Column manual

Metrosep A Supp 21 (6.01036.4x0)

Manual

8.0107.8015EN / v1 / 2024-09-01

Metrohm

Metrohm AG
CH-9100 Herisau
Switzerland
+41 71 353 85 85
info@metrohm.com
www.metrohm.com

Column manual

Metrosep A Supp 21 (6.01036.4x0)

Manual

8.0107.8015EN / v1 / 2024-09-01

Technical Communication
Metrohm AG
CH-9100 Herisau

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.

Disclaimer

Deficiencies arising from circumstances that are not the responsibility of Metrohm, such as improper storage or improper use, etc., are expressly excluded from the warranty. Unauthorized modifications to the product (e.g. conversions or attachments) exclude any liability on the part of the manufacturer for resulting damage and its consequences. Instructions and notes in the Metrohm product documentation must be strictly followed. Otherwise, Metrohm's liability is excluded.

Table of contents

1 General information 1

1.1 Ordering information ... 1

1.2 Technical specifications ... 1

2 Key aspects of working with separation columns 3

3 Eluent production 7

3.1 Chemicals ... 7

3.2 Production of standard eluent ... 7

4 Start-up 9

4.1 Connecting and rinsing the guard column ... 9

4.2 Connecting and rinsing the separation column ... 11

4.3 Conditioning ... 14

5 Applications 16

5.1 Standard chromatogram ... 16

5.1.1 Metrosep A Supp 21 - 150/4.0 ... 16

5.1.2 Metrosep A Supp 21 - 250/4.0 ... 17

5.2 Effects of temperature ... 19

5.3 Variation of the eluent ... 20

5.4 Variation of the methanol concentration ... 22

5.5 Determination of standard anions in mineral water samples ... 24

5.6 Determination of standard anions and oxyhalides in mineral water samples according to ISO 10304-1&4 ... 25

5.7 Determination of standard anions, oxyhalides and DCAA in mineral water samples according to US EPA 300.1 A+B ... 26

6 Troubleshooting 28

6.1 Regeneration ... 28

6.2 Decreasing resolution and asymmetrical peaks ... 29

6.3 Unstable retention times ... 30

6.4 Unknown peaks ... 30

6.5 Increasing backpressure ... 31

7 Literature **32**

Index **33**

1 General information

The Metrosep A Supp 21 is a high-performance separation column and is especially suitable for the determination of inorganic anions and oxyhalides with chemical and sequential suppression using hydroxide eluents. Due to its high capacity, the Metrosep A Supp 21 can readily handle samples with high ionic strength and with large fluctuations in concentration. The outstanding peak symmetries and high number of theoretical plates allow universal use in ion chromatography.

1.1 Ordering information

Table 1 Separation columns

Order number	Designation
6.01036.420	Metrosep A Supp 21 - 150/4.0
6.01036.430	Metrosep A Supp 21 - 250/4.0

Table 2 Guard column

Order number	Designation
6.01036.500	Metrosep A Supp 21 Guard/4.0

1.2 Technical specifications

Column material

Hydrophilic polystyrene/divinylbenzene copolymer with quaternary ammonium groups

Particle size

4.6 µm

Measurements

Order number	Measurements
6.01036.420	150 x 4.0 mm
6.01036.430	250 x 4.0 mm

pH range eluent

0–14

pH range sample

0–14

Temperature range

10–70 °C

Recommended standard temperature

Order number	Recommended standard temperature
6.01036.420	25 °C
6.01036.430	45 °C

Maximum pressure

Order number	Maximum pressure
6.01036.420	21 MPa (210 bar)
6.01036.430	25 MPa (250 bar)

Flow rate

Order number	Recommended flow rate	Maximum flow rate
6.01036.420	0.8 mL/min	1.4 mL/min
6.01036.430	0.8 mL/min	1.5 mL/min

Standard eluent

Order number	Standard eluent
6.01036.420	15–60 mmol/L potassium hydroxide
6.01036.430	18–80 mmol/L potassium hydroxide

Permitted organic additives

0–100% acetone, acetonitrile, isopropanol and methanol

Capacity

Order number	Capacity
6.01036.420	246 µmol (Cl^-)
6.01036.430	410 µmol (Cl^-)

Preparation

Rinse the column with 80 mmol/L potassium hydroxide for 3 to 4 hours. Thereby, use a flow gradient to set the column to the standard flow within 10 minutes.

Storage

Rinse the column with 20 mmol/L sodium sulfate and store at 4 to 8 °C.

Typical pressure

For columns with a guard column under standard conditions with sequential suppression:

Order number	Typical pressure
6.01036.420	15 MPa
6.01036.430	15 MPa

Column housing

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

Application

Determination of inorganic standard anions as well as oxyhalides and dichloroacetic acid with chemical and sequential suppression.

2 Key aspects of working with separation columns

Storage

Once the backpressure in the ion chromatograph has dissipated, remove the column at ambient temperature. Seal the column at both ends using the original stoppers (6.2744.060). Store the column in the standard eluent and at 4 to 30 °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. Metrohm recommends cleaning all vessels as follows before filling them:

1. Thoroughly rinse with ultrapure, UV-treated water (> 18.2 MΩ).
2. Swirl an acetonitrile-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. This is only possible if you are *not using membrane suppressors*. Organic solvents can destroy membrane suppressors. The Metrohm Suppressor Modules (MSM, MSM-HC and MSM-LC) are 100% resistant to solvents.

Chemical quality

All chemicals must have at least 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 / hydrogen carbonate balance in the eluent. The eluent becomes weaker over time. In order to prevent this, always outfit the eluent bottle with a CO_2 adsorber with the adsorber material soda lime.

Degassing the eluent

In order to prevent bubbles from forming, degas the produced eluent before using it in the IC system. To do this, create a vacuum for approximately 10 minutes using a water-jet pump or a membrane pump. Alternatively, use an ultrasonic bath or work with an eluent degasser.

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 moisture and carbon dioxide from getting into the eluent. Normally, the adsorber tube is filled with molecular sieve or – for sodium hydroxide and carbonate eluents – with soda lime (a weak CO_2 adsorber material).

Filter

Problems that occur in IC systems are usually related to particles. Particles are 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), an 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. Metrohm recommends replacing the filters regularly.

Filtering the eluent

Microfilter (0.45 µm) all eluents immediately before use.

Mechanical stress

Avoid mechanical stress on the column. For example, the column impacting a hard surface can cause a break or gap in the column packaging (separation phase material). This affects the chromatography results. The column is irreparably damaged as a result.

