Column manual

Metrosep A Supp 5 (6.1006.XX0)

Manual

8.107.8040EN / 2017-04-18

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This documentation has been prepared with great care. However, errors can never be entirely ruled out. Please send comments regarding possible errors to the address above.

Table of contents

1 General information 1

1.1 Ordering information ... 1

1.2 Technical specifications ... 2

2 Key aspects of working with separation columns 4

3 Eluent production 8

3.1 Chemicals ... 8

3.2 Production of the standard eluent ... 8

4 Start-up 9

4.1 Connecting and rinsing the guard column ... 9

4.2 Connecting the separation column ... 11

4.3 Conditioning ... 15

5 Applications 17

5.1 Standard chromatogram ... 17

5.2 Effects of temperature ... 19

5.3 Variation of the eluent ... 20

5.4 Bromate in wastewater / drinking water ... 24

5.5 Chromate determination in accordance with US EPA 218.7 ... 25

5.6 Trace analysis in coolant circuit of power plants ... 27

5.7 Multi-component analysis ... 28

5.8 Gradients application for late-eluting ions ... 30

5.9 Aerosol analysis with PILS ... 31

5.10 Denatured bioethanol ... 32

5.11 Ultra-high temperature processed milk ... 33

6 Troubleshooting 35

6.1 Regeneration ... 35

6.2 Decreasing resolution / peak shapes ... 37

6.3 Unstable retention times ... 37

6.4 Unknown peaks ... 38

6.5 Increasing backpressure ... 38

7 Literature **39**

Index **40**

1 General information

This anion separation column is specifically intended for the determination of inorganic and small organic anions using chemical and sequential suppression. The outstanding peak symmetries and high number of plates allow universal use in ion chromatography.

1.1 Ordering information

Table 1 4 mm columns

Order number	Designation
6.1006.550	Metrosep A Supp 5 - 50/4.0
6.1006.510	Metrosep A Supp 5 - 100/4.0
6.1006.520	Metrosep A Supp 5 - 150/4.0
6.1006.530	Metrosep A Supp 5 - 250/4.0

Table 2 2 mm columns

Order number	Designation
6.1006.220	Metrosep A Supp 5 - 150/2.0
6.1006.230	Metrosep A Supp 5 - 250/2.0

Table 3 Guard columns

Order number	Designation
6.1006.500	Metrosep A Supp 5 Guard/4.0
6.1006.540	Metrosep A Supp 5 S-Guard/4.0
6.1006.600	Metrosep A Supp 5 Guard/2.0
6.1006.610	Metrosep A Supp 5 S-Guard/2.0

1.2 Technical specifications

Column material: Polyvinyl alcohol with quaternary ammonium compounds

Particle size: 5 µm

Dimensions

Order number	Dimensions
6.1006.550	50 x 4.0 mm
6.1006.510	100 x 4.0 mm
6.1006.520	150 x 4.0 mm
6.1006.530	250 x 4.0 mm
6.1006.220	150 x 2.0 mm
6.1006.230	250 x 2.0 mm

pH range: 3 to 12

Temperature range: 20 to 60 °C

Recommended standard temperature: 25 °C

Maximum pressure

4 mm	15 MPa (150 bar)
2 mm	20 MPa (200 bar)

Flow rate

Order number	Recommended flow rate	Maximum flow rate
6.1006.550	0.7 mL/min	0.8 mL/min
6.1006.510	0.7 mL/min	0.8 mL/min
6.1006.520	0.7 mL/min	0.8 mL/min
6.1006.530	0.7 mL/min	0.8 mL/min
6.1006.220	0.18 mL/min	0.21 mL/min
6.1006.230	0.18 mL/min	0.21 mL/min

Standard eluent: 3.2 mmol/L of sodium carbonate, 1.0 mmol/L of sodium bicarbonate

Permitted organic additives

- *In the eluent*: 0 to 100% acetonitrile, acetone and methanol
- *In the sample matrix*: 0 to 100% acetonitrile, acetone and methanol

Capacity

Order number	Capacity
6.1006.550	28 µmol (Cl^-)
6.1006.510	56 µmol (Cl^-)
6.1006.520	70 µmol (Cl^-)
6.1006.530	107 µmol (Cl^-)
6.1006.220	18 µmol (Cl^-)
6.1006.230	27 µmol (Cl^-)

Preparation

1. Use a flow gradient to set the column to the standard flow within 2 minutes.
2. Then wait until the baseline is stable.

Storage

Store the column in the standard eluent and, ideally, at a temperature of 4 to 8 °C.

Typical pressure

For columns with a guard column under standard conditions

Order number	Typical pressure
6.1006.550	4.0 ± 2 MPa
6.1006.510	5.2 ± 2 MPa
6.1006.520	8.5 ± 2 MPa
6.1006.530	12.5 ± 2 MPa
6.1006.220	6.5 ± 2 MPa
6.1006.230	10.5 ± 2 MPa

Column housing

Smart column with a chip, called an iColumn, made of PEEK

Application

Determination of inorganic anions and small organic anions with chemical and sequential suppression.

2 Key aspects of working with separation columns

Storage

Once the backpressure in your ion chromatograph has dissipated, remove the column at ambient temperature. Seal the column at both ends using the original stoppers (6.2744.060). Store it in the standard eluent and, ideally, at a temperature between 4 and 8 °C.

Bacterial growth

Bacterial growth significantly affects chromatography and ruins separation columns. A vast array of problems in chromatography are caused by the growth of algae, bacteria and fungi.

In order to prevent bacterial growth, always use fresh eluents, rinsing solutions and regeneration solutions. Do not use any solutions that have not been used for a prolonged period. We recommend cleaning all vessels as follows before filling them:

1. Thoroughly rinse with ultrapure, UV-treated water (> 18.2 MΩ).
2. Swirl a methanol-water or acetone-water mixture around in the vessel.
3. Rinse again with ultrapure water.

If you notice the growth of bacteria or algae despite these precautionary measures, add 5% methanol, acetonitrile or acetone to the eluent. Only do this if you are *not using a membrane suppressor*. Membrane suppressors can be destroyed by organic solvents. The Metrohm Suppressor Module ("MSM", "MSM-HC" and "MSM-LC") are 100% solvent-resistant.

Chemical quality

All chemicals must have a quality of p.a. or puriss. Standard solutions must be intended specifically for ion chromatography.

Chemical stress

Even though specifications may indicate that separation phases do cover a large pH range, this does not mean they are chemically inert. Separation columns last longest when subjected to constant chemical conditions. Never allow a column to dry out and ensure the column is sealed well at all times.

CO_2

Carbon dioxide from the air affects the carbonate/bicarbonate balance in the eluent. The eluent becomes weaker over time. In order to prevent this, always outfit the eluent bottle with CO_2 adsorber material (such as soda lime). Also protect the eluents that have a weak buffer capacity (such as caustic soda eluents) from carbon dioxide.

Eluent bottles

The eluents are usually placed directly on the IC system in special eluent bottles. The bottles must feature an adsorber tube in order to prevent humidity and carbon dioxide from getting into the eluent. Normally, the adsorber tube is filled with a molecular sieve or for sodium hydroxide and carbonate eluents with soda lime (a weak CO_2 adsorber).

Degassing the eluent

In order to prevent bubbles from forming, we recommend degassing the produced eluent before using it in your IC system. To degas the eluent, create a vacuum for approximately ten minutes using a water-jet pump or oil pump. Alternatively, use an ultrasonic bath or work with an eluent degasser.

Filter

Problems that occur in IC systems are usually related to particles. These particles can be introduced from the following sources:

- Bacterial growth
- Unfiltered eluents
- The sample
- The rinsing solution and/or regeneration solution

Minimize this risk by using an aspiration filter (6.2821.090), inline filter (6.2821.120) and guard columns. The filters are part of the basic equipment for Metrohm ion chromatographs and are included in the scope of delivery. We also recommend changing the filters regularly.

Filtering the eluent

All eluents have to be microfiltered (0.45 µm) immediately before use.

Particles

All solutions, samples, regeneration solutions, water and eluents have to be free of particles. Particles clog separation columns over time (column pressure increases). Be especially conscious of ensuring that there are no particles present when producing eluents. The eluent continuously flows through the column at a rate of 500 to 1000 mL per workday compared to about 0.5 mL of the sample solution. Filter or dialyze your sample automatically with one of the Metrohm Inline Sample Preparation techniques (MISP).

Sample preparation cartridges

Sample preparation cartridges are used to prepare critical samples that cannot be injected directly into the separation column. They perform tasks such as removing organic contaminants or neutralizing heavily alkaline or acidic samples. Sample preparation cartridges are consumables that generally cannot be regenerated. Sample preparation cartridges do not replace guard columns, which should be standard on every separation column. As an alternative to sample preparation cartridges, Metrohm Inline Sample Preparation techniques (MISP) are available, such as for neutralizing alkaline samples.

Pulsation absorber

We recommend always using a pulsation absorber. Polymethacrylate columns and polyvinyl alcohol columns in particular must be protected from

the brief pressure surges that inevitably occur when switching the valves. Using the pulsation absorber (6.2620.150) already built into the Metrohm ion chromatographs provides this protection.

Mechanical stress

Mechanical loads on the column should be avoided. For example, the column impacting a hard surface can cause a break or gap in the column packing (separation phase material). This affects the chromatography results. The column would be irreparably damaged as a result.

Regenerating separation columns

If separation columns are operated with clean eluents and filled with samples free of particles, you can expect the column to have a long service life. This means regenerating the column is not required and, after a multitude of injections, no longer possible.

If the pressure in the column increases unexpectedly despite this or the separation performance decreases, the regeneration steps specified for every column can be carried out. Generally it is important to keep in mind that the regeneration takes place outside the analytical line. Connect the separation column to the pump directly. Route the regeneration solution through the column directly into a waste container. Before reinstalling the separation column, it must be properly rinsed with fresh eluent.

Shutting down the ion chromatograph

If you will not be working with the ion chromatograph for a prolonged period (> 1 week), we recommend removing the separation column and sealing it with the stoppers provided. Rinse the ion chromatograph, including all three suppressor chambers, with methanol/water (1:4). Store the separation column in the medium indicated on the column leaflet and ideally at a temperature between 4 and 8 °C if not specified otherwise.

If you are working with sodium hydroxide eluents, rinse the base out of the ion chromatograph with water before it has been left standing for two days.

When you return the instrument to operation, rinse the ion chromatograph with fresh eluent. Bring the separation column back up to the ambient temperature before you install it. Then increase the temperature if necessary.

Fun

Ion chromatography should be fun and should not stress you out. Metrohm puts all its work into ensuring you can work reliably with your IC systems with minimal maintenance, servicing and costs. Metrosep separation columns embody the attributes of quality, long service life and excellent results.

Environmental protection

A significant advantage of ion chromatography is that most of the work involves aqueous media. As a result, the chemicals used in ion chromatography are largely non-toxic and do not impact the environment. However,

if you are working with acids, bases, organic solvents or heavy metal standards, dispose of them properly after use.

