Column manual

Metrosep A Supp 17 - XXX/4.0 (6.01032.4X0)

Manual

8.0107.8001EN / 2021-03-30

Metrohm

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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.

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1 General information

This anion separation column is especially suitable for determinations of drinking water, raw water, wastewater and similar samples. Inorganic low-molecular anions are analyzed in this process.

1.1 Ordering information

Table 1 4-mm columns

Order number	Designation
6.01032.410	Metrosep A Supp 17 - 100/4.0
6.01032.420	Metrosep A Supp 17 - 150/4.0
6.01032.430	Metrosep A Supp 17 - 250/4.0

Table 2 4-mm guard column

Order number	Designation
6.01032.500	Metrosep A Supp 17 Guard/4.0
6.01032.510	Metrosep A Supp 17 S-Guard/4.0

1.2 Technical specifications

Column material Polystyrene/divinylbenzene copolymer with quaternary ammonium groups

Particle size 5 µm

Measurements

Order number	Measurements
6.01032.410	100 x 4.0 mm
6.01032.420	150 x 4.0 mm
6.01032.430	250 x 4.0 mm

pH range 0 to 14

Temperature range 10 to 70 °C

Recommended standard temperature 25 °C

Maximum pressure 18 MPa (180 bar)

Flow rate

Order number	Recommended flow rate	Maximum flow rate
6.01032.410	0.6 mL/min	1.8 mL/min
6.01032.420	0.6 mL/min	1.4 mL/min
6.01032.430	0.6 mL/min	0.9 mL/min

Standard eluent

5.0 mmol/L sodium carbonate and 0.2 mmol/L sodium hydrogen carbonate

Permitted organic additives

In the eluent

0 to 40% acetonitrile, acetone
0 to 100% methanol

In the sample matrix

0 to 40% acetonitrile, acetone
0 to 100% methanol

Capacity

Order number	Capacity
6.01032.410	43 µmol (Cl-)
6.01032.420	65 µmol (Cl-)
6.01032.430	109 µmol (Cl-)

Preparation

1. Use a flow gradient to set the column to the standard flow within 2 minutes.
2. Wait until the baseline sets.

Storage

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

Typical pressure

For columns with guard column at standard conditions:

Order number	Typical pressure
6.01032.410	5.7 ± 2 MPa
6.01032.420	7.6 ± 2 MPa
6.01032.430	10.9 ± 2 MPa

Column housing

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

Application

Determination of inorganic anions and low-molecular 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. Metrohm recommends 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 or acetonitrile to the eluent.

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.

Also protect eluents that have a weak buffer capacity (such as caustic soda eluents) from carbon dioxide.

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 CO_2 adsorber material (such as soda lime).

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. The adsorber

tube is usually filled with molecular sieve. For sodium hydroxide and carbonate eluents, soda lime (a weak CO_2 adsorber) is used.

Degassing the eluent

In order to prevent bubbles from forming, Metrohm recommends 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 an oil pump. 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), an inline filter (6.2821.120) and a guard column. 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 must 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 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, which should always be used with each separation 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

Metrohm recommends using a pulsation absorber (6.2620.150). Polymethacrylate columns and polyvinyl alcohol columns in particular must be protected from the brief pressure surges that inevitably occur when switching the valves.

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 is 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 if the separating efficiency 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), Metrohm recommends removing the separation column and sealing it with the stoppers provided. Rinse the ion chromatograph 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.

When you return the instrument to operation, rinse the ion chromatograph 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. 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 in significantly reduced quantity to avoid impacting the chromatography. Guard columns remove critical con-

taminants 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

Metrohm recommends 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 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 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 resistivity greater than 18.2 MΩ·cm and should be free of particles. Therefore, Metrohm recommends 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

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

3.1 Chemicals

Recommended chemicals

- Sodium carbonate
  Merck order number: 1.06393.1000
- Sodium hydrogen carbonate,
  Merck order number: 1.06329.1000
- Ultrapure water of type I (see ASTM D1193)
  Resistance > 18 MΩ·cm (25 °C)
  TOC < 10 µg/L

3.2 Production of standard eluent

To produce 2 L of the standard eluent with 5.0 mmol/L sodium carbonate and 0.2 mmol/L sodium hydrogen carbonate, the following steps must be carried out:

Producing 2 L of standard eluent

Required accessories

- Eluent bottle (6.1608.120)
- Bottle cap (6.1602.200) equipped with CO_2 adsorber
- Ultrapure water
- Sodium carbonate
- Sodium hydrogen carbonate

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 Weigh and add 1059.9 mg of sodium carbonate and 33.6 mg of sodium hydrogen carbonate.

5 Rinse the column during 2 to 3 hours with the eluent.

This eluent (5.0 mmol/L of sodium carbonate and 0.2 mmol/L of sodium hydrogen carbonate) and chemical suppression can be used to achieve background conductivity of < 17 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 guard column leaflet.

NOTICE

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

NOTICE

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 column leaflet and the the product information 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).