Particles

All solutions, samples, regeneration solutions, water and eluents must be free of particles. Particles clog separation columns over time. This causes an increase in column pressure. 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 1,000 mL per workday compared to about 0.5 mL of the sample solution. Filter or dialyze the 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 must not 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 the guard column. Always use a guard column for each separation column. Replace the guard column 3 to 4 times during the service life of the analytical column. As an alternative to sample preparation cartridges, Metrohm Inline Sample Preparation techniques (MISP) can be used, such as for neutralizing alkaline samples.

Pulsation absorber

Always use a pulsation absorber (6.2620.150). Protect polyvinyl alcohol columns in particular from the brief pressure surges that occur when

switching the valves. Using the pulsation absorber (6.2620.150) already built into the Metrohm ion chromatographs provides this protection.

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 despite this or if the separating efficiency decreases, carry out the specified regeneration steps. Perform the regeneration outside the analytical line. For regeneration, connect the separation column to the pump directly. Route the regeneration solution through the column directly into a waste container. Rinse the separation column properly with fresh eluent. Then, reinstall the separation column.

Shutting down the ion chromatograph

If the ion chromatograph is not used for a prolonged period (> 1 week), remove the separation column and seal it with the stoppers provided. Rinse the ion chromatograph, including all 3 suppressor chambers, with a methanol-water mixture (1:4). Store the separation column in the medium indicated on the column leaflet. Unless otherwise stated on the column leaflet, store the column at 4 to 30 °C.

Prior to start-up, rinse the ion chromatograph with ultrapure water and then with fresh eluent. Bring the separation column back to 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. When 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. Metrohm strongly recommends using guard columns. Guard columns normally contain the same stationary phase as separation columns. However, the quantity is significantly reduced to avoid impacting the chromatography. Guard columns remove critical contaminants that can react with column material. Guard columns also 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

Replace the guard column 3 to 4 times during the service life of the analytical column. Guard columns are available for all Metrosep separation columns.

Water quality

Aqueous media are mostly used in work involving ion chromatography. This means that water quality is a critical factor for good chromatography. If the water quality is inadequate, the results will be inadequate as well. Water with inadequate quality can damage instruments and separation columns. The ultrapure water being used must have a specific resistance greater than 18.2 MΩ·cm and should be free of particles. Therefore, filter the water using a 0.45-µm filter and treat it with UV light. Modern ultrapure water systems for laboratory use ensure this level of water quality (Type I).

3 Eluent production

Metrohm recommends selecting a high degree of purity for chemicals for standard production and eluent production.

3.1 Chemicals

Recommended chemicals for eluents:

- Potassium hydroxide 4 mol/L, Merck, order number: 67109
- Potassium hydroxide 47%, Merck, order number 1.05545
- Ultra pure water type I (according to ASTM D1193)
  Resistivity >18.2 MΩ·cm (25 °C)
  TOC < 10 µg/L

3.2 Production of standard eluent

Produce as follows to produce 2 L of standard eluent with 15, 18 or 100 mmol/L of potassium hydroxide:

Producing 2 L of standard eluent

Accessories

- Eluent bottle (6.1608.120)
- Bottle cap (6.1602.200) equipped with CO_2 adsorber
- Ultrapure water
- Potassium hydroxide

1 Pre-rinse the eluent bottle with ultrapure water several times.

2 Fill 2 L of ultrapure water into the eluent bottle.

3 Use the eluent degasser.

If no eluent degasser is available, degas the ultrapure water for 5 to 10 minutes using a vacuum pump. Degassing avoids problems with air bubbles in the high-pressure pump.

4 Pipet the volume of potassium hydroxide from the below table into the eluent.

Final concentration	15 mmol/L	18 mmol/L	100 mmol/L
Potassium hydroxide 4 mol/L	7.5 mL	9.0 mL	50 mL
Potassium hydroxide 47%	2.43 mL	2.91 mL	16.19 mL

5 Rinse the column with 80 mmol/L potassium hydroxide for 3 to 4 h.

This eluent (18 mmol/L potassium hydroxide) and sequential suppression can be used to achieve a background conductivity of less than 0.4 µS/cm. The noise 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 cartridge leaflet.

NOTE

Metrohm recommends always working with guard columns. Guard columns 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 regional Metrohm representative), the column leaflet and the product information or in consultation with your regional Metrohm representative.

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

CAUTION

New guard columns are filled with 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 manufacturer specification).

NOTE

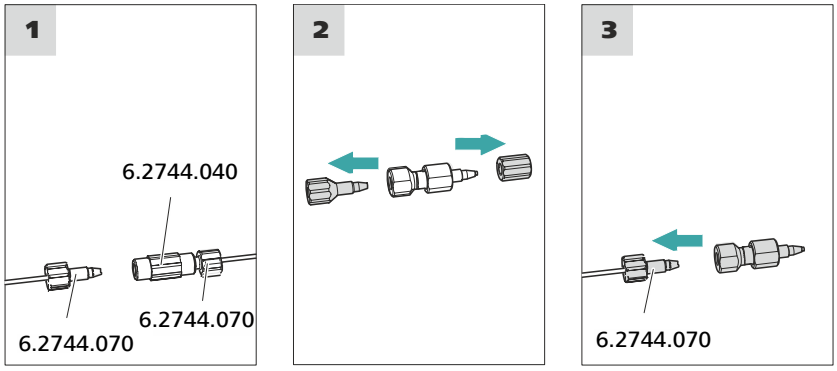
Do not connect the guard column until after the initial start-up of the instrument. Until then, replace the guard column and the separation column with couplings (6.2744.040).

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 stoppers or 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 and rinsing 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 from your regional Metrohm representative.

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

A test chromatogram accompanies every column. The column leaflet can be found online at *http://www.metrohm.com* with the corresponding article. 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 free of charge from your responsible regional Metrohm representative.

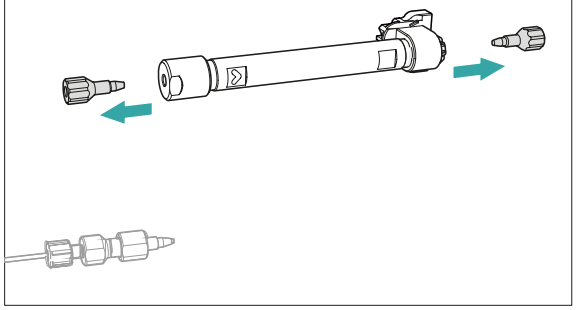
CAUTION

New IC Columns are filled with 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).