Guard columns

Guard columns are used to protect separation columns. We strongly recommend their use. They normally contain the same stationary phase also used in the separation columns, but the quantity is significantly reduced to avoid impacting the chromatography. Guard columns remove critical contaminants that could react with column material; they also effectively remove particles and bacterial contaminants. Replace the guard column in the following cases:

- If the backpressure in the system increases
- If the chromatography results deteriorate

We recommend using 3 to 4 guard columns over the service life of the separation column.

Guard columns are available for all Metrosep separation columns.

Water quality

Aqueous media is used in most work involving ion chromatography. This means water quality is a critical factor for good chromatography. If the water quality is inadequate, the results will be as well. In addition, there is a risk of damaging instruments and separation columns when using water with inadequate quality. The ultrapure water being used should have a specific resistivity greater than 18.2 MΩ·cm and should be free of particles. Therefore, we recommend filtering the water using a 0.45 µm filter and treating it with UV light. Modern ultrapure water systems for laboratory use ensure this level of water quality (Type I).

3 Eluent production

We recommend choosing a high level of purity for chemicals for both standard production and eluent production.

3.1 Chemicals

Recommended chemicals

- Sodium carbonate, Na_2CO_3, 99.95-100.5%
  Sigma Aldrich order number: 223484
- Sodium bicarbonate, $NaHCO_3$, 99.7-100.3%
  Sigma Aldrich order number: S6014
- Ultrapure water of type I (see ASTM D1193)
  Resistance > 18 MΩ·cm (25°C)
  TOC < 10 µg/L

3.2 Production of the standard eluent

Proceed as follows to produce 2 L of standard eluent with 3.2 mmol/L of sodium carbonate and 1.0 mmol/L of sodium bicarbonate:

Producing 2 L of standard eluent

1
- Prerinse the eluent bottle with ultrapure water several times.
- Set out 2 L of ultrapure water.

2 If the eluent is not degassed using an eluent degasser:

- Degas the ultrapure water for the eluent for 5 to 10 minutes using a vacuum pump. This avoids the problems with air bubbles in a high-pressure pump.

3 Weigh out and add the following quantities of salts:

- Sodium carbonate: 678 mg
- Sodium bicarbonate: 168 mg

These eluents (3.2 mmol/L of sodium carbonate and 1.0 mmol/L of sodium bicarbonate) and sequential suppression can be used to achieve background conductivity of < 1 µS/cm. The noise level is typically less than 0.2 nS/cm.

4 Start-up

4.1 Connecting and rinsing the guard column

Guard columns protect separation columns and significantly increase their service life. The guard columns available from Metrohm are either actual guard columns or guard column cartridges used together with a cartridge holder. The process of installing a guard column cartridge into the corresponding holder is described in the guard column leaflet.

NOTE

Metrohm recommends always working with guard columns. They protect the separation columns and can be replaced regularly as needed.

NOTE

Information regarding which guard column is suitable for your separation column can be found in the **Metrohm Column Program** (which is available from your Metrohm representative), the leaflet provided along with your separation column or the product information about the separation column at *http://www.metrohm.com* (Ion Chromatography product area), or it can be obtained directly from your representative.

CAUTION

New guard columns are filled with a solution and sealed with stoppers or caps on both sides.

Before inserting the guard column, ensure that this solution can be mixed with the eluent being used (follow the information provided by the manufacturer).

NOTE

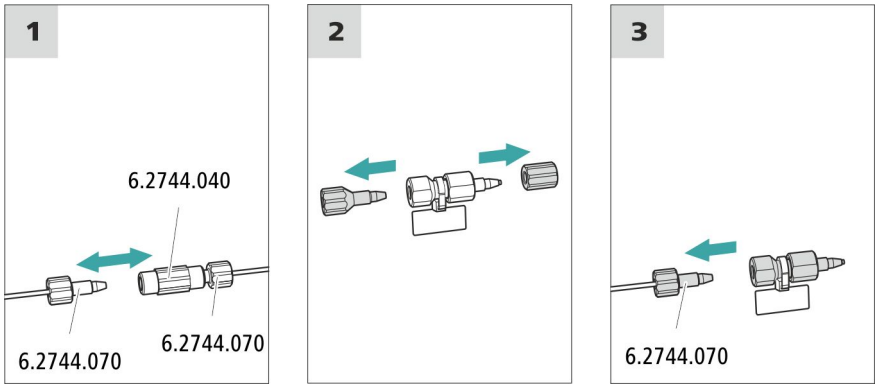
The guard column may not be connected until after the instrument has already been put into operation once . The guard column and the separation column have to be replaced by a coupling (6.2744.040) until then.

Accessories

For this step, you need the following accessories:

- Guard column (suitable for separation column)

Connecting the guard column



1 Removing the coupling

Remove the coupling (6.2744.040) installed between the column inlet capillary and the column outlet capillary for the initial start-up.

2 Preparing the guard column

- Remove the stopper and the sealing cap from the guard column.

3 Connecting the guard column

CAUTION

When inserting the guard column, ensure that it is inserted correctly based on the marked flow direction (if specified).

- Fasten the inlet of the guard column to the column inlet capillary using a short pressure screw (6.2744.070).

- If the guard column is connected to the separation column using a connection capillary, fasten this connection capillary to the guard column outlet with a pressure screw.

Rinsing the guard column

1 Rinsing the guard column

- Place a beaker under the guard column's outlet.
- Start manual control in MagIC Net and select the high-pressure pump: **Manual ▸ Manual control ▸ Pump**
  - **Flow**: **in accordance with column leaflet**
  - **On**
- Rinse the guard column with eluent for approx. 5 minutes.
- Stop the high-pressure pump in the manual control in MagIC Net again: **Off**.

4.2 Connecting the separation column

The smart separation column (iColumn) is the heart of ion chromatographic analysis. It separates the different components according to their interactions with the column. Metrohm separation columns are equipped with a chip where their technical specifications and history (start-up, operating hours, injections etc) are stored.

NOTE

Information regarding which separation column is suitable for your application can be found in the **Metrohm Column Program**, the product information for the separation column or it can be obtained through your representative.

You can find product information for your separation column at *http://www.metrohm.com* in the Ion Chromatography product area.

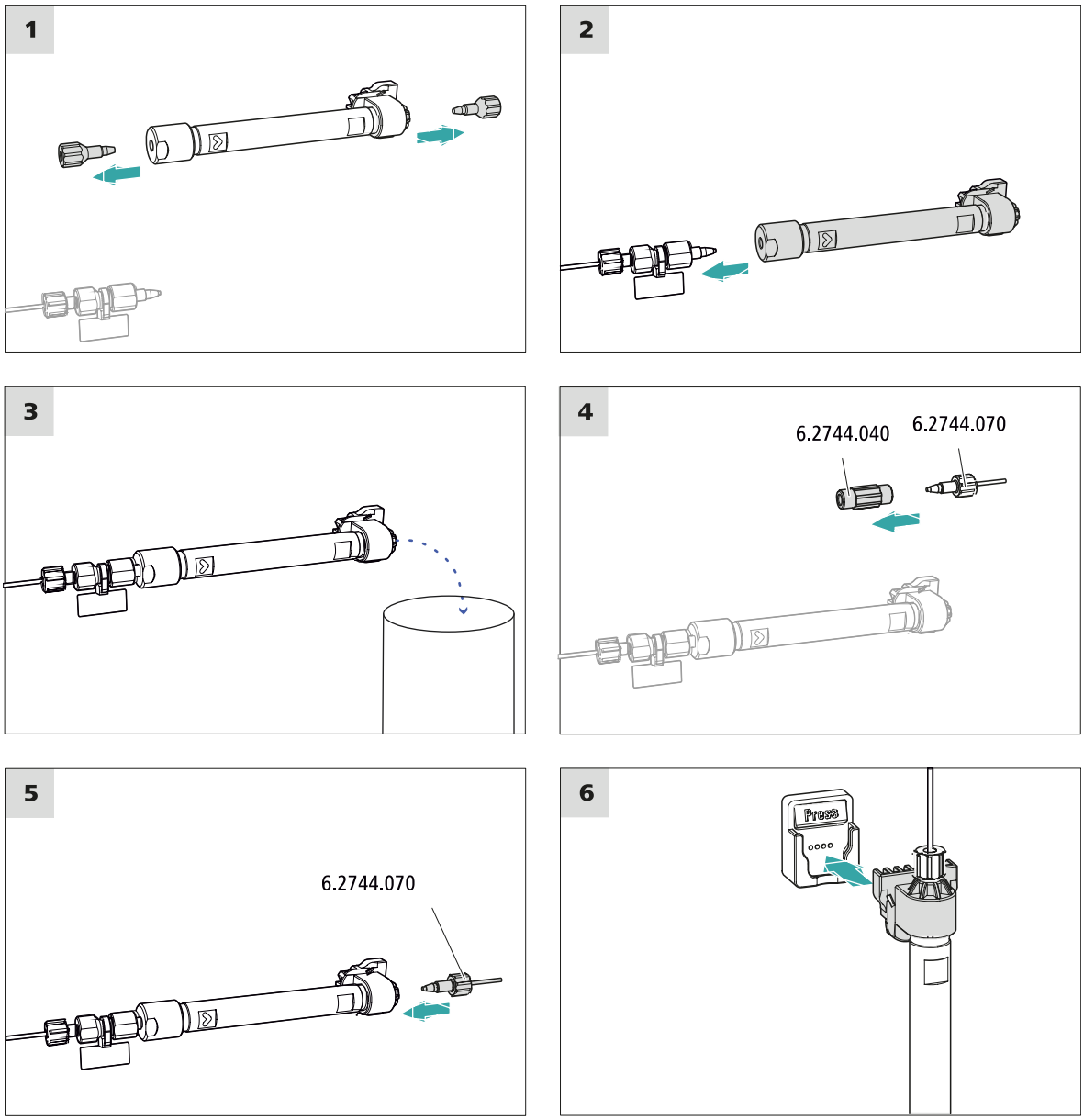
A test chromatogram and a leaflet accompanies every column. Detailed information on special IC applications can be found in the corresponding "**Application Bulletins**" or "**Application Notes**". You can find these online at *http://www.metrohm.com* in the Applications area or request them from your responsible Metrohm representative free of charge.

CAUTION

New separation columns are filled with a solution and sealed with stoppers on both sides. Before inserting the column, ensure that this solution can be mixed with the eluent being used (follow the information provided by the manufacturer).

NOTE

Connect the separation column only after the initial start-up of the instrument. Until that point, insert a coupling (6.2744.040) instead of the guard column and separation column.



Connecting the separation column

1 Removing the stoppers

- Remove the stoppers from the separation column.

2 Installing the inlet of the separation column

CAUTION

When inserting the column, ensure that it is inserted correctly based on the marked flow direction.

