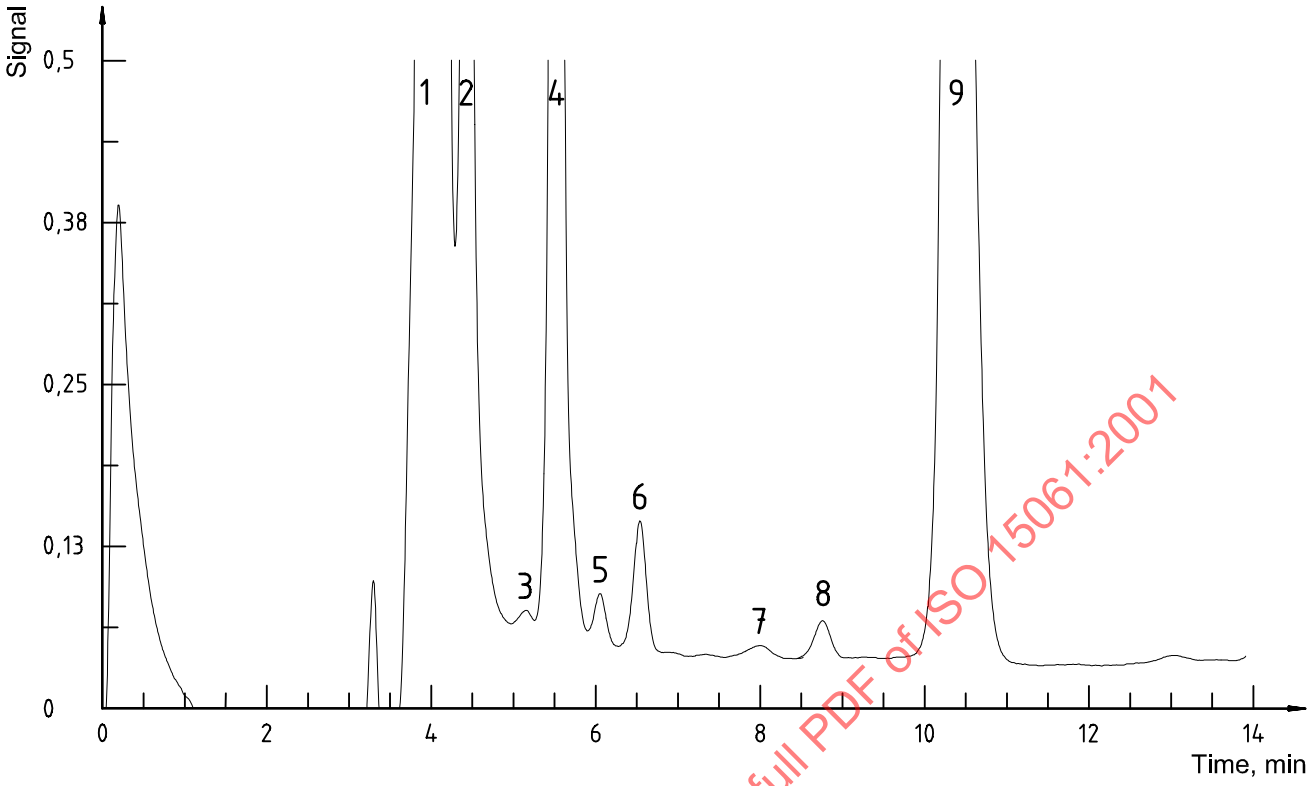
Key

1 Formate, lactate, propionate, acetate or butyrate
2 Valerate or unknown
3 Unknown
4 Chlorite
5 Monobromoacetate
6 0,8 µg/l bromate
7 Chloride

NOTE 1 Verified identification of peaks 6 and 7. Uncertain identification of the other peaks.

NOTE 2 Sample preparation: preconcentration of 2 ml of sample after use of Ag- and H-cartridges according to 9.1.

Figure 2 — Example chromatogram of an ozonylated treated raw water sample prepared conforming to this International Standard