NOTICE

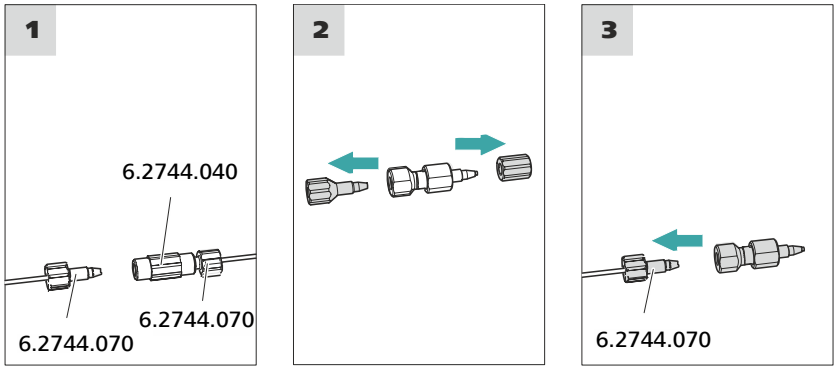
Only connect the guard column after the initial start-up of the instrument . Until then, replace the guard column and the separation column with couplings (6.2744.040).

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 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.

NOTICE

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 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 from your responsible Metrohm representative free of charge.

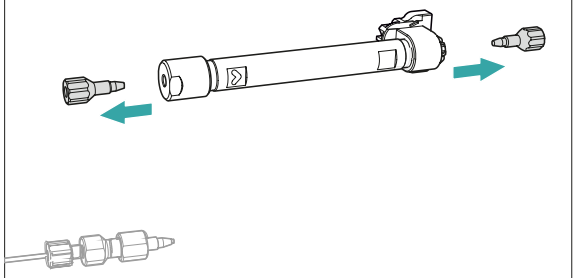
CAUTION

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).

NOTICE

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



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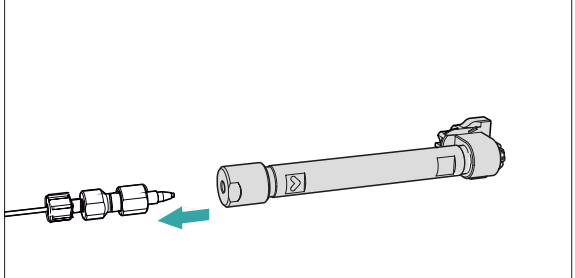

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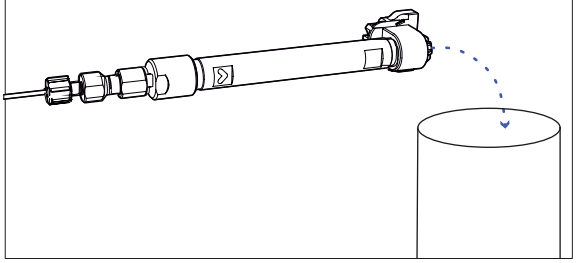

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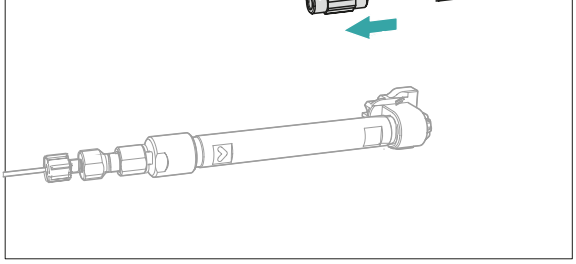

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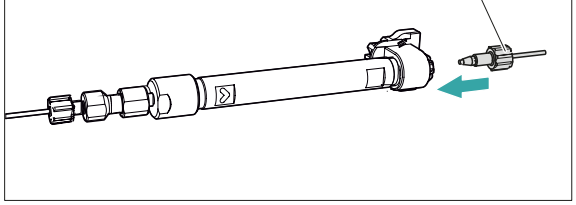

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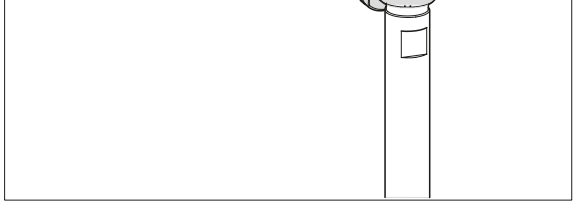


Connecting the separation column

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

NOTICE

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

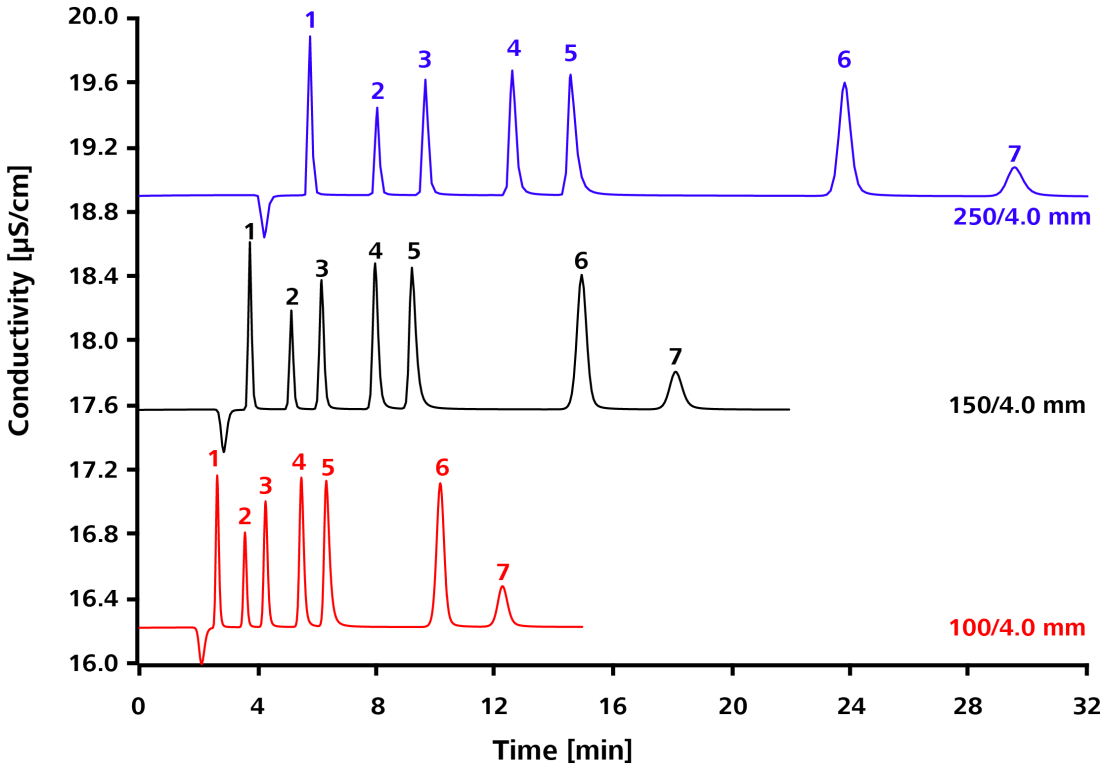
Continue rinsing the system with eluent until the desired stability level for the baseline has been attained .