There are three options:

- Attach the column inlet directly onto the guard column or,
- if the guard column is connected to the separation column using a connection capillary: Connect the column inlet to the guard column outlet capillary using a PEEK pressure screw (6.2744.070) or,
- if no guard column is used (not recommended): Connect the column inlet capillary to the inlet of the separation column using a short pressure screw (6.2744.070).

3 Rinsing the separation column

- Place a beaker under the outlet of the separation column.
- Start manual control in MagIC Net and select the high-pressure pump: **Manual ▸ Manual control ▸ Pump**
  - **Flow**: Increase gradually up to the flow rate recommended in the column leaflet.
  - **On**
- Rinse the separation column with eluent for approx. 10 minutes.
- Stop the high-pressure pump in the manual control in MagIC Net again: **Off**.

4 Removing the coupling

- Remove the coupling (6.2744.040) from the column outlet capillary.

5 Installing the outlet of the separation column

- Fasten the column outlet capillary to the column outlet using a short PEEK pressure screw (6.2744.070).

6 Inserting the separation column

- Insert the separation column with the chip into the column holder until you hear it snap in place.

The separation column is now detected by MagIC Net.

4.3 Conditioning

In the following cases, the system must be conditioned with eluent until a stable baseline has been reached:

- After installation
- After each time the instrument is switched on
- After each eluent change

NOTE

The conditioning time can lengthen considerably if the composition of the eluent is modified.

Conditioning the system

1 Preparing the software

CAUTION

Ensure that the configured flow rate is not higher than the flow rate permitted for the corresponding column (refer to the column leaflet and chip data record).

- Start the **MagIC Net** computer program.
- Open the **Equilibration** tab in MagIC Net: **Workplace ▸ Run ▸ Equilibration**.
- Select (or create) a suitable method.
  Also see: *MagIC Net Tutorial* and online help.

2 Preparing the instrument

- Ensure that the column is inserted correctly in accordance with the flow direction marked on the sticker (arrow has to point in the direction of flow).
- Ensure that the eluent aspiration tubing is immersed in the eluent and that there is enough eluent in the eluent bottle.

3 Starting equilibration

- Start the equilibration in MagIC Net: **Workplace ▸ Run ▸ Equilibration ▸ Start HW**.

- Visually inspect whether all capillaries and their connections from the high-pressure pump to the detector are leak-tight. If eluent is leaking out anywhere, tighten the corresponding pressure screw further, or loosen the pressure screw, check the end of the capillary and shorten it using the capillary cutter if necessary and retighten the pressure screw.

4 Conditioning the system

Continue rinsing the system with eluent until the desired stability level for the baseline has been attained .

The instrument is now ready for measuring samples.

5 Applications

5.1 Standard chromatogram

Sample preparation: -

Detection: Conductivity

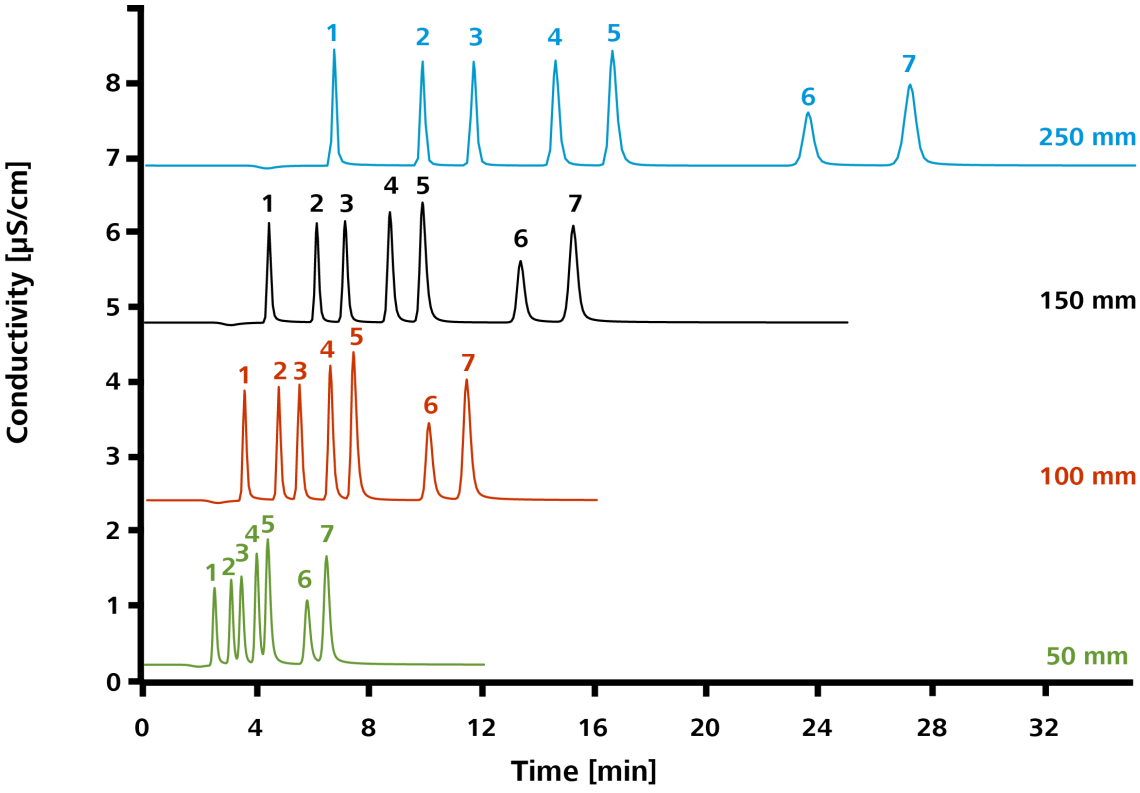
Suppression: 4 mm: Sequential suppression with MSM and MCS
2 mm: Chemical suppression with MSM-LC

Temperature: 30 °C

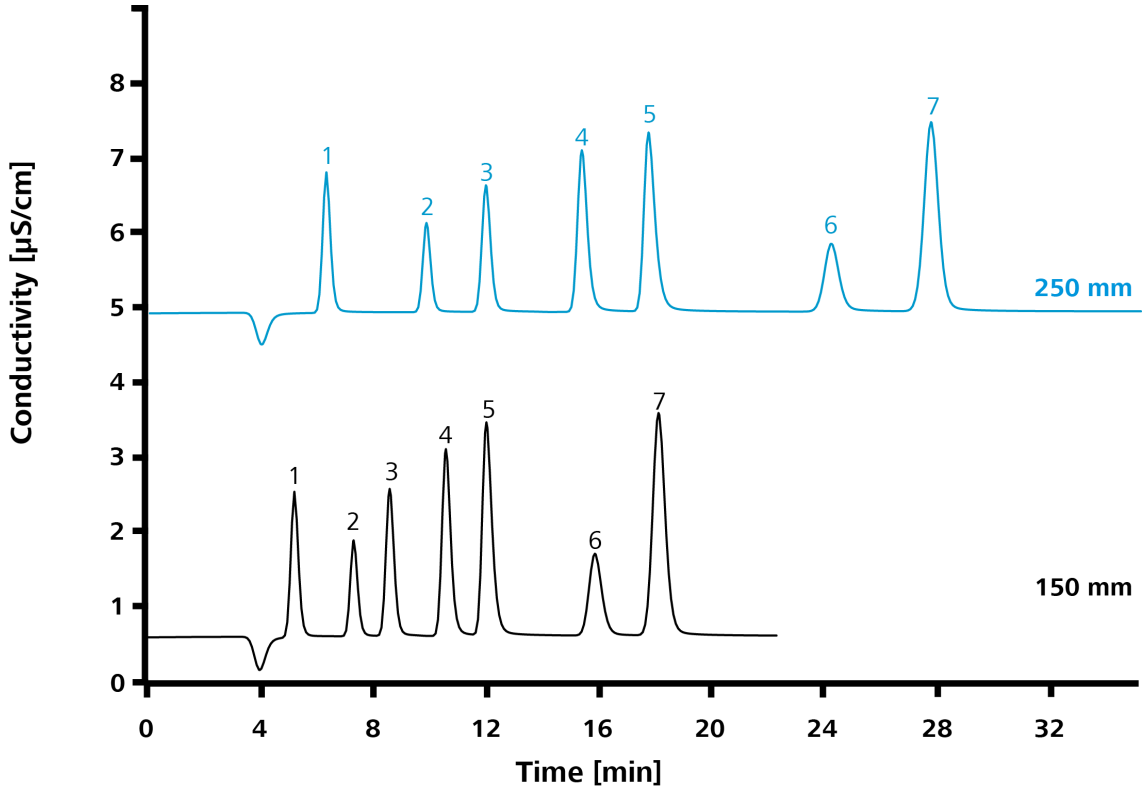
Loop: 4 mm: 20 µL
2 mm: 10 µL

Flow rate: 4 mm: 0.7 mL/min
2 mm: 0.18 mL/min

Eluent: 3.2 mmol/L of Na_2CO_3, 1.0 mmol/L of $NaHCO_3$



	A Supp 5 - XXX/4.0
1	2 mg/L of fluoride
2	3 mg/L of chloride
3	5 mg/L of nitrite
4	10 mg/L of bromide
5	10 mg/L of nitrate
6	10 mg/L of phosphate
7	10 mg/L of sulfate



	A Supp 5 - X50/2.0
1	2 mg/L of fluoride
2	2 mg/L of chloride
3	5 mg/L of nitrite
4	10 mg/L of bromide
5	10 mg/L of nitrate
6	10 mg/L of phosphate
7	10 mg/L of sulfate