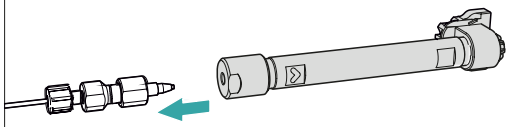
NOTE

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.

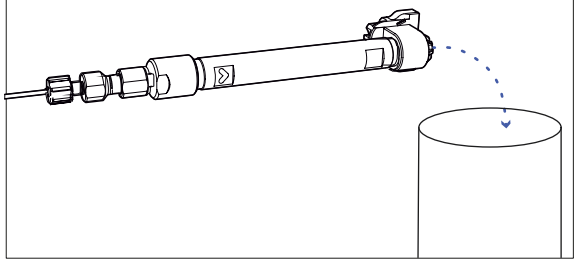
1



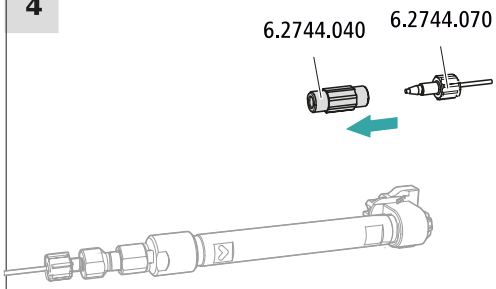
2



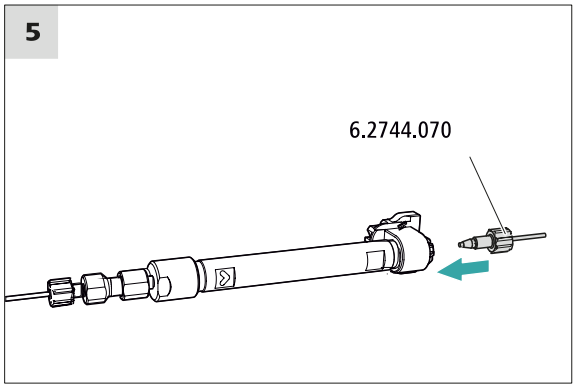
3



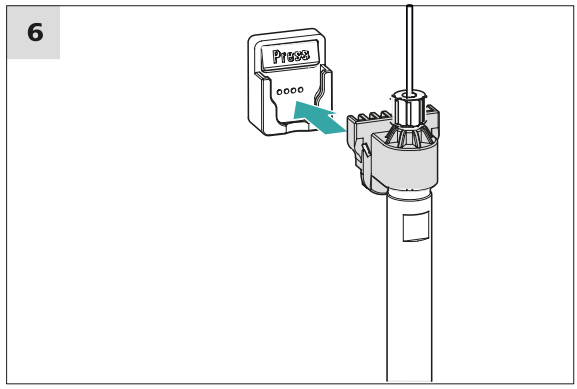
4



5



6



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 3 possibilities:

- 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.
  See also: *MagIC Net tutorial* and online help.

2 Preparing the instrument

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

3 Starting the 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

5.1.1 Metrosep A Supp 21 - 150/4.0

Column: Metrosep A Supp 21 - 150/4.0

Sample preparation: –

Detection: Conductivity

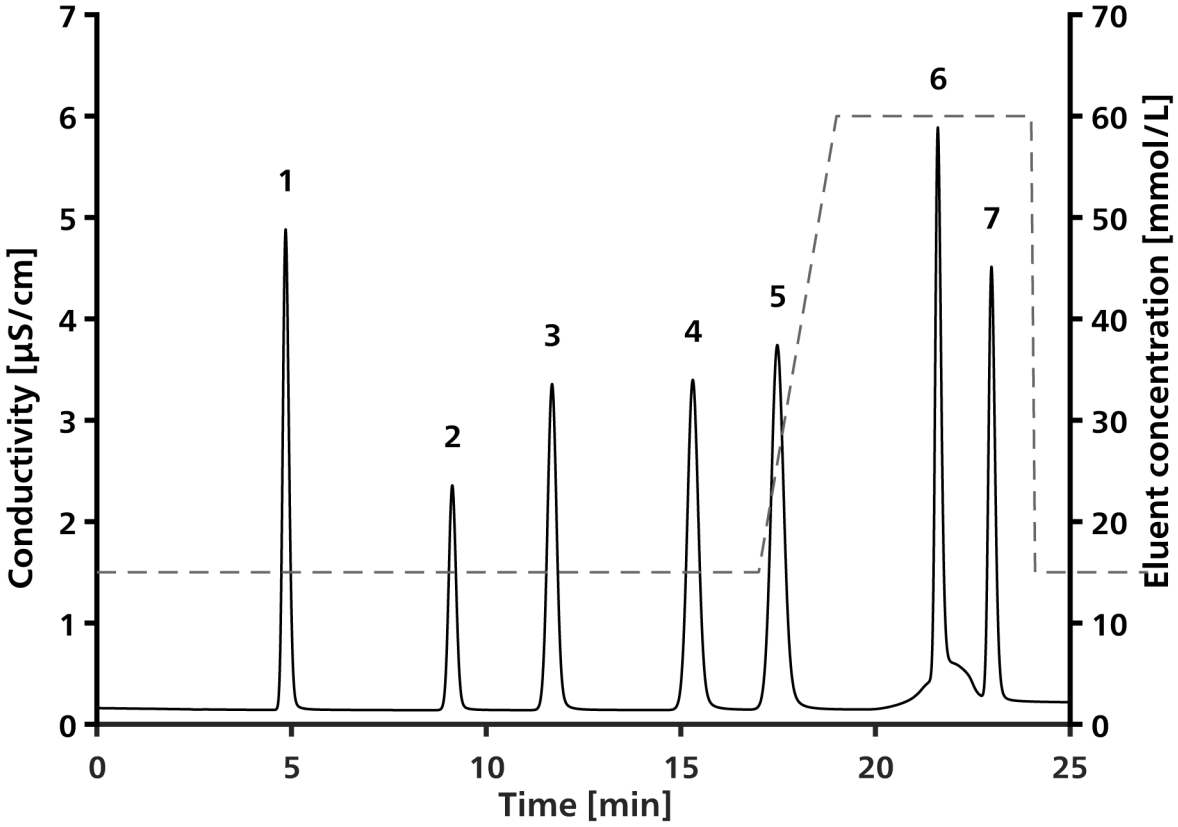
Suppression: Sequential suppression with MSM-HC A and MCS