The instrument is now ready for measuring samples.

5 Applications

5.1 Standard chromatogram

Columns:	Metrosep A Supp 17 - 100/4.0 Metrosep A Supp 17 - 150/4.0 Metrosep A Supp 17 - 250/4.0
Sample preparation:	-
Detection:	Conductivity
Suppression:	Chemical suppression with MSM A
Temperature:	25 °C
Flow rate:	0.6 mL/min
Loop:	10 µL
Eluent:	5.0 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$



Metrosep A Supp 17 - xx0/4.0		mg/L
1	Fluoride	2
2	Chloride	2
3	Nitrite	5
4	Bromide	10
5	Nitrate	10
6	Sulfate	10
7	Phosphate	10

5.2 Effects of temperature

Column: Metrosep A Supp 17 - 150/4.0

Sample preparation: -

Detection: Conductivity

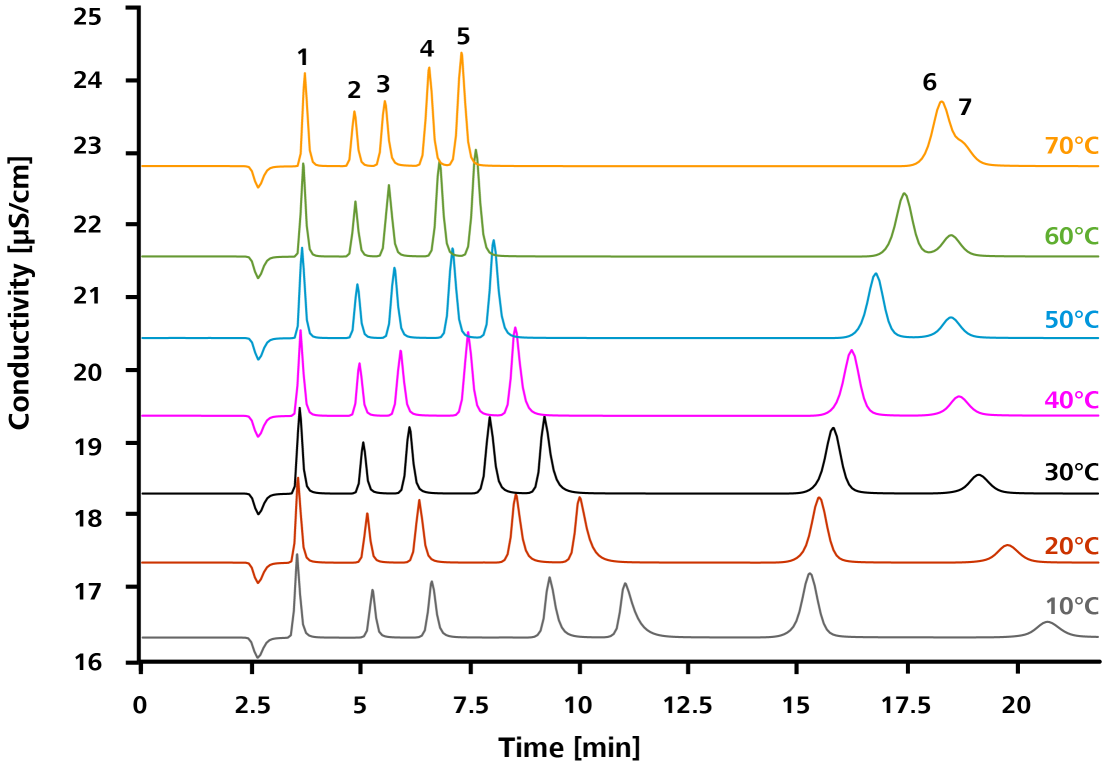
Suppression: Chemical suppression with MSM A

Temperature: 10 to 70 °C

Loop: 20 µL

Flow rate: 0.6 mL/min

Eluent: 5.0 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$



Metrosep A Supp 17 - 150/4.0		mg/L
1	Fluoride	2
2	Chloride	2
3	Nitrite	5
4	Bromide	10
5	Nitrate	10
6	Sulfate	10
7	Phosphate	10

An increase in temperature results in slightly shorter retention times of the monovalent ions. The shorter the retention time, the less the decrease. For nitrate, tailing decreases. Phosphate is only slightly influenced by temperature. The retention time first decreases and then stabilizes after 40 °C. The retention time of sulfate, however, increases until a co-elution with phosphate takes place at 70 °C.