5.2 Effects of temperature

Column: Metrosep A Supp 5 - 250/2.0

Sample preparation: –

Detection: Conductivity

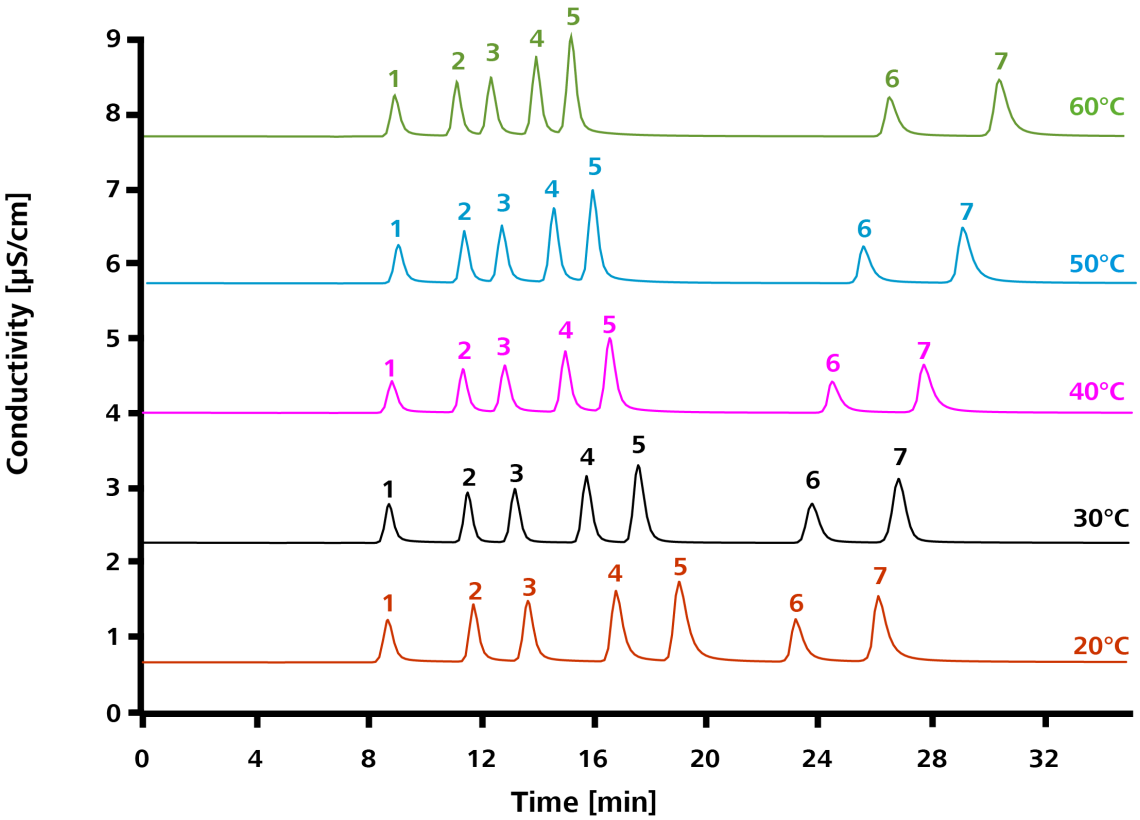
Suppression: Sequential suppression with MSM-LC and MCS

Temperature: 20 to 60 °C

Loop: 10 µL

Flow rate: 0.18 mL/min

Eluent: 3.2 mmol/L of Na_2CO_3, 1.0 mmol/L of $NaHCO_3$



	A Supp 5 - 250/2.0
1	2 mg/L of fluoride
2	3 mg/L of chloride

	A Supp 5 - 250/2.0
3	5 mg/L of nitrite
4	10 mg/L of bromide
5	10 mg/L of nitrate
6	10 mg/L of phosphate
7	10 mg/L of sulfate

5.3 Variation of the eluent

Variation with constant Na_2CO_3/$NaHCO_3$ ratio

Column: Metrosep A Supp 5 - 150/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Chemical suppression with MSM

Temperature: Ambient temperature

Loop: 20 µL

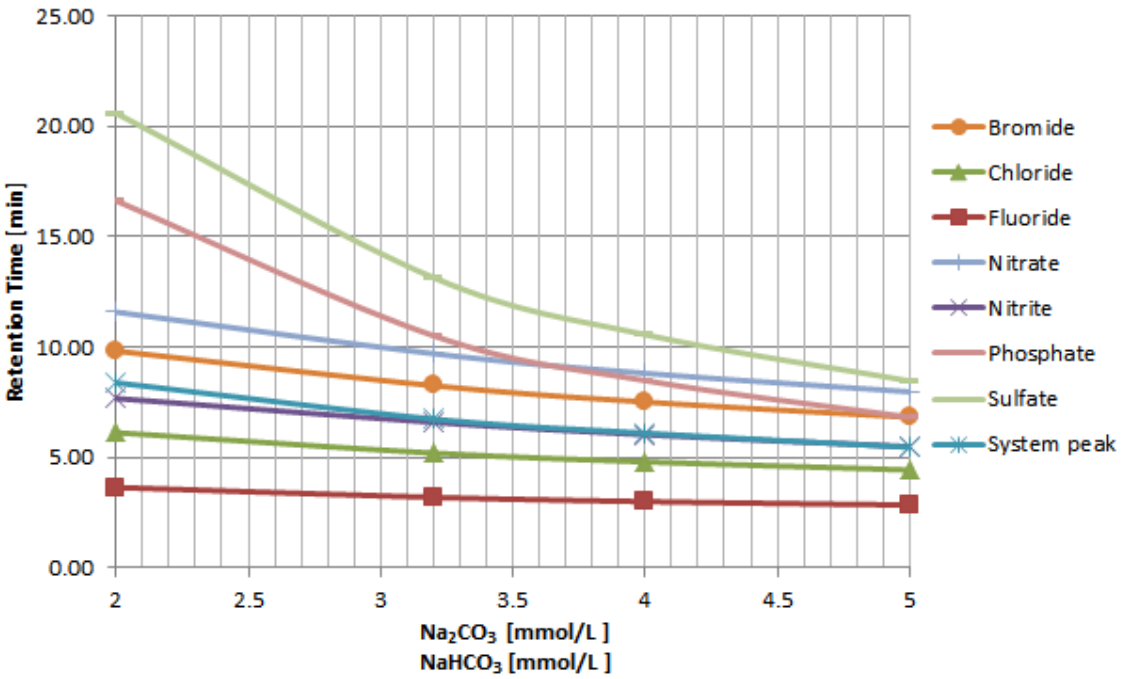
Flow rate: 0.7 mL/min

Eluent: A) 2.0 mmol/L of Na_2CO_3, 2.0 mmol/L of $NaHCO_3$

B) 3.2 mmol/L of Na_2CO_3, 3.2 mmol/L of $NaHCO_3$

C) 4.0 mmol/L of Na_2CO_3, 4.0 mmol/L of $NaHCO_3$

D) 5.0 mmol/L of Na_2CO_3, 5.0 mmol/L of $NaHCO_3$



The retention time becomes shorter with increasing Na_2CO_3/$NaHCO_3$ concentration. In particular, the times for the polyvalent anions phosphate and sulfate are significantly reduced. At 4.0 mmol/L of Na_2CO_3 / 4.0 mmol/L of $NaHCO_3$, phosphate elutes before nitrate; at 5.0 mmol/L of Na_2CO_3 / 5.0 mmol/L of $NaHCO_3$, it coelutes with bromide. The resolution between the system peak and nitrite decreases as concentration increases.

$NaHCO_3$ variation with constant Na_2CO_3

Column: Metrosep A Supp 5 - 150/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Chemical suppression with MSM

Temperature: Ambient temperature

Loop: 20 µL

Flow rate: 0.7 mL/min

Eluent: A) 3.2 mmol/L of Na_2CO_3, 0 mmol/L of $NaHCO_3$

B) 3.2 mmol/L of Na_2CO_3, 0.5 mmol/L of $NaHCO_3$

C) 3.2 mmol/L of Na_2CO_3, 1.0 mmol/L of $NaHCO_3$

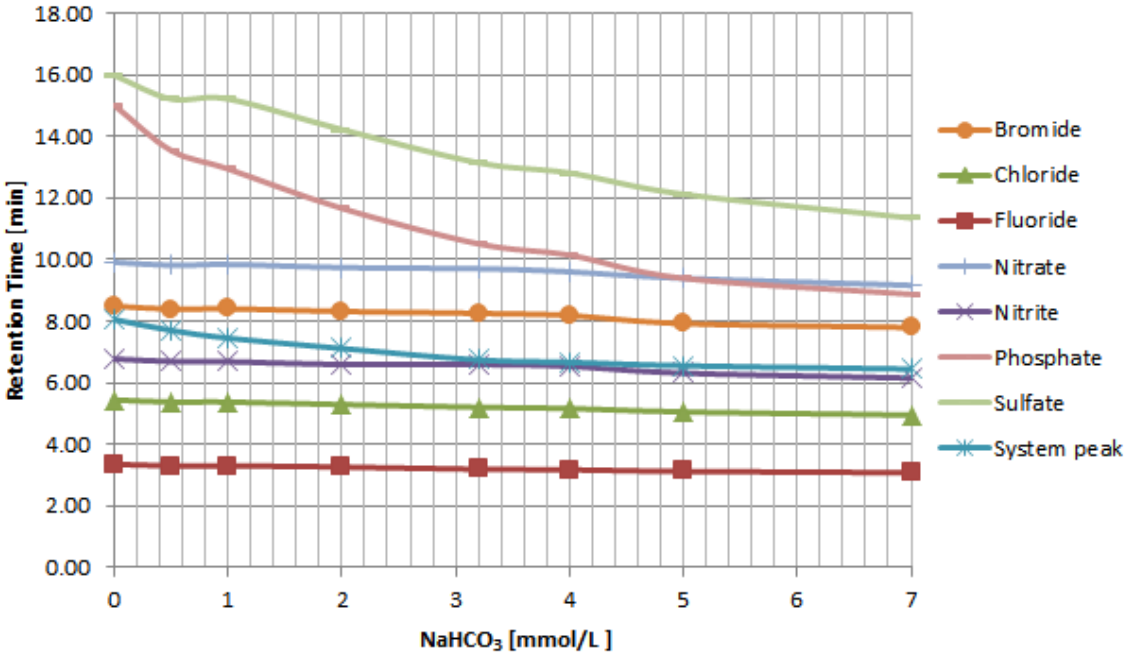
D) 3.2 mmol/L of Na_2CO_3, 2.0 mmol/L of $NaHCO_3$

E) 3.2 mmol/L of Na_2CO_3, 3.2 mmol/L of $NaHCO_3$

F) 3.2 mmol/L of Na_2CO_3, 4.0 mmol/L of $NaHCO_3$

G) 3.2 mmol/L of Na_2CO_3, 5.0 mmol/L of $NaHCO_3$

H) 3.2 mmol/L of Na_2CO_3, 7.0 mmol/L of $NaHCO_3$



For fluoride, chloride, nitrite, bromide and nitrate, almost no decrease in retention time is observed as $NaHCO_3$ concentration increases, unlike for the system peak, phosphate and sulfate. As $NaHCO_3$ concentration increases, the system peak shifts from bromide to nitrite. The higher the $NaHCO_3$ content, the lower the pH, resulting in a shifted disassociation balance for phosphate and sulfate. Both move much closer to monovalent ions.