Temperature: 25 °C

Loop: 20 µL

Flow rate: 0.8 mL/min

Eluent: 15–60 mmol/L KOH



	Metrosep A Supp 21 - 150/4.0	mg/L
1	Fluoride	2
2	Chloride	2
3	Nitrite	5
4	Bromide	10
5	Nitrate	10
6	Sulfate	5
7	Phosphate	10

5.1.2 Metrosep A Supp 21 - 250/4.0

Column: Metrosep A Supp 21 - 250/4.0

Sample preparation: –

Detection: Conductivity

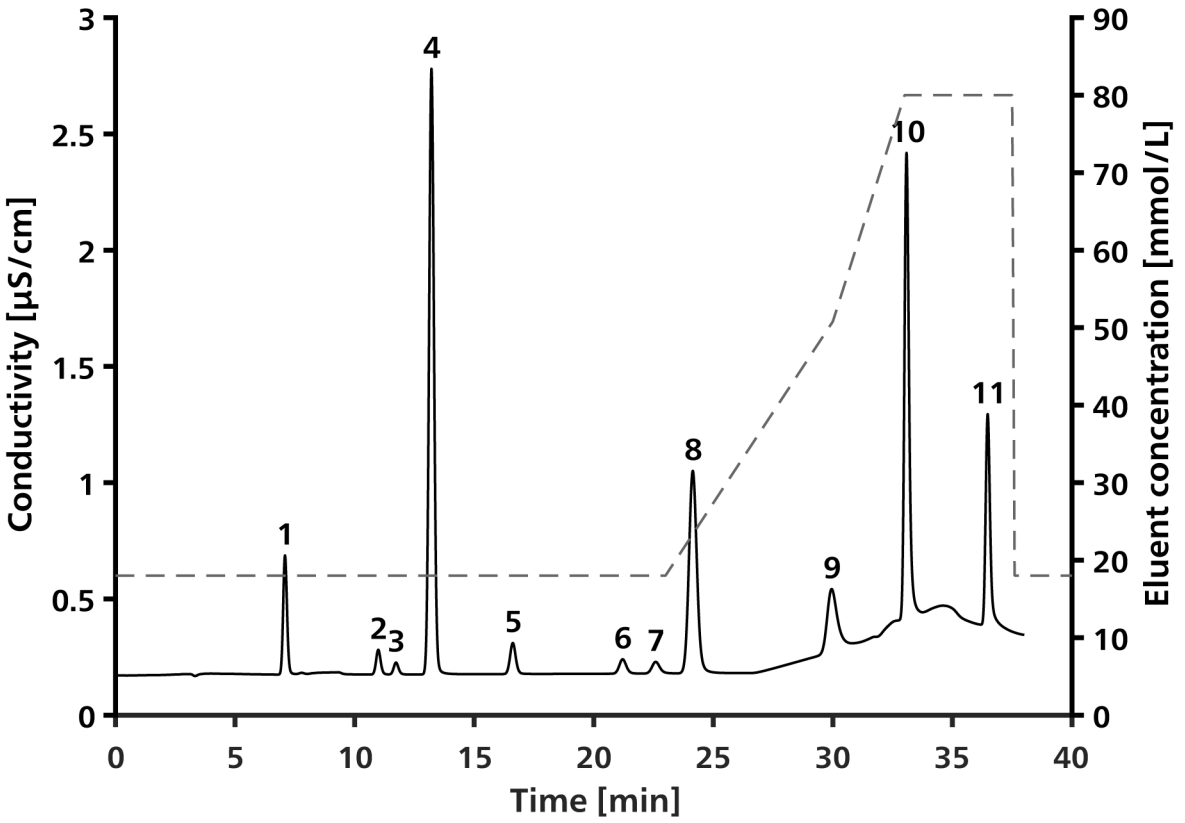
Suppression: Sequential suppression with MSM-HC A and MCS

Temperature: 45 °C

Loop: 50 µL

Flow rate: 0.8 mL/min

Eluent: 18–80 mmol/L KOH



	Metrosep A Supp 21 - 250/4.0	mg/L
1	Fluoride	0.1
2	Chlorite	0.1
3	Bromate	0.1
4	Chloride	1
5	Nitrite	0.1
6	Bromide	0.1
7	Chlorate	0.1
8	Nitrate	1
9	Dichloroacetate	1
10	Sulfate	1
11	Phosphate	1

5.2 Effects of temperature

Column: Metrosep A Supp 21 - 250/4.0

Sample preparation: –

Detection: Conductivity

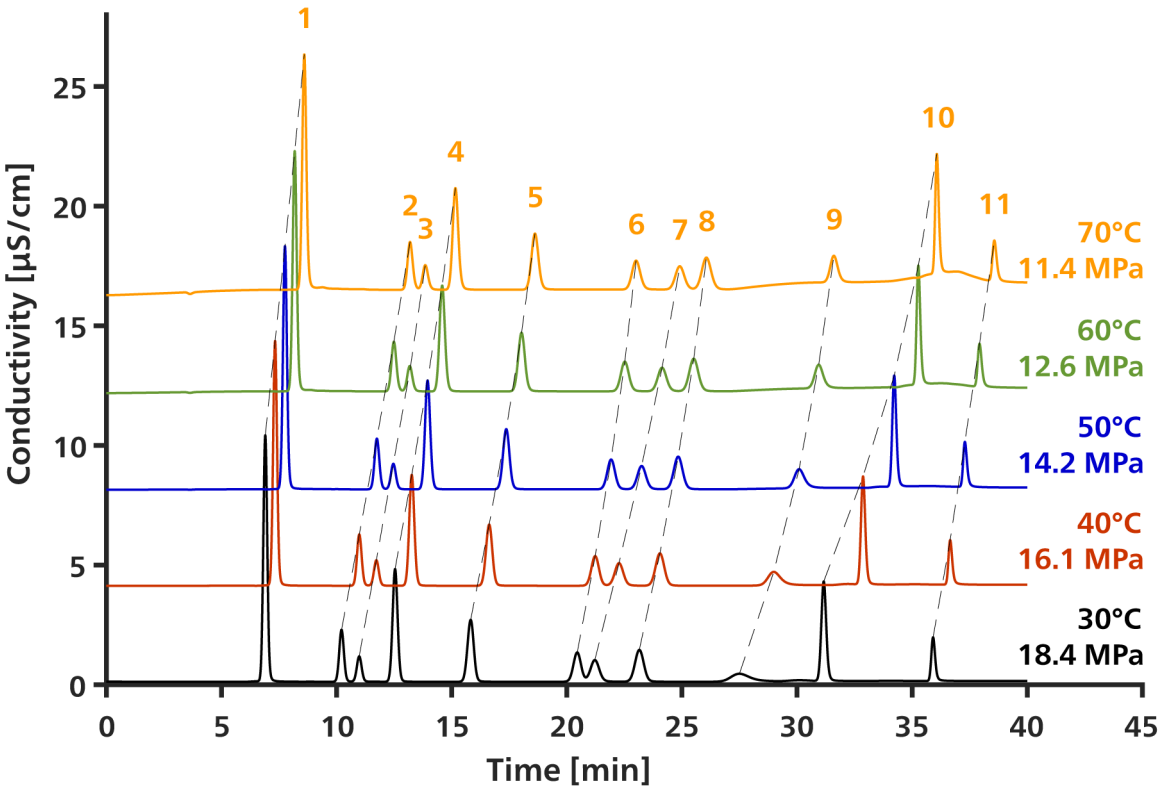
Suppression: Sequential suppression with MSM-HC A and MCS