5.3 Eluent flow rate variation

Metrosep A Supp 17 - 100/4.0

Column: Metrosep A Supp 17 - 100/4.0

Sample preparation: -

Detection: Conductivity

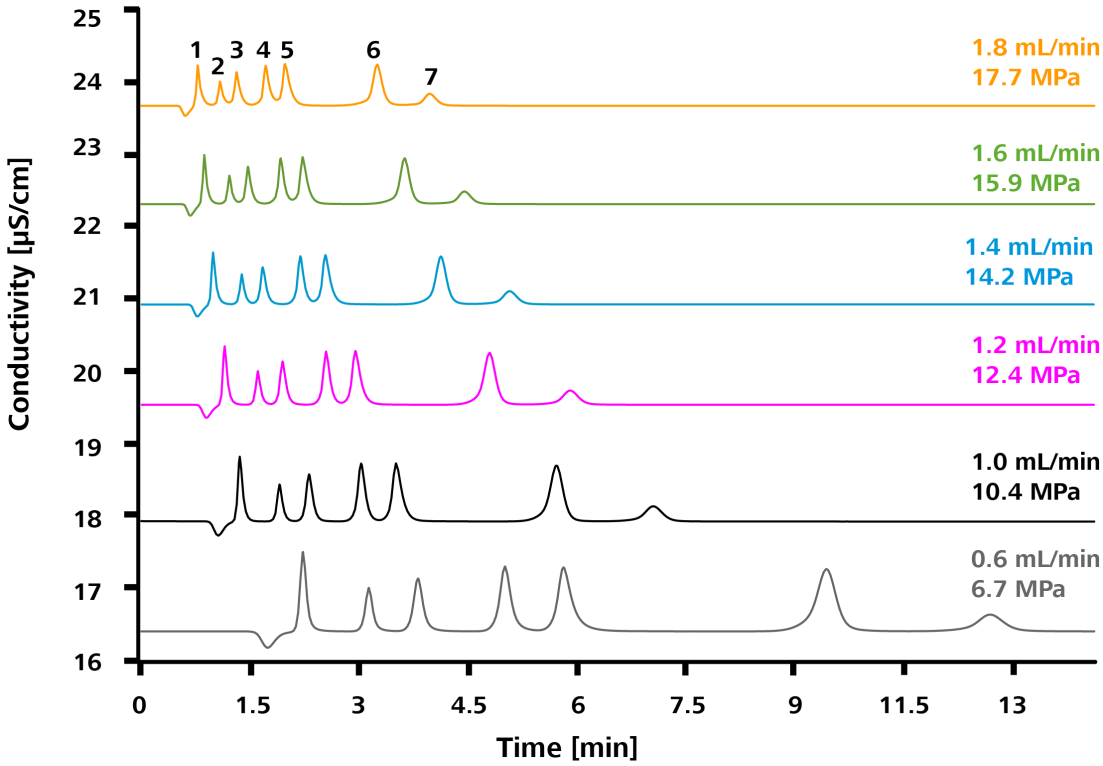
Suppression: Chemical suppression with MSM A

Temperature: 25 °C

Loop: 20 µL

Flow rate: 0.6 mL/min to 1.8 mL/min

Eluent: 5.0 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$



Metrosep A Supp 17 - 100/4.0		mg/L
1	Fluoride	2
2	Chloride	2

Metrosep A Supp 17 - 100/4.0		mg/L
3	Nitrite	5
4	Bromide	10
5	Nitrate	10
6	Sulfate	10
7	Phosphate	10

The retention times of all standard ions decrease with increasing flow rate. Fluoride approaches the injection peak.