Na_2CO_3 variation with constant $NaHCO_3$

Column:	Metrosep A Supp 5 - 150/4.0
Sample preparation:	–
Detection:	Conductivity
Suppression:	Chemical suppression with MSM
Temperature:	Ambient temperature
Loop:	20 µL

Flow rate: 0.7 mL/min

Eluent: A) 0.5 mmol/L of Na_2CO_3, 5.0 mmol/L of $NaHCO_3$

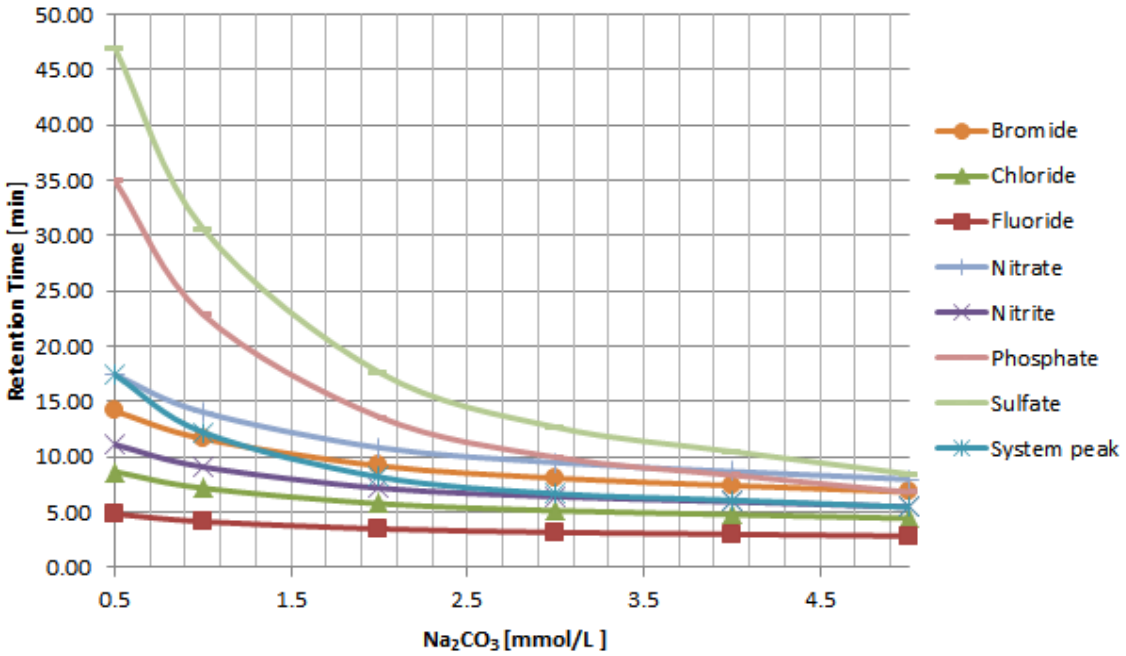
B) 1 mmol/L of Na_2CO_3, 5.0 mmol/L of $NaHCO_3$

C) 2 mmol/L of Na_2CO_3, 5.0 mmol/L of $NaHCO_3$

D) 3 mmol/L of Na_2CO_3, 5.0 mmol/L of $NaHCO_3$

E) 4 mmol/L of Na_2CO_3, 5.0 mmol/L of $NaHCO_3$

F) 5 mmol/L of Na_2CO_3, 5.0 mmol/L of $NaHCO_3$



For fluoride, chloride, nitrite, bromide and nitrate, a decrease in retention time is observed as Na_2CO_3 concentration increases. In turn, there is a significant reduction for the system peak, phosphate and sulfate. As Na_2CO_3 concentration increases, the system peak shifts from nitrate to bromide to nitrite.

Variation of the organic modifier acetone

Column: Metrosep A Supp 5 - 150/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Chemical suppression with MSM

Temperature: Ambient temperature

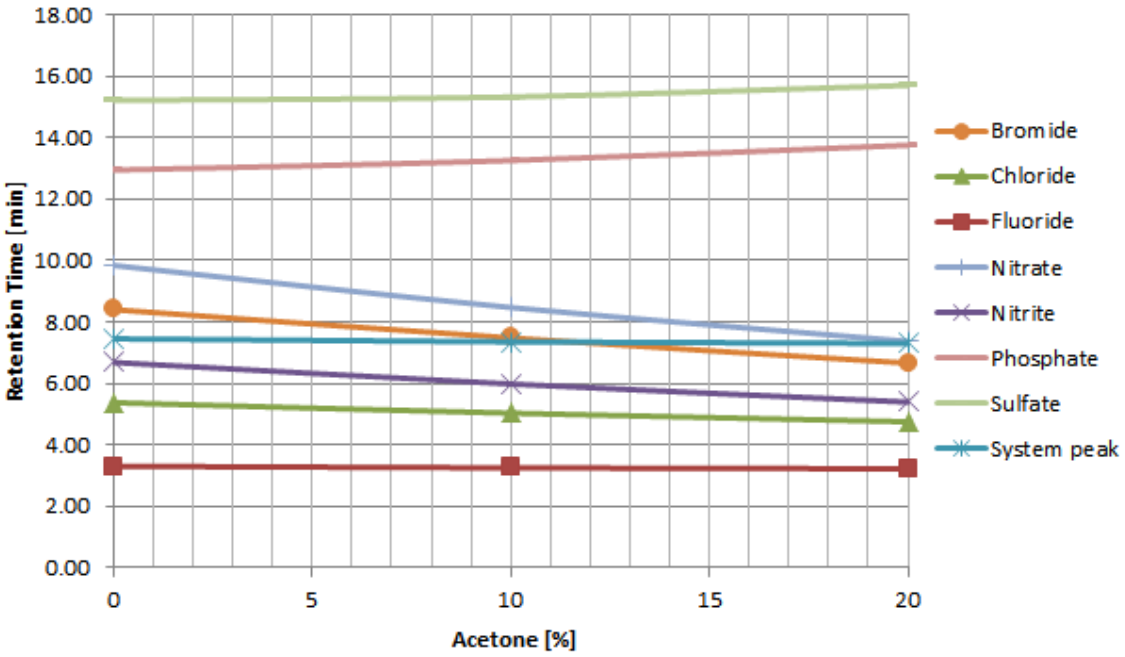
Loop: 20 µL

Flow rate: 0.7 mL/min

Eluent: A) 3.2 mmol/L of Na_2CO_3, 1.0 mmol/L of $NaHCO_3$, 0% acetone

B) 3.2 mmol/L of Na_2CO_3, 1.0 mmol/L of $NaHCO_3$, 10% acetone

C) 3.2 mmol/L of Na_2CO_3, 1.0 mmol/L of $NaHCO_3$, 20% acetone



Acetone does not have any effect on the retention time of the system peak. Fluoride, chloride, nitrite and nitrate move in the opposite direction of phosphate and sulfate as acetone content increases. The system peak coelutes with bromide at 10% acetone and with nitrate at 20% acetone.

5.4 Bromate in wastewater / drinking water

Column: Metrosep A Supp 5 - 250/4.0

Sample preparation: –

Detection: Conductivity

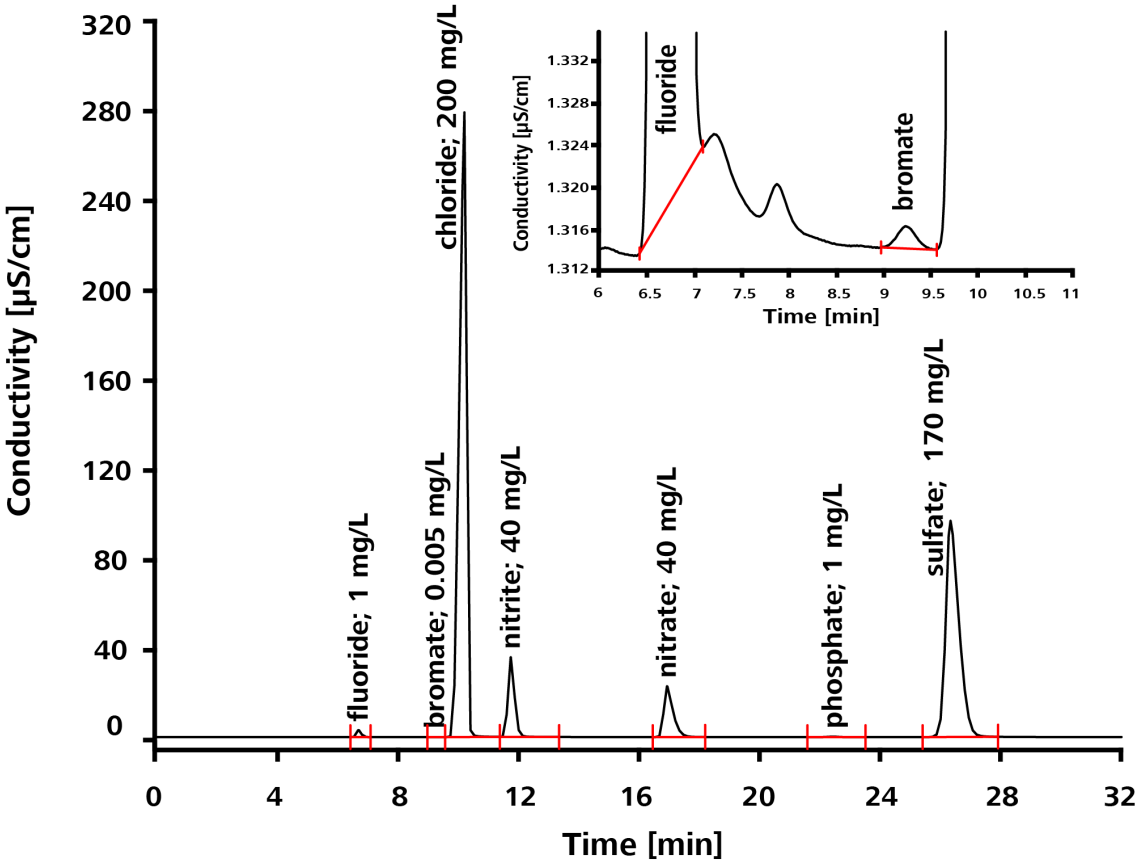
Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.7 mL/min

Eluent: 3.2 mmol/L of Na_2CO_3, 1.0 mmol/L of $NaHCO_3$



5.5 Chromate determination in accordance with US EPA 218.7

Column: Metrosep A Supp 5 - 150/4.0

Sample preparation: Adjust the pH of the sample using 1 mL of buffer solution (33 g/L of $(NH_4)_2SO_4$) per 100 mL

Postcolumn reagent: 2 mmol/L of 1,5-Diphenylcarbazide

Detection: VIS detection at 530±21 nm (650±21 nm reference channel)