Key

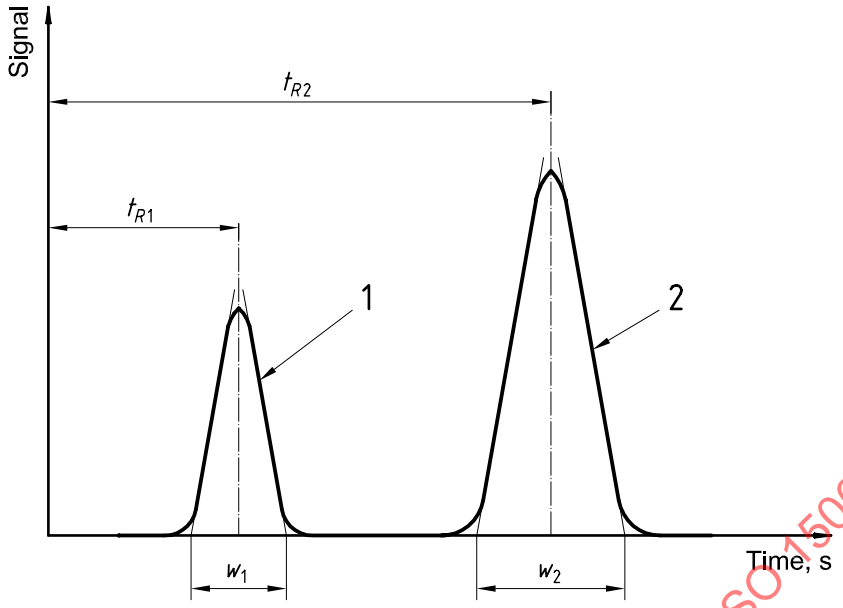
1	Formate	6	Chlorite
2	Propionate, acetate, or butyrate	7	Unknown
3	Valerate	8	Bromate
4	Unknown	9	Chloride
5	Unknown		

NOTE 1 Verified identification of peaks 8 and 9. Uncertain identification of the other peaks.

NOTE 2 Sample preparation: preconcentration of 2 ml of sample after use of Ag- and H-cartridges according to 9.1.

NOTE 3 Elution sequences and retention times (t_R) can vary, depending on the type of column and the eluent composition.

Figure 3 — Chromatogram of a river sample (River Meuse, sample spiked with 3 µg/l bromate) prepared conforming to this International Standard



Key

1 Peak 1
2 Peak 2

Figure 4 — Graphical representation of the parameters to calculate the peak resolution R

Calculate the peak resolution R using equation (1):

$$R_{2,1} = \frac{2 \cdot (t_{R2} - t_{R1})}{w_2 + w_1} \quad (1)$$

where

$R_{2,1}$ is the resolution for the peak pair 2,1;

t_{R1} is the retention time, in seconds, of the first peak;

t_{R2} is the retention time, in seconds, of the second peak;

w_1 is the peak width, in seconds on the time axis, of the first peak;

w_2 is the peak width, in seconds on the time axis, of the second peak.

NOTE w_1, w_2 are the base widths of the isosceles triangles constructed over the Gaussian peaks.

9 Sampling and sample pretreatment

9.1 General requirements

9.1.1 Sampling and sampling preservation procedures shall be in accordance with ISO 5667-1, ISO 5667-2 and ISO 5667-3. Treat the calibration solutions (6.13) and the blank solution (6.15) in the same manner as the sample solution (see Figure 5, steps 1 to 5 and 9.1.3 to 9.2.6).