Metrosep A Supp 17 - 250/4.0

Column: Metrosep A Supp 17 - 250/4.0

Sample preparation: -

Detection: Conductivity

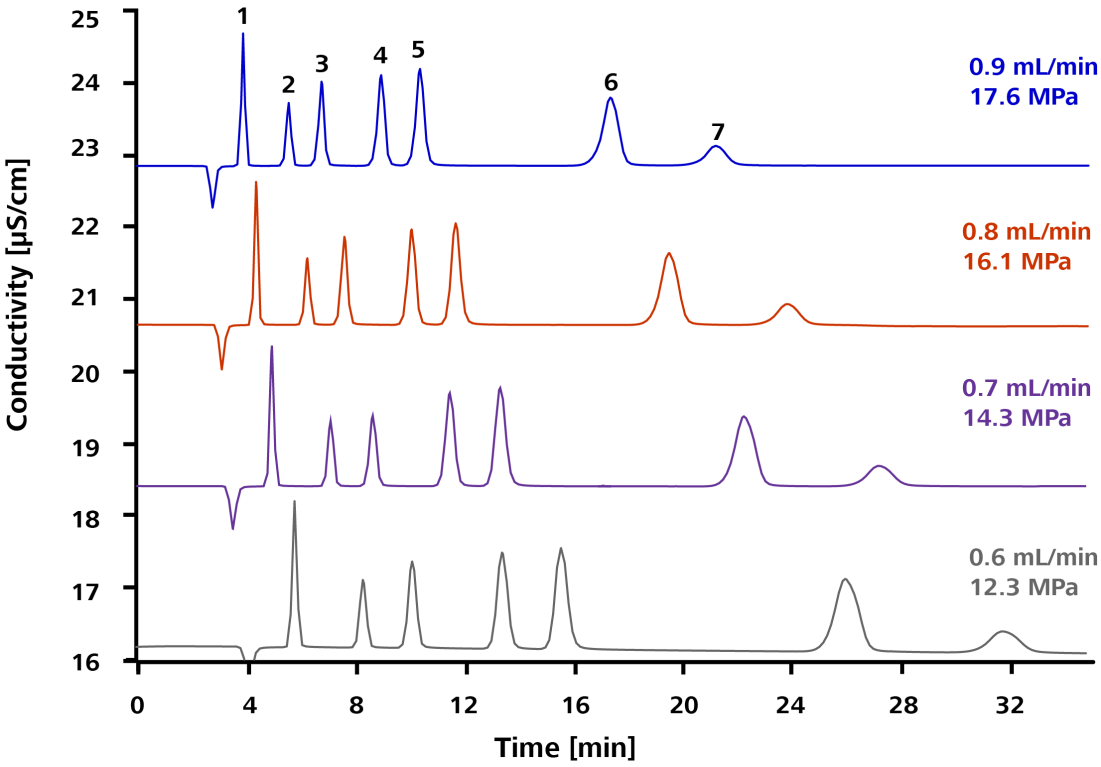
Suppression: Chemical suppression with MSM A

Temperature: 25 °C

Loop: 20 µL

Flow rate: 0.6 mL/min to 0.9 mL/min

Eluent: 5.0 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$



Metrosep A Supp 17 - 250/4.0		mg/L
1	Fluoride	2
2	Chloride	2
3	Nitrite	5
4	Bromide	10
5	Nitrate	10
6	Sulfate	10
7	Phosphate	10

The retention times of all standard ions decrease with increasing flow rate.

5.4 Variation of the eluent

Variation with constant Na_2CO_3/$NaHCO_3$ ratio

Column: Metrosep A Supp 17 - 150/4.0

Sample preparation: -

Detection: Conductivity

Suppression: Sequential suppression with MSM A and MCS

Temperature: 25 °C

Loop: 20 µL

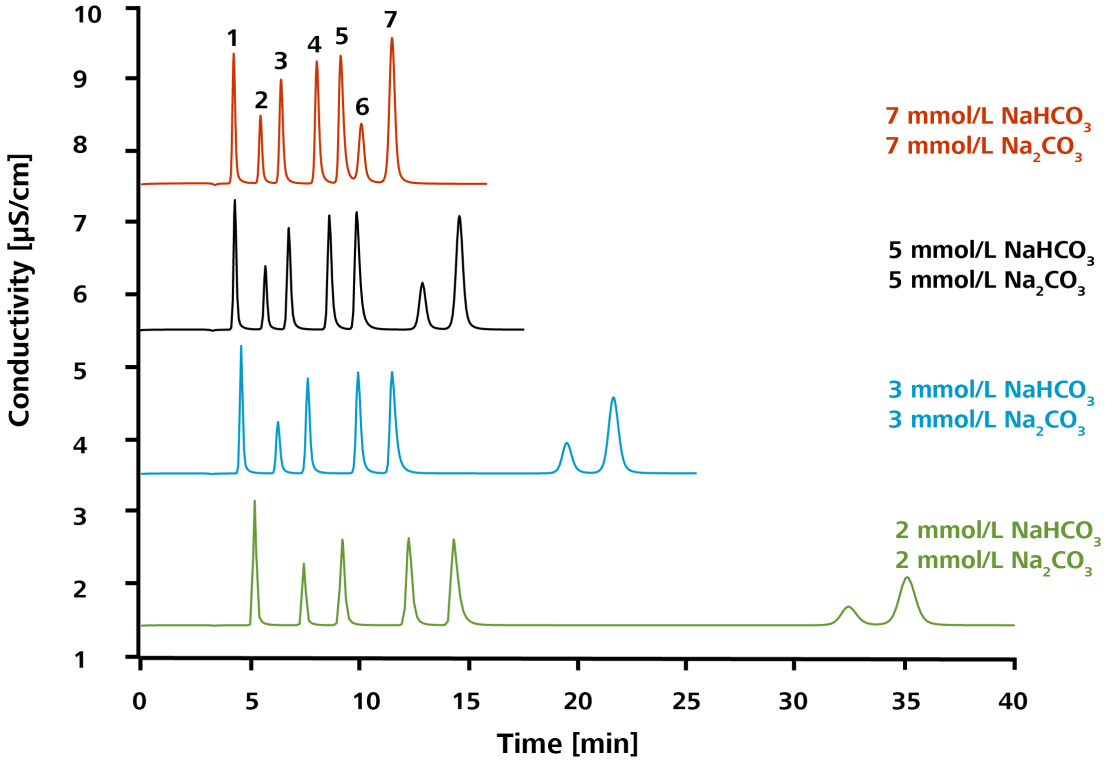
Flow rate: 0.6 mL/min

Eluent: A) 2 mmol/L of Na_2CO_3, 2 mmol/L of $NaHCO_3$

B) 3 mmol/L of Na_2CO_3, 3 mmol/L of $NaHCO_3$

C) 5 mmol/L of Na_2CO_3, 5 mmol/L of $NaHCO_3$

D) 7 mmol/L of Na_2CO_3, 7 mmol/L of $NaHCO_3$



Metrosep A Supp 17 - 150/4.0		mg/L
1	Fluoride	2
2	Chloride	2
3	Nitrite	5
4	Bromide	10
5	Nitrate	10
6	Phosphate	10
7	Sulfate	10

The retention time becomes shorter with increasing Na_2CO_3/$NaHCO_3$ concentration. The retention times for the polyvalent anions phosphate and sulfate are strongly shortened in particular. With 1 mmol/L of Na_2CO_3 / 1 mmol/L of $NaHCO_3$, phosphate co-elutes with sulfate with a retention time of 66 minutes (not shown).