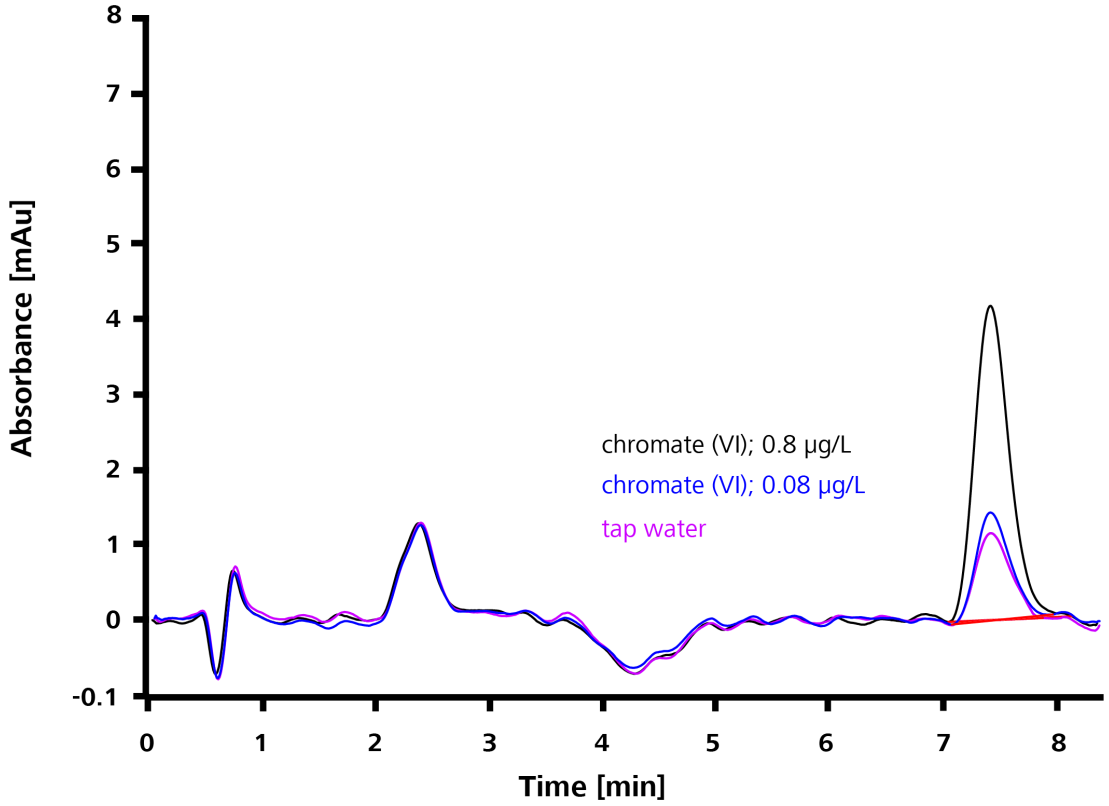
Suppression: –

Temperature: 45 °C

Loop: 1325 µL

Flow rate: 0.7 mL/min

Eluent: 12.8 mmol/L of Na_2CO_3, 4.0 mmol/L of $NaHCO_3$, 2.5 g/L of $(NH_4)_2SO_4$



	Concentration		Recovery [%]
N = 3	**Mean value [µg/L]**	**RSD [%]**	**Mean value**
Drinking water…	0.33	0.71	–
…with 0.08 µg/L chromate spike	0.41	0.41	99.9
…with 0.8 µg/L chromate spike	1.13	0.23	99.8

5.6 Trace analysis in coolant circuit of power plants

Column: Metrosep A Supp 5 - 150/4.0

Sample preparation: Inline preconcentration

Detection: Conductivity

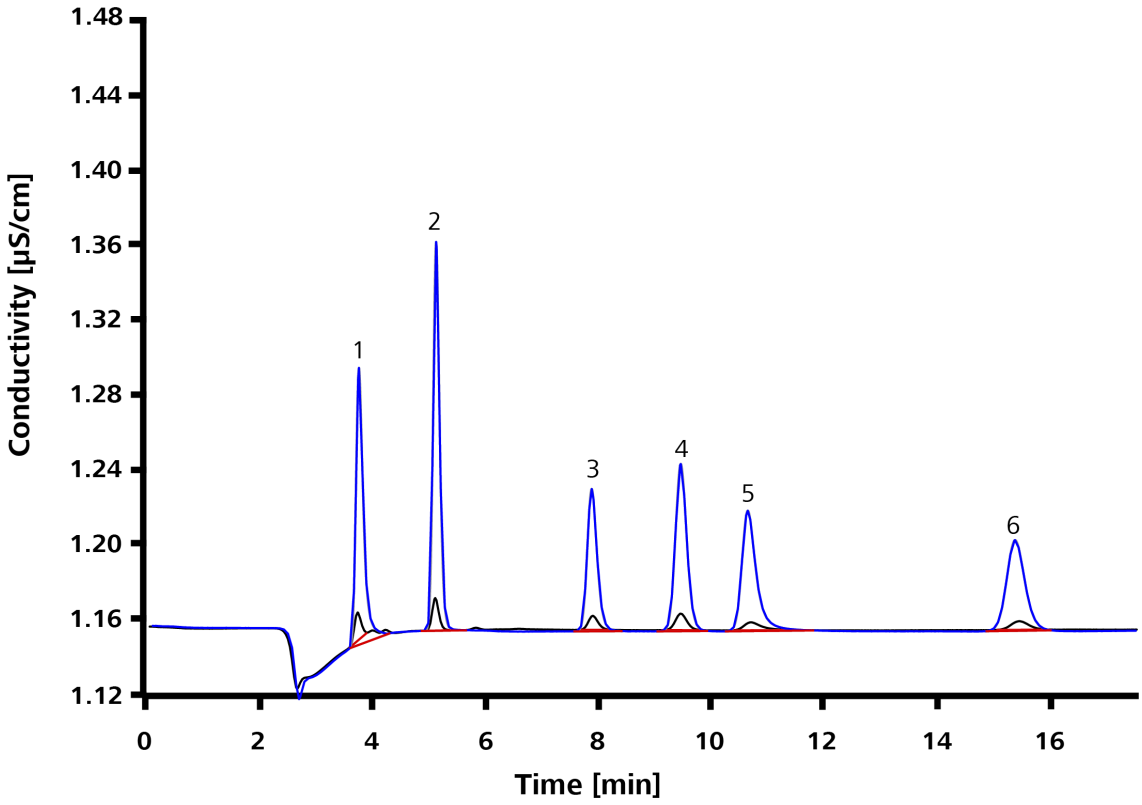
Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

Loop: 2000 µL with preconcentration at A PCC HC

Flow rate: 0.8 mL/min

Eluent: 4.8 mmol/L of Na_2CO_3, 1.5 mmol/L of $NaHCO_3$



	Black	Blue
1	0.1 µg/L of fluoride	1 µg/L of fluoride
2	0.2 µg/L of chloride	2 µg/L of chloride

	Black	Blue
3	0.2 µg/L of nitrate	2 µg/L of nitrate
4	0.2 µg/L of sulfate	2 µg/L of sulfate
5	0.2 µg/L of oxalate	2 µg/L of oxalate
6	0.2 µg/L of chromate	2 µg/L of chromate

5.7 Multi-component analysis

4 mm column

Column: Metrosep A Supp 5 - 250/4.0

Sample preparation: –

Detection: Conductivity

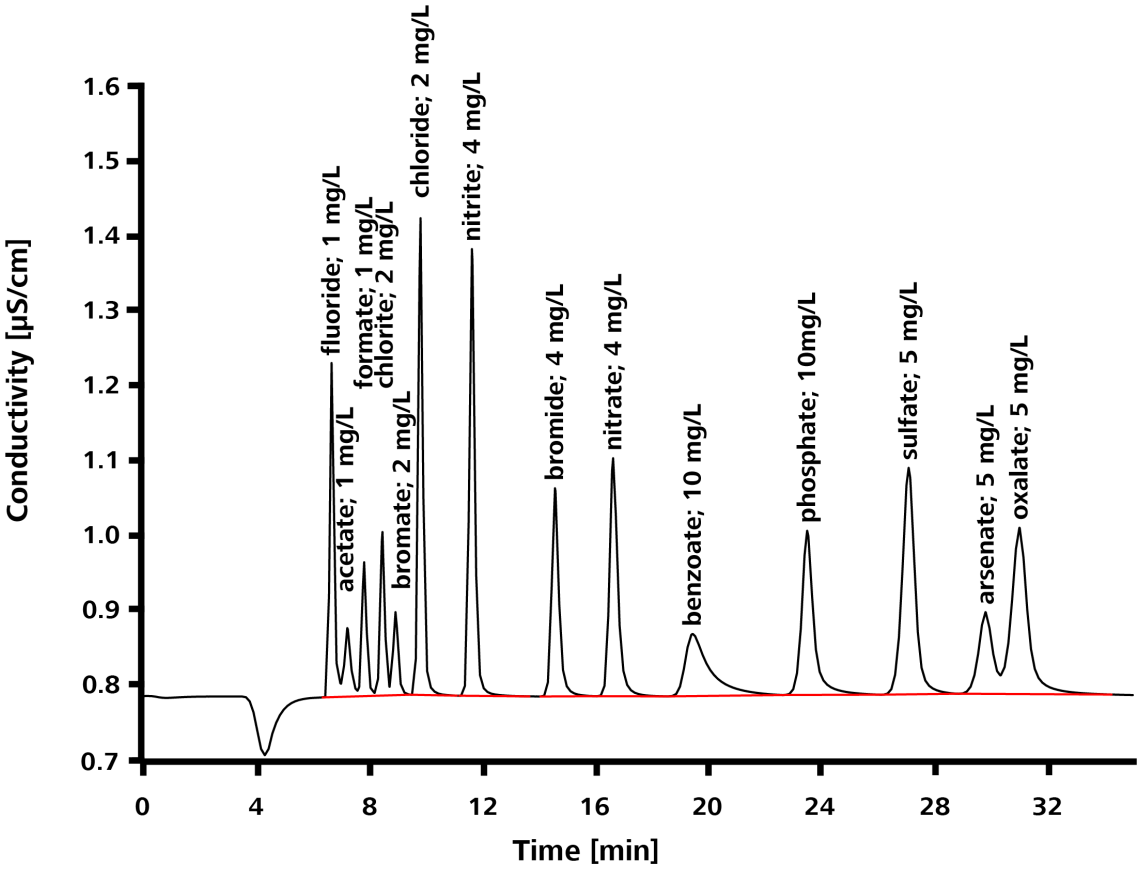
Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