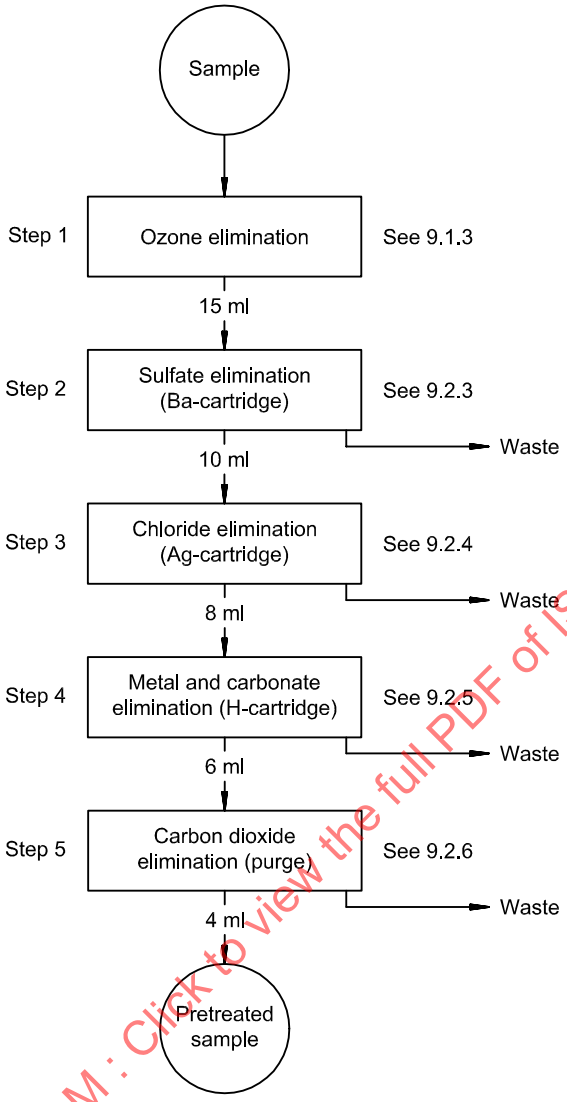


Figure 5 — Pretreatment steps for samples, calibration and blank solution

9.1.2 Use clean polyethylene vessels for sampling.

9.1.3 Avoid any further formation of bromate after sampling by immediately removing any ozone present. For example, add 50 mg of ethylenediamine (6.9) to 1 l of sample immediately after sampling (see Figure 5, step 1).

9.1.4 Store the sample in a polyethene vessel at 2 °C to 6 °C until analysis is carried out.

9.2 Elimination of dissolved sulfate, chloride, carbonate, hydrogen carbonate and metals

9.2.1 If considered necessary, remove chloride, sulfate, carbonate and hydrogen carbonate with the aid of the ion-exchange cartridges described, by carrying out the following elution steps with a constant flowrate of between 1 ml/min and 1,5 ml/min (see Figure 5, steps 2 to 4). Rinse ion-exchange cartridges with water (6.1) before use according to the manufacturer's instructions. In addition, purge the sample with an inert gas (e.g. N_2 or He) to eliminate carbon dioxide (formed from carbonate and hydrogen carbonate salts).

The presence of nitrate, chloride, carbonate and sulfate may affect the capacity of the concentrator column and may lead to poor recovery of bromate. This effect should be checked for every matrix by standard addition, and the recovery of bromate should be in the range 80 % to 120 %.

9.2.2 Prepare the samples as described in 9.1.

9.2.3 Pass approximately 15 ml of the sample through a cation exchanger in the Ba-form (cartridge, 7.2) to remove dissolved sulfate ions from the sample (see Figure 5, step 2). Discard the first portion of 2 ml.

9.2.4 Pass approximately 10 ml of the remaining sample through a strongly acid cation exchanger in the Ag-form (cartridge, 7.2) to remove dissolved halides from the sample (see Figure 5, step 3). Discard the first portion of 2 ml.

9.2.5 Pass approximately 8 ml of the remaining sample through a cation exchanger in the H-form (cartridge, 7.2) to remove dissolved metal ions, carbonate and hydrogen carbonate from the sample (see Figure 5, step 4). Discard the first portion of 2 ml.

NOTE Alternatively, connect all the clean-up columns/cartridges (see Figure 5, steps 2 to 4). In this case, the first 3 ml of eluate of the sample leaving the last cartridge should be discarded (see Figure 5, step 4).

9.2.6 Purge the remaining sample for approximately 5 min with an inert gas (e.g. N_2, He) in order to eliminate carbon dioxide from the sample (see Figure 5, step 5), and analyse the resulting eluate of the sample using the ion chromatographic system.

10 Procedure

10.1 General

Set up the ion chromatographic system (7.1) according to the instrument manufacturer's instructions.

Run the starting eluent; once the baseline is stable analysis can begin.

If metal clean-up, concentrator columns and suppressor devices are being used, regenerate according to the instrument manufacturer's instructions before use.

Perform the calibration as described in 10.2. Measure the samples and blank solution (6.15) as described in 10.3.

10.2 Calibration

Inject the pretreated bromate calibration solutions (6.13 and clause 9). In calculating concentrations, use the characteristic that the area (or height) of the peak (signal) is proportional to the concentration of the bromate ion.

When the analytical system is first evaluated, and at intervals afterwards, establish a calibration function in accordance with ISO 8466-1 or ISO 8466-2 for the measurement as follows.

a) Prepare the bromate calibration solutions as described in 6.13 and clause 9.

b) Analyse the calibration solutions chromatographically.

c) Use the data obtained to calculate the regression line in accordance with ISO 8466-1 or ISO 8466-2.

d) Subsequently, verify the continuing validity of the established calibration function (10.5).

10.3 Measurement of bromate

After establishing the calibration function, inject the pretreated sample (clause 9) into the chromatograph and measure the peaks as described above (clause 10).

Identify the bromate peak by comparing the retention time with that of bromate in the standard solutions (6.12). Take into account the fact that the retention times can be dependent on concentration and matrix.

If a concentrator column is not used, the use of a precolumn is recommended, especially for the injection of waters strongly contaminated with organics (see 3.5 and Figure 1). It serves to protect the analytical separator column.

NOTE 1 In general, two different types of precolumns can be used: those containing the same resin material as the analytical separator column and those packed with a macroporous polymer.