Temperature: 30–70 °C

Loop: 50 µL

Flow rate: 0.8 mL/min

Eluent: 18–80 mmol/L KOH



	Metrosep A Supp 21 - 250/4.0	mg/L
1	Fluoride	2
2	Chlorite	2

	Metrosep A Supp 21 - 250/4.0	mg/L
3	Bromate	2
4	Chloride	2
5	Nitrite	2
6	Bromide	2
7	Chlorate	2
8	Nitrate	2
9	Dichloroacetate	2
10	Sulfate	2
11	Phosphate	2

The Metrosep A Supp 21 can be used at temperatures from 10 to 70 °C. With increasing temperature, the retention times of all ions are increased. Chlorate is delayed more than its neighboring peaks bromide and nitrate, such that the chlorate peak moves from bromide to nitrate with increasing temperature. For optimal separation of bromide, chlorate and nitrate, a temperature of 45°C is required.

Increasing the temperature also causes the column backpressure to decrease considerably. At 70 °C, the backpressure is only approx. 11.4 MPa, whereas the column backpressure at 30 °C is much higher (about 18.4 MPa). Due to the high backpressure at low temperatures, standard flow measurements at 10 °C are not possible on the Metrosep A Supp 21 - 250/4.0 column.

5.3 Variation of the eluent

Column: Metrosep A Supp 21 - 250/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM-HC A and MCS

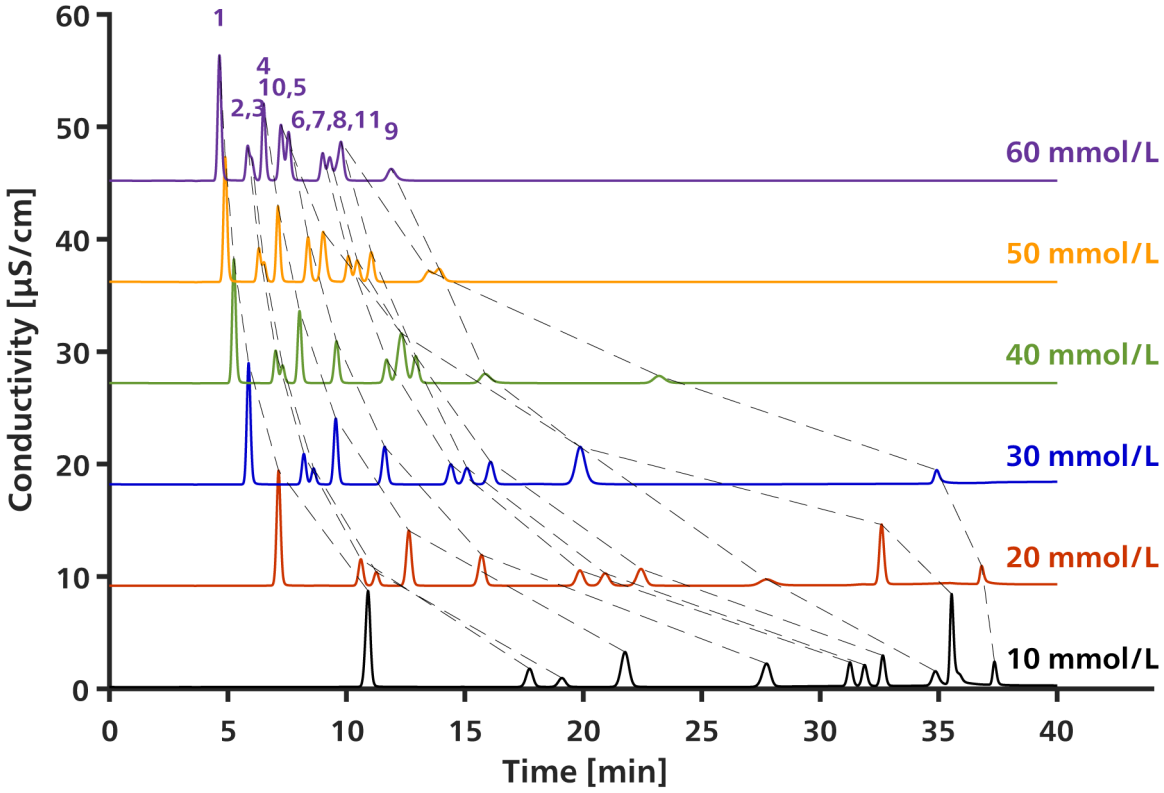
Temperature: 45 °C

Loop: 50 µL

Flow rate: 0.8 mL/min

Eluent:
1. 10–80 mmol/L KOH
2. 20–80 mmol/L KOH

3. 30–80 mmol/L KOH
4. 40–80 mmol/L KOH
5. 50–80 mmol/L KOH
6. 60–80 mmol/L KOH



	Metrosep A Supp 21 - 250/4.0	mg/L
1	Fluoride	2
2	Chlorite	2
3	Bromate	2
4	Chloride	2
5	Nitrite	2
6	Bromide	2
7	Chlorate	2
8	Nitrate	2
9	Dichloroacetate	2
10	Sulfate	2
11	Phosphate	2

With increasing eluent concentration, all anions are significantly accelerated. In this process, the multivalent anions sulfate and phosphate are accelerated faster than the monovalent anions. With a stronger eluent, the peaks are sharper and correspondingly higher. At an eluent concentration of 30 mmol/L KOH, sulfate already coelutes with dichloroacetate and at an eluent concentration of 60 mmol/L KOH, sulfate elutes just before nitrite, while phosphate coelutes with nitrate.