Loop: 5 µL

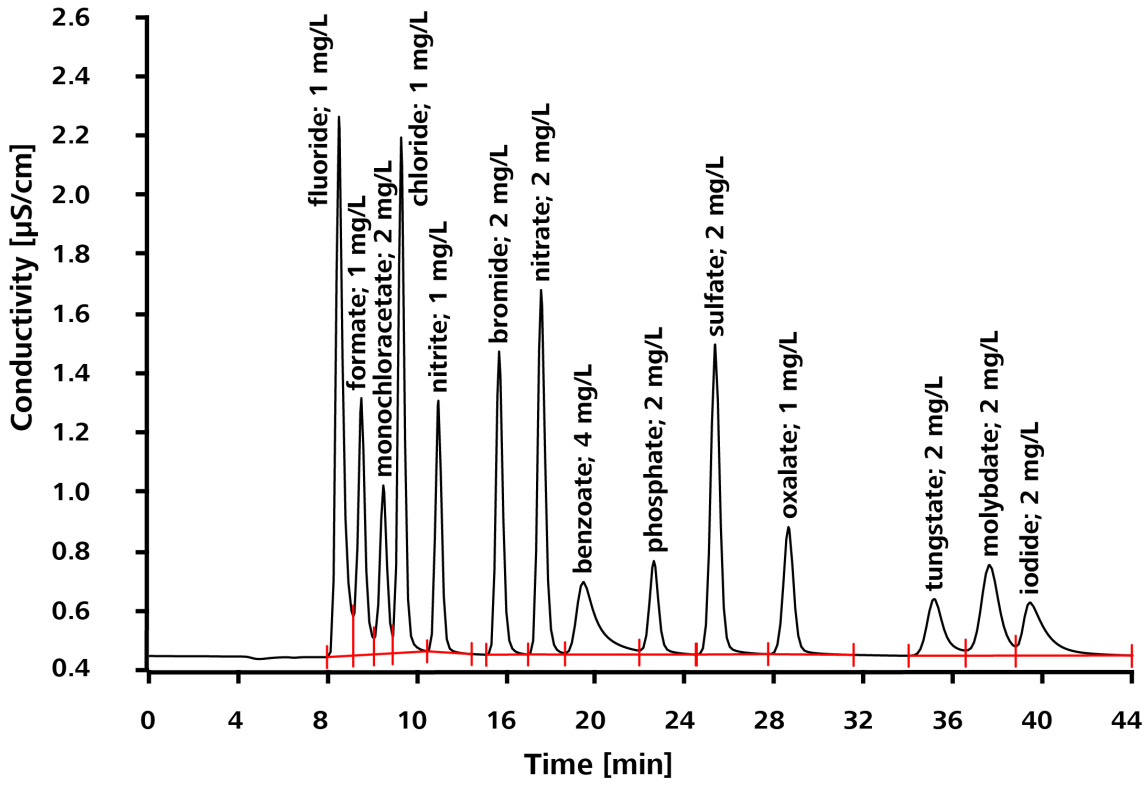
Flow rate: 0.7 mL/min

Eluent: 3.2 mmol/L of Na_2CO_3, 1.0 mmol/L of $NaHCO_3$



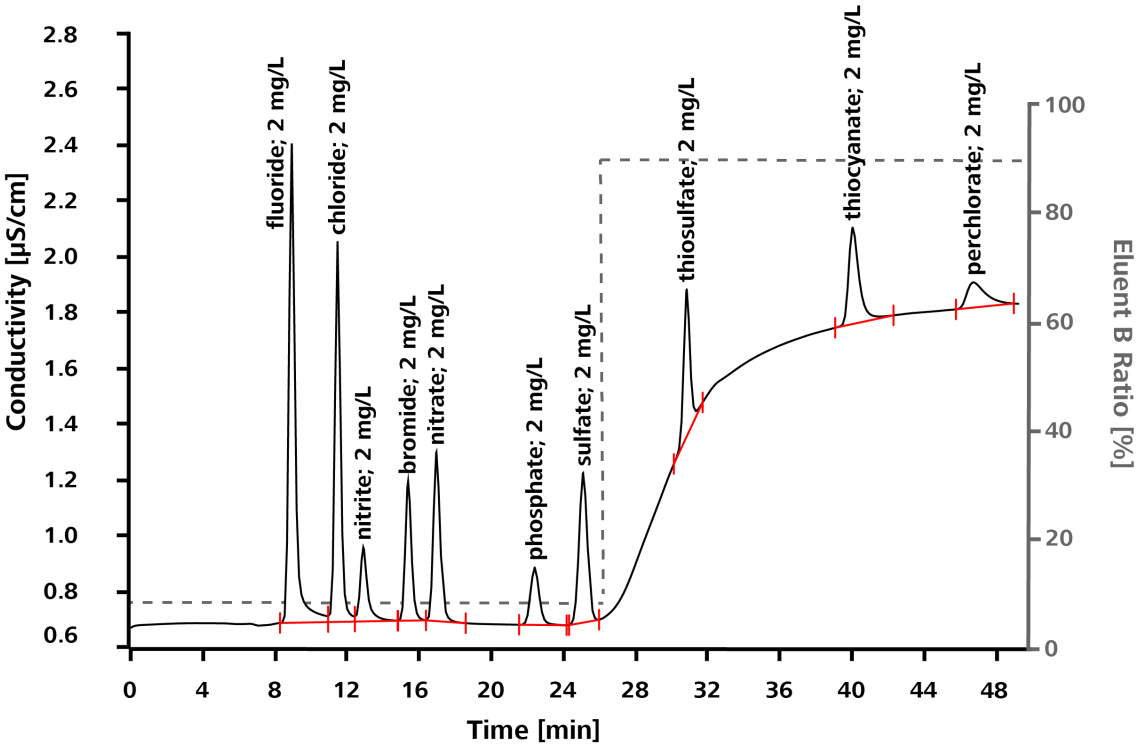
2-mm column

Column:	Metrosep A Supp 5 - 250/2.0
Sample preparation:	–
Detection:	Conductivity
Suppression:	Sequential suppression with MSM and MCS
Temperature:	30 °C
Loop:	10 µL
Flow rate:	0.18 mL/min
Eluent:	3.2 mmol/L of Na_2CO_3, 1.0 mmol/L of $NaHCO_3$



5.8 Gradients application for late-eluting ions

Column	Metrosep A Supp 5 - 250/2.0
Sample preparation:	-
Detection:	Conductivity
Suppression:	Sequential suppression with MSM and MCS
Temperature:	30 °C
Loop:	5 µL
Flow rate:	0.18 mL/min
Eluent:	Eluent A: 2% acetone in ultrapure water Eluent B: 3.2 mmol/L of Na_2CO_3, 1.0 mmol/L of $NaHCO_3$



5.9 Aerosol analysis with PILS

Column: Metrosep A Supp 5 - 100/4.0

Sample preparation: PM_{10} aerosols, i.e. air particles < 10 µm, are selected using a cyclone. The aerosols are put into an aqueous solution with an internal standard (LiBr) using a particle into liquid sampler (PILS).

Use a particle into liquid sampler (PILS) to collect PM_{10} (particulate matter < 10 µm) using an internal lithium bromide standard

Detection: Conductivity

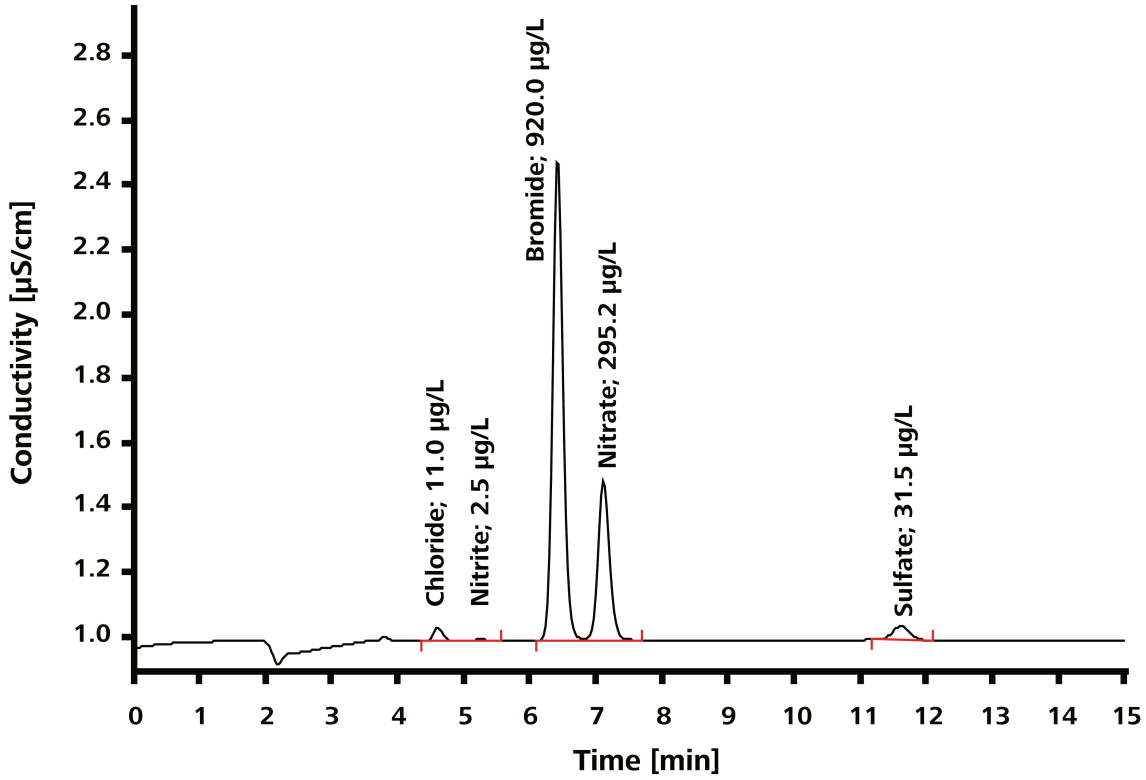
Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

Loop: 250 µL

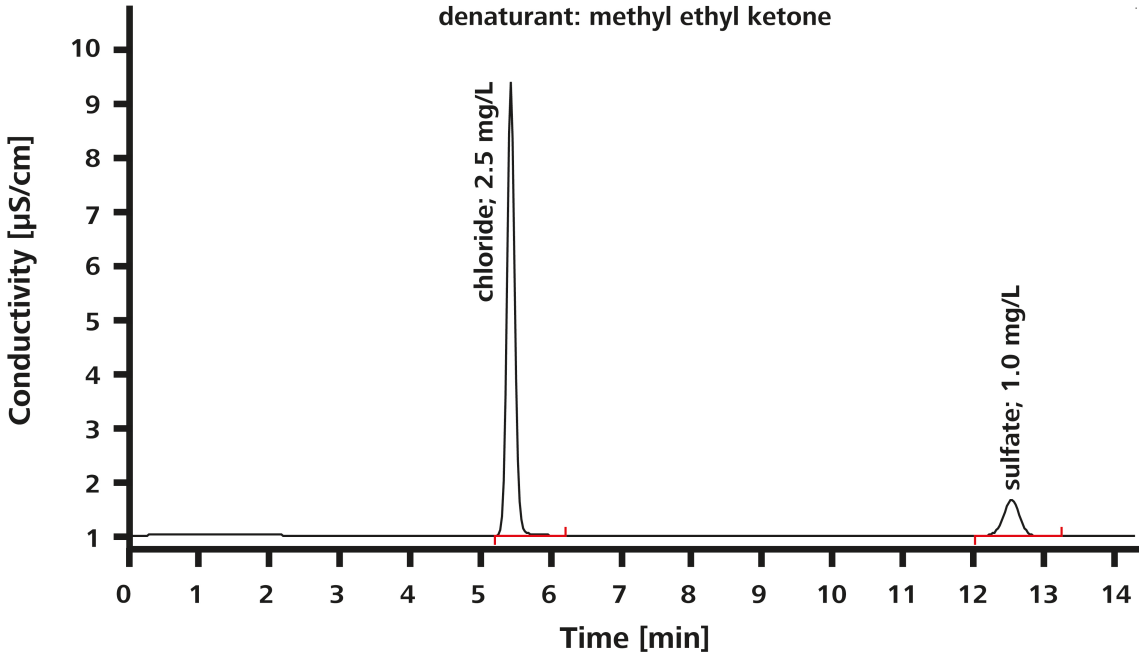
Flow rate: 0.7 mL/min

Eluent: 3.2 mmol/L of Na_2CO_3, 1.0 mmol/L of $NaHCO_3$



5.10 Denatured bioethanol

Column:	Metrosep A Supp 5 - 150/4.0
Sample preparation:	Direct injection in accordance with ASTM D 7319
Detection:	Conductivity
Suppression:	Sequential suppression with MSM and MCS
Temperature:	35 °C
Loop:	10 µL
Flow rate:	0.7 mL/min
Eluent:	3.2 mmol/L of Na_2CO_3, 1.0 mmol/L of $NaHCO_3$