If the bromate concentration of the sample exceeds the calibration range, dilute the sample and re-analyse it.

If the bromate concentration of the sample falls short of the calibration range, establish a separate calibration function for the lower working range, preconcentrate the bromate solution, if necessary, and analyse it.

NOTE 2 There are a number of available systems which can carry out a preconcentration step. The manufacturer's instructions for each system should be followed. Annex C contains an example of a possible system configuration.

If matrix interferences are expected, use the method of standard addition to confirm the results (verify the peaks by comparing the retention time of the spiked sample with those of the original sample).

Measure the blank solution (6.15) in the same manner.

10.4 Confirmation of bromate results

If required, confirm bromate concentrations greater than 2 µg/l by UV detection (λ = 200 nm) as follows.

a) Calculate the bromate slopes of the CD (b_1) and the UV detector (b_2) from calibration experiments according to 10.2, and calculate factor B using equation (2).

$$B = \frac{b_1}{b_2} \tag{2}$$

where

b_1 is the slope of the calibration function for the CD detector, e.g. mm · l/mg; µV · s · l/mg;

b_2 is the slope of the calibration function for the UV detector, e.g. mm · l/mg; µV · s · l/mg.

b) Analyse a bromate calibration solution, e.g. $\rho(BrO_3)$ = 10 µg/l.

c) Record the measured CD value (Y_1) and the measured UV value (Y_2) for bromate.

d) Calculate the ordinate intercept for CD (a_1) and UV (a_2) according to 10.2.

e) Calculate the response ratio r [see equation (3)]:

$$r = \left(\frac{Y_1 - a_1}{Y_2 - a_2} \right) \tag{3}$$

where

r is the response ratio;

Y_1 is the measured value (size of signal) for the CD detector, in terms of peak height or peak area, respectively in millimetres or microvolt seconds;

Y_2 is the measured value (size of signal) for the UV detector, in terms of peak height or peak area, respectively in millimetres or microvolt seconds;

a_1 is the ordinate intercept of the calibration function (calculated blank) for the CD detector, e.g. mm, µV · s (10.2);

a_2 is the ordinate intercept of the calibration function (calculated blank) for the UV detector, e.g. mm, µV · s (10.2).

r [see equation (3)] shall be in the range of $B \pm 10$ %. If r exceeds the range of 10 %:

— use the method of standard addition;

— calculate r again; if r still exceeds the range of $B \pm 10$ % then mark the result as “bromate not confirmed”.

10.5 Validity check of the calibration function

In order to verify the continuing validity of the calibration function, measure standard solutions of different bromate concentrations in the lower and upper thirds of the working range. Carry this out after the set-up procedure (see clause 10) and after each sample series at least, but in any case after 20 measurements. Recalibrate, if necessary.

11 Calculation

Calculate the mass concentration, ρ, in micrograms per litre, of bromate in the solution using the peak areas or peak heights (10.3) in accordance with ISO 8466-1 or ISO 8466-2.

Take into account all of the dilution steps.

12 Expression of results

Results shall be reported to a maximum of two significant figures.

EXAMPLES

Bromate (BrO_3^-) 5,1 µg/l

Bromate (BrO_3^-) 0,6 µg/l

13 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

b) identity of the water sample;

c) expression of the results in accordance with clause 12;

d) description of sample pretreatment, if relevant;

e) any deviation from this method.

Annex A
(informative)

Eluents

A.1 Examples of eluents of Type 1 to be used for bromate separation

A.1.1 General

Solutions of sodium hydroxide and salt solutions of weakly dissociated acids, such as sodium carbonate/sodium hydrogen carbonate, sodium hydrogen carbonate and sodium tetraborate, can be used.

A.1.2 Sodium hydrogen carbonate concentrate I

The addition of the following eluent concentrate is appropriate for the eluent preparation (A.1.3):

Place 58,8 g of sodium hydrogen carbonate (see 6.2) into a 1 000 ml volumetric flask, dissolve in water (6.1) and dilute to volume with water (6.1).

The solution contains 0,7 mol/l of sodium hydrogen carbonate. This solution is stable for several months if stored at 2 °C to 6 °C.

A.1.3 Sodium hydrogen carbonate eluent I

The following eluent is applicable for the determination of bromate:

Pipette 5 ml of the concentrate (A.1.2) into a 5 000 ml volumetric flask and dilute to volume with water (6.1).

The solution contains 0,000 7 mol/l of sodium hydrogen carbonate. The solution should be renewed every 3 d.