5.4 Variation of the methanol concentration

Column: Metrosep A Supp 21 - 250/4.0

Sample preparation: –

Detection: Conductivity

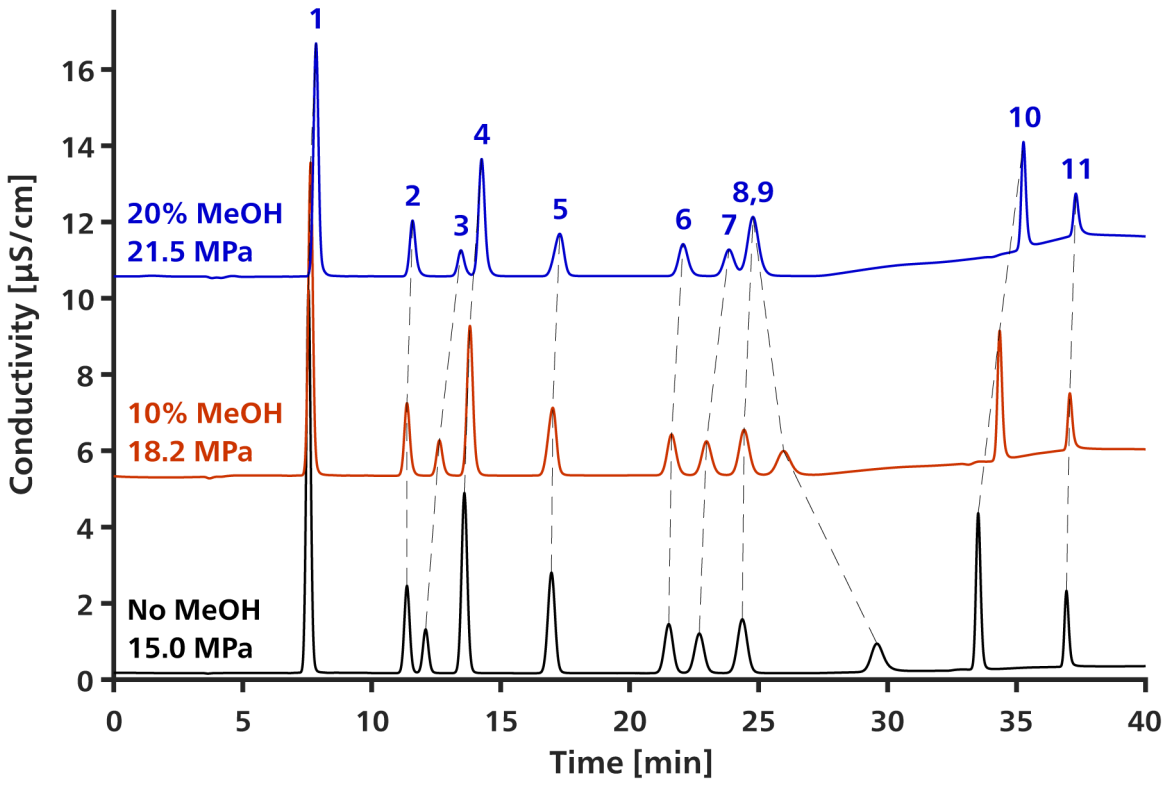
Suppression: Sequential suppression with MSM-HC A and MCS

Temperature: 45 °C

Loop: 50 µL

Flow rate: 0.8 mL/min

Eluent:
1. 18–80 mmol/L KOH, 0% methanol
2. 18–80 mmol/L KOH, 10% methanol
3. 18–80 mmol/L KOH, 20% methanol



	Metrosep A Supp 21 - 250/4.0	mg/L
1	Fluoride	2
2	Chlorite	2
3	Bromate	2
4	Chloride	2
5	Nitrite	2
6	Bromide	2
7	Chlorate	2
8	Nitrate	2
9	Dichloroacetate	2
10	Sulfate	2
11	Phosphate	2

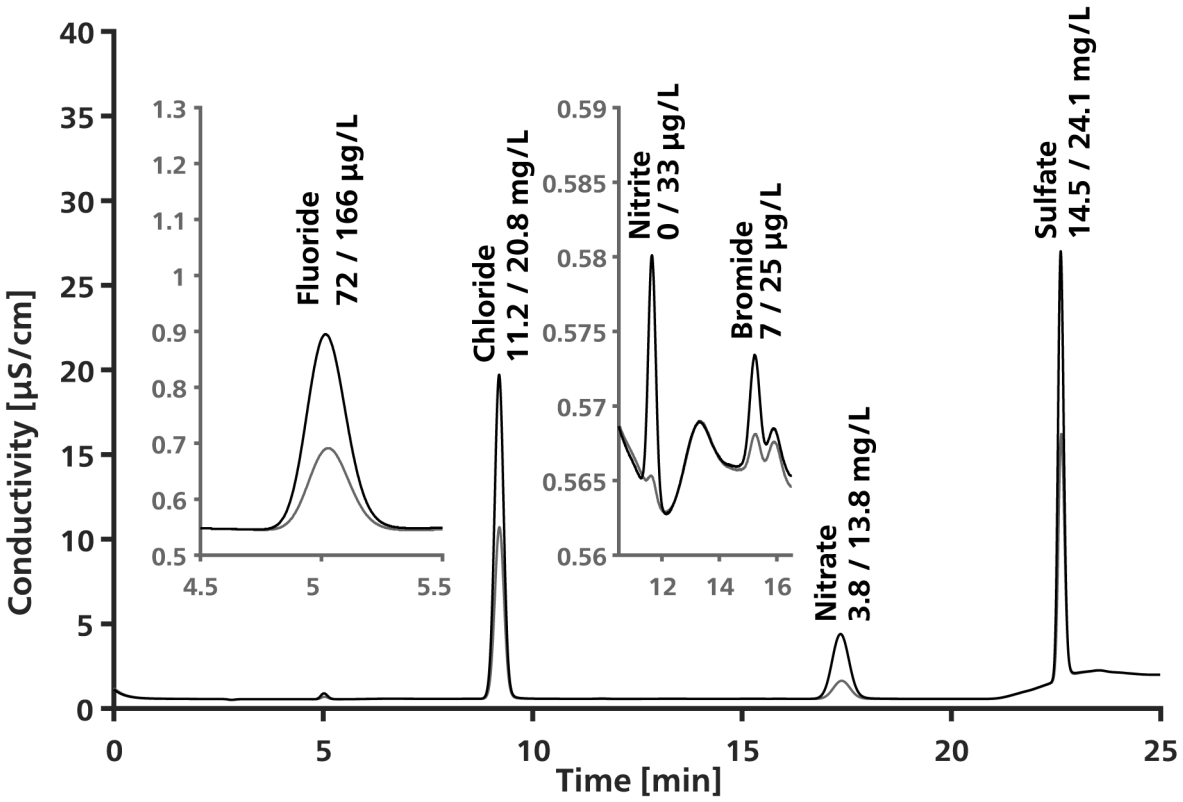
In some cases, the use of an organic modifier is useful or even necessary. The eluent can be made more stable against bacterial contamination by adding a modifier, or the modifier can help improve the rinsing-out of the organic parts of a sample from the separation column. With the addition of an organic modifier, the backpressure of the column and the selectivity