5.11 Ultra-high temperature processed milk

Column: Metrosep A Supp 5 - 100/4.0

Sample preparation: Metrohm Inline Dialysis

Detection: Conductivity

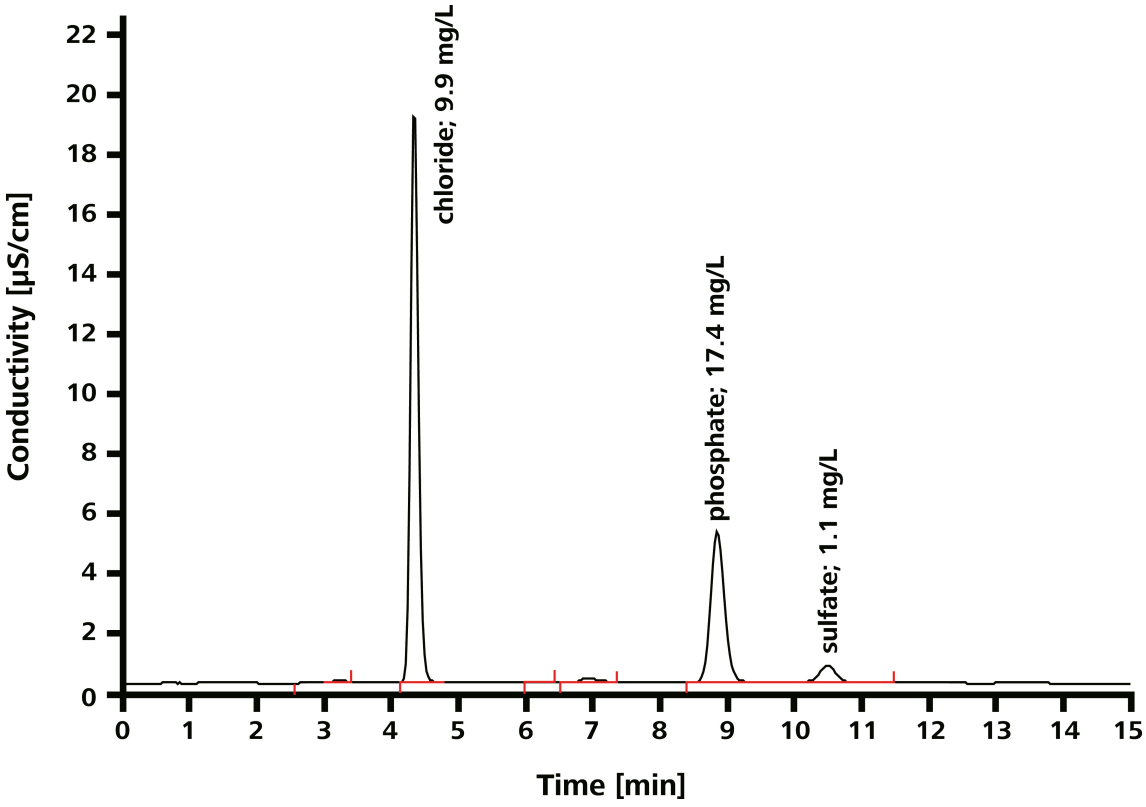
Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.7 mL/min

Eluent: 3.2 mmol/L of Na_2CO_3, 1.0 mmol/L of $NaHCO_3$


Conductivity [µS/cm]
22
20
18
16
14
12
10
8
6
4
2
0
chloride; 9.9 mg/L
phosphate; 17.4 mg/L
sulfate; 1.1 mg/L
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
Time [min]

6 Troubleshooting

6.1 Regeneration

CAUTION

Do not regenerate the column as a preventive measure!

Each regeneration process causes stress on the separation column and reduces its service life *see "Regenerating separation columns", page 6*.

Problem

- The backpressure increases
- Double peaks occur
- Tailing effects occur
- The retention times become shorter
- The resolution deteriorates

Correction

Regenerating the separation column

If the problems listed above occur, start by replacing the guard column. Only regenerate the separation column as described below if this measure does not help.

1 Disconnecting the separation column from the IC system

Disconnect the separation column outlet from the detector inlet.

2 Regenerating the separation column

The separation column has to be regenerated differently depending on the type of contamination:

- Contamination with low-valence hydrophilic ions *(see Table 4, page 36)*.
- Contamination with high-valence hydrophobic ions or organic contamination *(see Table 5, page 36)*
- Shifted system peak: Regeneration method with column oven *(see Table 6, page 36)*

Table 4 Contamination with low-valence hydrophilic ions

	Rinse with	Duration [min]	Flow rate [mL/min]	
			2-mm columns	4-mm columns
1	Ultrapure water	25	0.1	0.3
2	32 mmol/L of Na_2CO_3, 10 mmol/L of $NaHCO_3$	100	0.1	0.3
3	Ultrapure water	25	0.1	0.3
4	Eluent	100	0.1	0.3

Table 5 Contamination with high-valence hydrophobic ions or organic contamination

	Rinse with	Duration [min]	Flow rate [mL/min]	
			2-mm columns	4-mm columns
1	Ultrapure water	25	0.1	0.3
2	5% acetonitrile	20	0.1	0.3
3	100% acetonitrile	60	0.1	0.3
4	50% acetonitrile	10	0.1	0.3
5	Ultrapure water	50	0.1	0.3
6	Eluent	100	0.1	0.3

Table 6 Shifted system peak: Regeneration method with column oven

		Duration	Flow rate [mL/min]	
			2-mm columns	4-mm columns
1	Rinse with concentrated eluent of 1 mol/L of Na_2CO_3	25 min	0.1	0.4
2	Heat to 45 to 50 °C without rinsing	10 - 12 h	0	0
3	Rinsing with normal eluent	min. 40 min	0.1	0.4

6.2 Decreasing resolution / peak shapes

Problem

The resolution of peaks deteriorates or peak shapes are asymmetrical.

Causes and prevention

Causes	Prevention/correction
The separation column has been overloaded	The separation column can be overloaded by factors such as high salt content in the sample matrix. ▪ Dilute the sample. ▪ Inject less sample.
There are dead volumes in the IC system	▪ Check that all of the capillaries have a diameter of ≤ 0.25 mm (6.1831.010). If not, replace those capillaries. ▪ Check that all of capillaries have been installed correctly. The step-by-step installation process is shown in the IC Maintenance multimedia guide.

6.3 Unstable retention times

Problem

The retention times are unstable.

Causes and prevention

Causes	Prevention/correction
Carbonate in the eluent	Carbon dioxide from the air affects the carbonate/bicarbonate balance in the eluent. The eluent becomes weaker over time. ▪ Always keep the eluent bottle and bottles with eluent concentrates well sealed. ▪ Always use a CO_2 adsorber.
Air bubbles in the eluent	Air bubbles make the eluent flow unstable. Backpressure is one indicator of unstable flow. Backpressure should remain stable within ±0.1 MPa. ▪ Deaerate the high-pressure pump. ▪ Use an eluent degasser.

6.4 Unknown peaks

Problem

The chromatogram contains wide, unknown peaks.

Causes and prevention

Causes	Prevention/correction
Analytes eluting late	Some wider unknown peaks can be the result of sample components eluting late. In these cases, this is the result of the previous injection. ▪ Extend the chromatogram duration.

6.5 Increasing backpressure

Problem

The backpressure increases.

Causes and prevention

Causes	Prevention/correction
Particles on the guard column	▪ Replace the guard column.
Particles on the separation column	Rinse the separation column in the direction opposite the flow direction. ▪ Hold the column outlet in a beaker. ▪ Rinse the separation column for approximately 1 h. ▪ Install the separation column back in the flow direction.
Particles in the sample	▪ Sample preparation, e.g. removing particles through inline ultrafiltration.

7 Literature

We recommend referring to the following literature for more detailed information:

- Application Note S-229 Oxalate, thiosulfate and thiocyanate in amines
- Application Note S-239 Oxyhalides and standard anions according to EPA 300.1 applying the A Supp 5 - 250/4.0
- Application Note S-265 Semi-continuous determination of anions in aerosol applying PILS-IC
- Application Note S-269 MiPT – Metrohm intelligent Partial Loop Injection Technique
- Application Note S-275 Formate, acetate, oxalate and molybdate besides standard anions
- Application Note U-47 Anions in cooling lubricant after Inline Dialysis
- Application Note S-103 Five anions in water for infusion solutions
- Application Note S-323 Fast IC: Drinking water analysis including fluoride within less than seven minutes
- Application Note AN S-154 Eleven anions with high pressure gradient elution
- Direct injection ion chromatography for the control of chlorinated drinking water: simultaneous estimation of nine haloacetic acids and quantitation of bromate, chlorite and chlorate along with the major inorganic anions; p. 443-451; Journal of Water and Health, Vol. 12; No. 3;2014
- Poster: Determination of hexavalent chromium in drinking water according to a U.S. EPA Method; 8.000.6087
- Optimal Sample Preservation and Analysis of Cr(VI) in Drinking Water Samples by High Resolution Ion Chromatography Followed by Post Column Reaction and UV/Vis Detection; p.74-80; Journal of Analytical Sciences, Methods and Instrumentation; 2/2012
- Sulphur and halide determination by combustion ion chromatography; p. 9-12; LC-GC Europe; May 31st, 2010
- Metrosep A Supp 5-xxx/2.0 Flyer, 8.107.5003
- Monograph: Analysis of water samples and water constituents with Metrohm instruments, page 66ff. (8.038.5003)
- Column catalog, 8.000.5117

Index

B

Baseline
- Condition 16

C

Column
- see "Separation column" 11

Conditioning 16

E

Eluent 8

Equilibration 15

F

Flow rate 2

G

Guard column
- Installation 9
- Rinse 11

I

IC column
- see "Separation column" 11

Installation
- Guard column 9
- Separation column 11

O

Order number 1

R

Rinse
- Guard column 11
- Separation column 14

S

Separation column
- Installation 11
- Rinse 14

Specification 2

Storage 3