$NaHCO_3$ variation with constant Na_2CO_3

Column: Metrosep A Supp 17 - 150/4.0

Sample preparation: -

Detection: Conductivity

Suppression: Sequential suppression with MSM A and MCS

Temperature: 25 °C

Loop: 20 µL

Flow rate: 0.6 mL/min

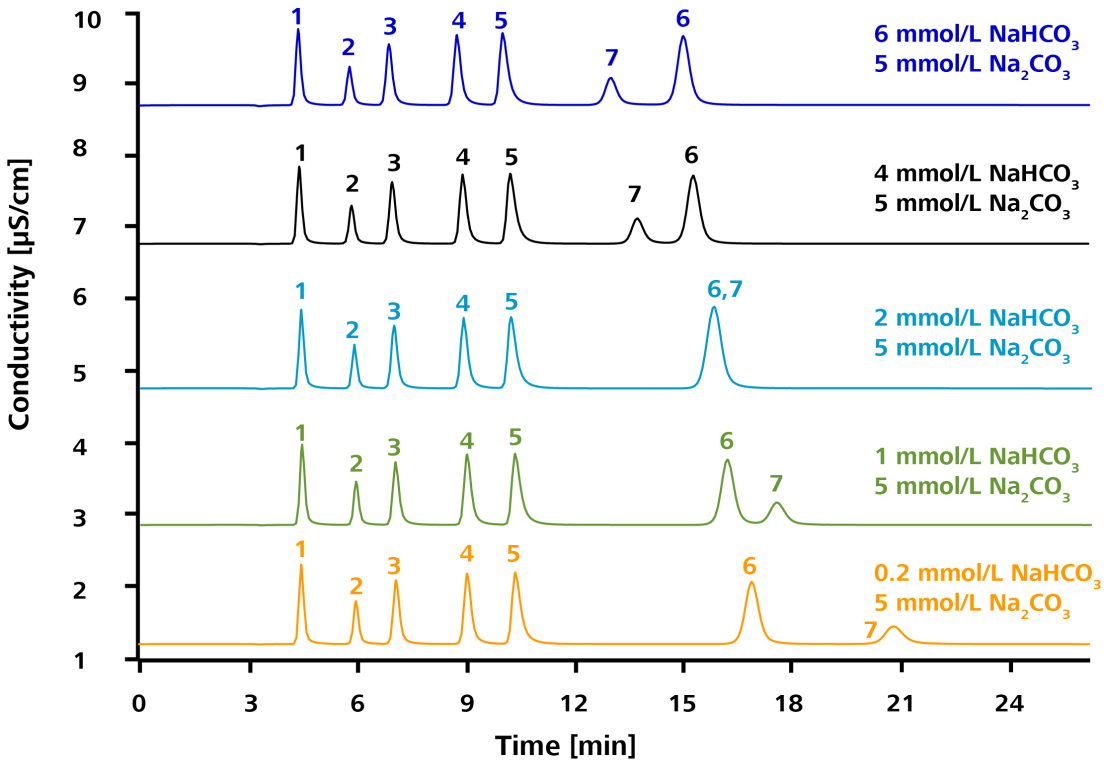
Eluent: A) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$

B) 5 mmol/L of Na_2CO_3, 1 mmol/L of $NaHCO_3$

C) 5 mmol/L of Na_2CO_3, 2 mmol/L of $NaHCO_3$

D) 5 mmol/L of Na_2CO_3, 4 mmol/L of $NaHCO_3$

E) 5 mmol/L of Na_2CO_3, 6 mmol/L of $NaHCO_3$



Metrosep A Supp 17 - 150/4.0		mg/L
1	Fluoride	2
2	Chloride	2
3	Nitrite	5
4	Bromide	10
5	Nitrate	10
6	Sulfate	10
7	Phosphate	10

When the $NaHCO_3$ concentration is increased, the retention time of the monovalent anions and sulfate decrease only slightly. For phosphate, however, the retention time decreases heavily, until phosphate and sulfate co-elute at 2 mmol/L of $NaHCO_3$. After 3 mmol/L of $NaHCO_3$, sulfate elutes even after phosphate. The effect decreases with increasing $NaHCO_3$ concentration.