of all anions change. An increase of the methanol content in the eluent shifts the bromate peak away from the chlorite peak towards the chloride peak. Also the chlorate peak is shifted towards nitrate, while the dichloroacetate peak is accelerated into the nitrate peak. Adding methanol to the eluent tends to cause deterioration of the shapes of the peaks and thus also the peak heights.

The column backpressure increases with increasing methanol content in the eluent due to the increasing viscosity of the eluent mixture. Without methanol in the eluent, the pressure is approx. 15 MPa. With 20% methanol in the eluent, it is already approx. 22 MPa.

5.5 Determination of standard anions in mineral water samples

Column:	Metrosep A Supp 21 - 150/4.0
Sample preparation:	–
Detection:	Conductivity
Suppression:	Sequential suppression with MSM-HC A and MCS
Temperature:	25 °C
Loop:	20 µL
Flow rate:	0.8 mL/min
Eluent:	15–60 mmol/L KOH

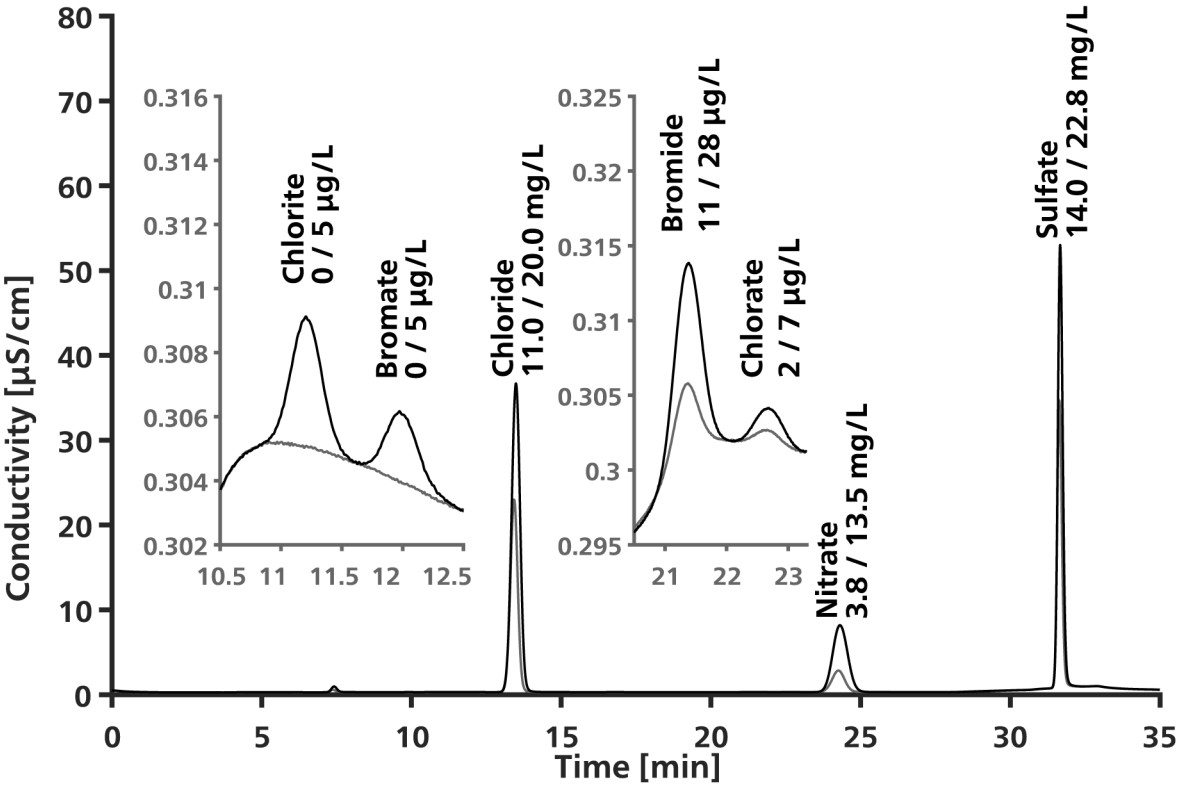


The grey chromatogram corresponds to the mineral water sample directly injected while the black chromatogram corresponds to the same sample spiked with 10 mg/L chloride, nitrate, and sulfate, 100 µg/L fluoride, and 20 µg/L nitrite and bromide.

5.6 Determination of standard anions and oxyhalides in mineral water samples according to ISO 10304-1&4

Column:	Metrosep A Supp 21 - 250/4.0
Sample preparation:	–
Detection:	Conductivity
Suppression:	Sequential suppression with MSM-HC A and MCS
Temperature:	45 °C
Loop:	50 µL
Flow rate:	0.8 mL/min

Eluent: 18–80 mmol/L KOH



The grey chromatogram corresponds to the sample directly injected while the black chromatogram corresponds to the sample spiked with 10 mg/L chloride, nitrate, and sulfate, 100 µg/L fluoride, 20 µg/L nitrite and bromide, and 5 µg/L chlorite, bromate, and chlorate.