A.1.4 Borate eluent I

The following eluent is applicable for the determination of bromate:

Place 76,3 g of disodium tetraborate decahydrate (6.4) into a 5 000 ml volumetric flask, dissolve in approximately 4 000 ml of water (6.1), and dilute to volume with water (6.1).

The solution contains 0,04 mol/l of disodium tetraborate. The solution should be renewed every 3 d.

A.2 Examples of eluents of Type 2 to be used to remove strongly retained ions

A.2.1 General

Solutions of sodium hydroxide and salt solutions of weakly dissociated acids, such as sodium carbonate/sodium hydrogen carbonate, sodium hydrogen carbonate and sodium tetraborate, can be used.

A.2.2 Sodium carbonate/sodium hydrogen carbonate concentrate II

The addition of the following eluent concentrate is appropriate for the eluent preparation (A.2.3).

Place 10,6 g of sodium carbonate (6.3) and 8,4 g of sodium hydrogen carbonate (6.2) into a 1 000 ml volumetric flask, dissolve in water (6.1) and dilute to volume with water (6.1).

The solution contains 0,1 mol/l of sodium carbonate and 0,1 mol/l of sodium hydrogen carbonate. The solution is stable for several months if stored at 2 °C to 6 °C.

A.2.3 Sodium carbonate/sodium hydrogen carbonate eluent II

The following eluent is applicable for the removal of strongly retained ions from the separator column.

Place 50 ml of the concentrate (A.2.2) into a 500 ml volumetric flask and dilute to volume with water (6.1).

The solution contains 0,01 mol/l of sodium carbonate and 0,01 mol/l of sodium hydrogen carbonate. The solution should be renewed every 3 d.

A.2.4 Borate eluent II

The following eluent is applicable for the removal of strongly retained ions from the separator column.

Place 477 g of disodium tetraborate decahydrate (6.4) into a 5 000 ml volumetric flask, dissolve in approximately 4 000 ml of water (6.1), and dilute to volume with water (6.1).

The solution contains 0,25 mol/l of disodium tetraborate. The solution should be renewed every 3 d.

Annex B
(informative)

Regeneration solutions

B.1 General

If metal clean-up columns and/or suppressor devices are used, these should be regenerated on a regular basis. The timing of the regeneration shall be determined for each system, and the following regenerant solutions may be used.

B.2 Example of a regenerant solution for metal clean-up columns

The use of nitric acid (6.7) is applicable for the regeneration of metal clean-up columns.

B.3 Example of a regenerant solution for suppressor devices

The use of sulfuric acid is applicable for the regeneration of suppressor devices.

Pipette 3,5 ml of sulfuric acid (6.8) into approximately 4 000 ml of water (6.1) in a 5 000 ml volumetric flask and dilute to volume with water (6.1).

The solution contains 0,012 6 mol/l of H_2SO_4 and is stable for several months if stored at $\leqslant$ 30 °C.

Annex C
(informative)

Example of column-switching technique

C.1 General

Set up the ion chromatographic system in accordance with clause 10.

The manufacturer's instructions should always be followed. The times required for rinsing and equilibration of the system depend, e.g., on the properties of both the given ion-exchange column and the used eluent. Check these times experimentally. All times stated in C.2 to C.7 are intended as examples only. All valve and pump operations can be externally controlled by a time-sequence device (e.g. PC-based chromatography data station). Regenerate the metal clean-up column in accordance with the manufacturer´s instructions.

C.2 System set-up and load of the sample loop

Switch valve 1 (see Figure C.1) allowing the passage of water (6.1) through the metal clean-up column onto the concentrator column. Rinse the system with water (6.1). Fill the injection loop with the pretreated sample.

C.3 Sample injection and preconcentration

Inject 2 ml of the pretreated sample (9.2.6) via valve 1 (see Figure C.1) through the metal clean-up column onto the concentrator column.

C.4 Elution and separation of bromate

Switch valve 2 (see Figure C.1) into position so that eluent type 1 (see clause A.1) is pumped through the concentrator and the separator column.

C.5 Recording

Record the conductivity detector's output signal (e.g. peak area or height).

C.6 Reconditioning of columns

Deliver eluent type 2 (see clause A.2) using the analytical pump (see Figure C.1) through the concentrator and separator column. Rinse both columns for 5 min with eluent type 2 (see clause A.2).

C.7 Equilibration of the IC system

Using the chromatograph's pump (see Figure C.1), deliver eluent type 1 (see clause A.1) through concentrator and separator column. Rinse both columns for 5 min with eluent type 1 (see clause A.1). Equilibrate the ion chromatograph (clause 10) according to the instrument manufacturer's instructions (e.g. the instrument is ready for operation as soon as the baseline is stable).