Na_2CO_3 variation with constant $NaHCO_3$

Column: Metrosep A Supp 17 - 150/4.0

Sample preparation: -

Detection: Conductivity

Suppression: Sequential suppression with MSM A and MCS

Temperature: 25 °C

Loop: 20 µL

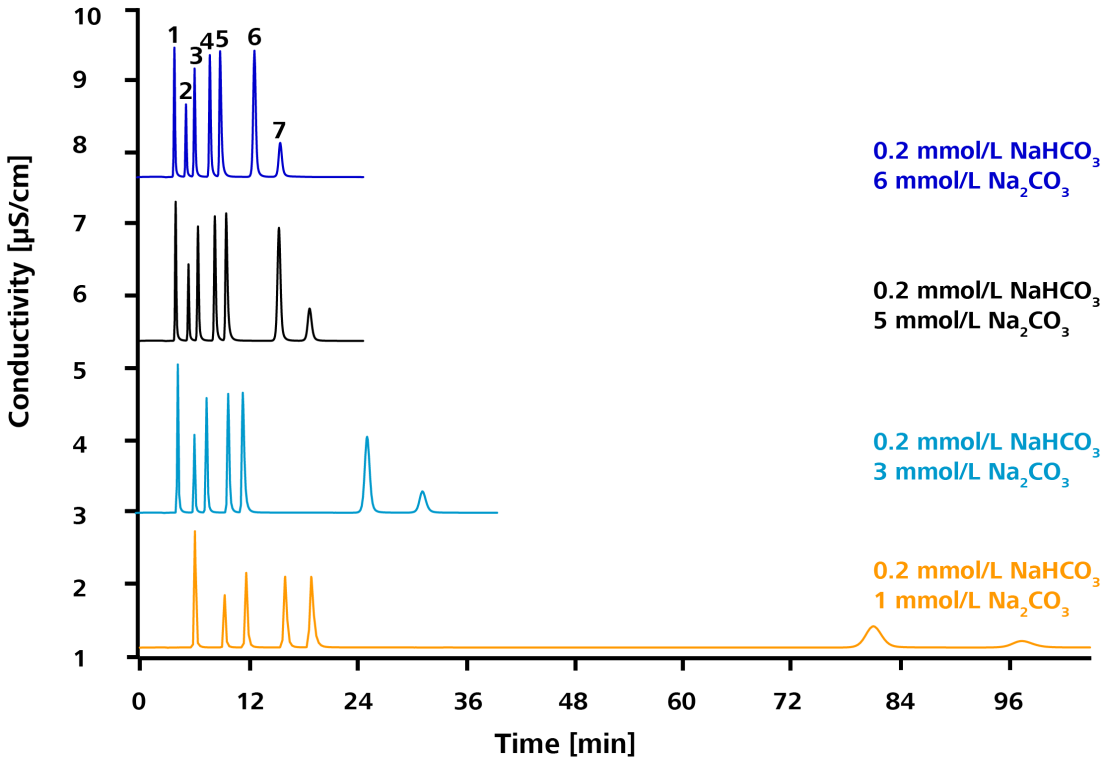
Flow rate: 0.6 mL/min

Eluent: A) 1 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$

B) 3 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$

C) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$

D) 6 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$



Metrosep A Supp 17 - 150/4.0		mg/L
1	Fluoride	2
2	Chloride	2
3	Nitrite	5
4	Bromide	10
5	Nitrate	10
6	Sulfate	10
7	Phosphate	10

The retention times of phosphate and sulfate can be shortened disproportionately by increasing the Na_2CO_3 concentration.