5.7 Determination of standard anions, oxyhalides and DCAA in mineral water samples according to US EPA 300.1 A+B

Column: Metrosep A Supp 21 - 250/4.0

Sample preparation: –

Detection: Conductivity

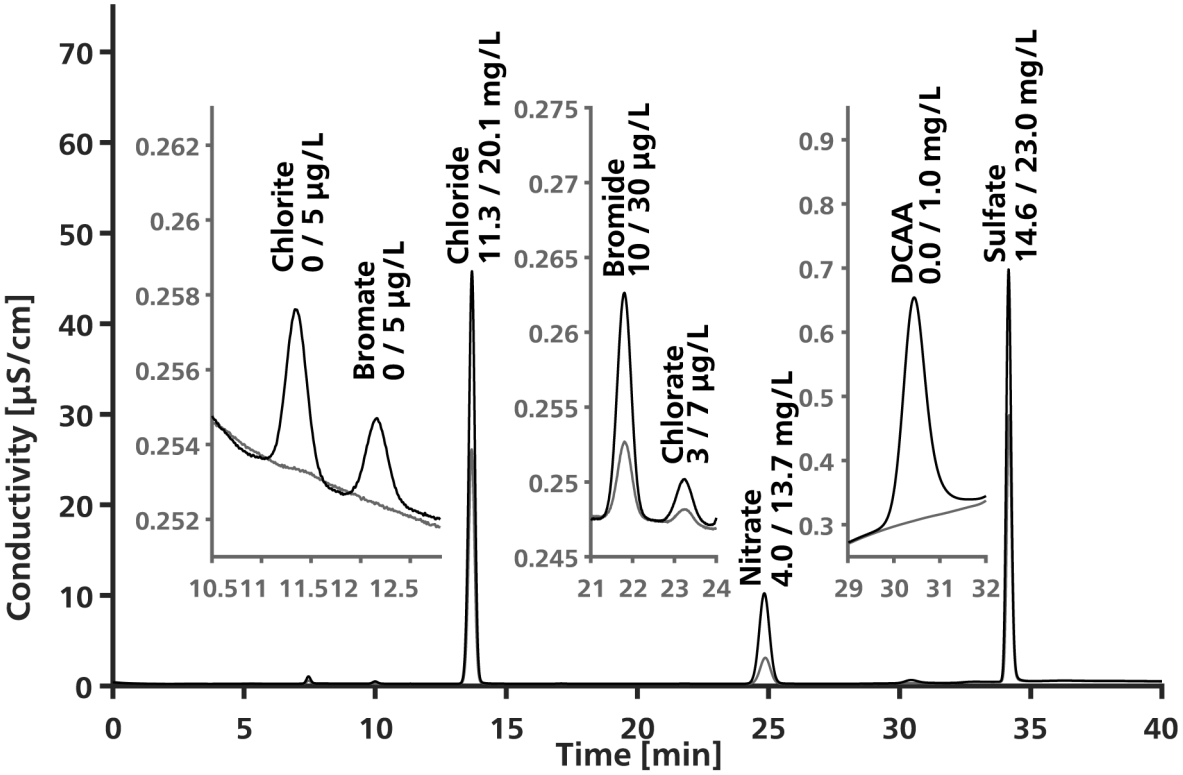
Suppression: Sequential suppression with MSM-HC A and MCS

Temperature: 45 °C

Loop: 50 µL

Flow rate: 0.8 mL/min

Eluent: 18–80 mmol/L KOH



The grey chromatogram corresponds to the sample directly injected while the black chromatogram corresponds to the sample spiked with 10 mg/L chloride, nitrate, and sulfate, 1 mg/L dichloroacetate, 100 µg/L fluoride, 20 µg/L nitrite and bromide, and 5 µg/L chlorite, bromate, and chlorate.

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 5*.

Problem

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

Correction

Regenerating the separation column

Start by replacing the guard column if the above problems occur. Regenerate the separation column as described below if this measure does not help.

1 Disconnecting the separation column from the IC system

Disconnect the column outlet from the downstream functional units such as suppressor or detector.

Collect the flow of liquid in a beaker.

2 Regenerating the separation column

NOTE

Ensure that the maximum pressure is never exceeded during regeneration. If the pressure becomes too high, reduce the flow rate.

Depending on the type of contamination, regenerate the separation column as follows:

- Contamination with organic components *(see table 3, page 29)*

- Contamination with inorganic components *(see table 4, page 29)*.

When using organic modifiers for the regeneration, pay attention to the maximum backpressure.

Table 3 Contamination with organic components

	Rinse with	Duration [h]	Flow rate [mL/min]	Flow direction
1	Ultrapure water	1	0.5	Direction against the flow
2	Acetonitrile-water mixture (50:50)	2	0.5	Direction against the flow
3	Ultrapure water	1	0.5	Direction against the flow
4	Eluent	2	0.7	Regular

Table 4 Contamination with inorganic components

	Rinse with	Duration [h]	Flow rate [mL/min]	Flow direction
1	80 mmol/L KOH	2	0.5	Direction against the flow
2	Eluent	2	0.7	Regular

6.2 Decreasing resolution and asymmetrical peaks

Problem

The resolution of peaks deteriorates or peak shapes are asymmetrical.

Causes and prevention

Causes	Prevention or correction
The separation column has been overloaded.	The separation column can be overloaded by factors such as a high salt content in the sample matrix. ▪ Dilute the sample. ▪ Inject less sample.

Causes	Prevention or correction
There are dead volumes in the IC system.	▪ Check that all of the capillaries have an inner diameter of ≤ 0.25 mm (6.1831.010). If not, use capillaries with a smaller inner diameter. ▪ Check that all of the capillaries are correctly installed. The IC Maintenance multimedia guide shows the installation process step-by-step.

6.3 Unstable retention times

Problem

The retention times are unstable.

Causes and prevention

Causes	Prevention or correction
Carbonate in the eluent	Carbon dioxide from the air affects the carbonate / hydrogen carbonate 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 rate unstable. Backpressure is one indicator of an unstable flow rate. The backpressure must remain stable within ±0.1 MPa. ▪ Purge the high-pressure pump. ▪ Use the eluent degasser.

6.4 Unknown peaks

Problem

The chromatogram contains wide, unknown peaks.

Causes and prevention

Causes	Prevention or correction
Analytes eluting late	Somewhat wider, unknown peaks can be the result of sample components eluting late. They are the result of the previous injection. ▪ Extend the chromatogram duration.

6.5 Increasing backpressure

Problem

The backpressure increases.

Causes and prevention

Causes	Prevention or correction
Particles on the guard column	▪ Replace the guard column.
Particles on the separation column	Rinse the separation column at a reduced flow rate in the direction opposite to 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

Metrohm recommends the following literature for more detailed information:

- Column catalog, 8.000.5347

Index

B

Baseline
Condition 15

C

Capacity 2
Column
see "Separation column" 11
Conditioning 15

E

Eluent 7
Equilibration 14

F

Flow rate 2

G

Guard column
Installation 9
Rinse 9, 10

I

IC column
see "Separation column" 11
Installation
Guard column 9
Separation column 11

O

Order number 1

R

Rinse
Guard column 9, 10
Separation column 13
Rinsing
Separation column 11

S

Separation column
Installation 11
Rinse 13
Rinsing 11
Specification 1