Variation of organic modifier: Acetone

Column: Metrosep A Supp 17 - 100/4.0

Sample preparation: -

Detection: Conductivity

Suppression: Chemical suppression with MSM A

Temperature: 25 °C

Loop: 20 µL

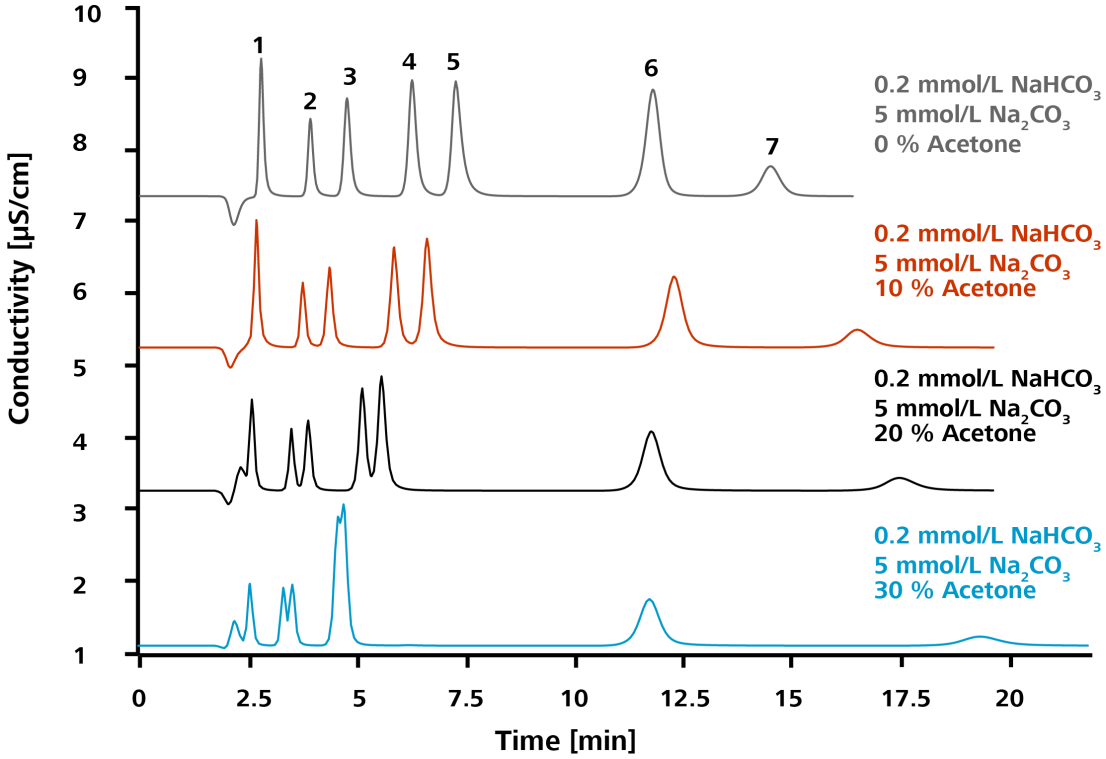
Flow rate: 0.6 mL/min

Eluent: A) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$, 0% acetone

B) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$, 10% acetone

C) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$, 20% acetone

D) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$, 30% acetone



Metrosep A Supp 17 - 100/4.0		mg/L
1	Fluoride	2
2	Chloride	2
3	Nitrite	5
4	Bromide	10
5	Nitrate	10
6	Sulfate	10
7	Phosphate	10

The backpressure increases gradually as acetone content increases. The pressure is 9.7 MPa at 10%. The retention time of phosphate increases. The resolution between bromide and nitrate as well as between chloride and nitrite decreases with an increasing acetone content. An interference peak forms at the injection peak with increasing solvent content.

Variation of organic modifier: Methanol

Column: Metrosep A Supp 17 - 100/4.0

Sample preparation: -

Detection: Conductivity

Suppression: Chemical suppression with MSM

Temperature: 25 °C

At 40% modifier, increase the temperature to 50 °C; otherwise, the back-pressure is too high.

Loop: 20 µL

Flow rate: 0.6 mL/min

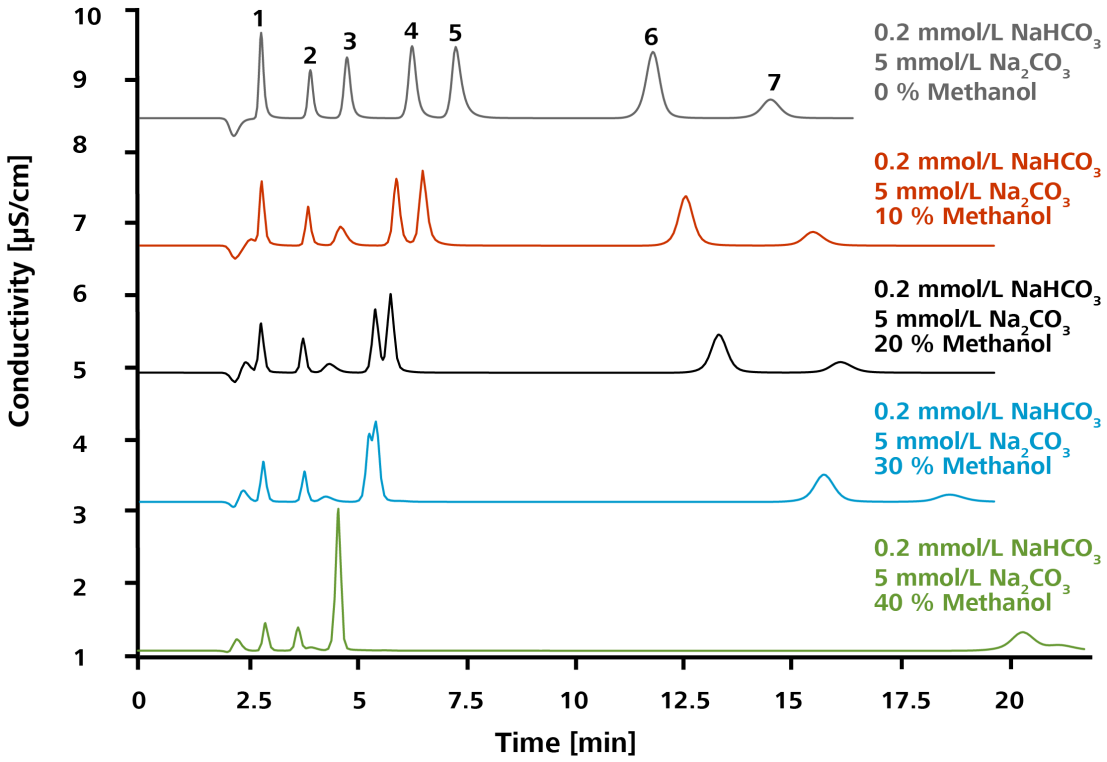
Eluent: A) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$, 0% methanol

B) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$, 10% methanol

C) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$, 20% methanol

D) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$, 30% methanol

E) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$, 40% methanol



Metrosep A Supp 17 - 100/4.0		mg/L
1	Fluoride	2
2	Chloride	2
3	Nitrite	5
4	Bromide	10
5	Nitrate	10
6	Sulfate	10
7	Phosphate	10

The backpressure increases gradually as methanol content increases. The pressure is 6.9 MPa at 10%. The retention time of phosphate and sulfate increases and the peaks merge at 40% methanol. The resolution between bromide and nitrate as well as between chloride and nitrite decreases with increasing methanol content. They co-elute after 40%. An interference peak forms at the injection peak with increasing solvent content.

Variation of organic modifier: Acetonitrile

Column: Metrosep A Supp 17 - 100/4.0

Sample preparation: -

Detection: Conductivity

Suppression: Chemical suppression with MSM A

Temperature: 25 °C

Loop: 20 µL

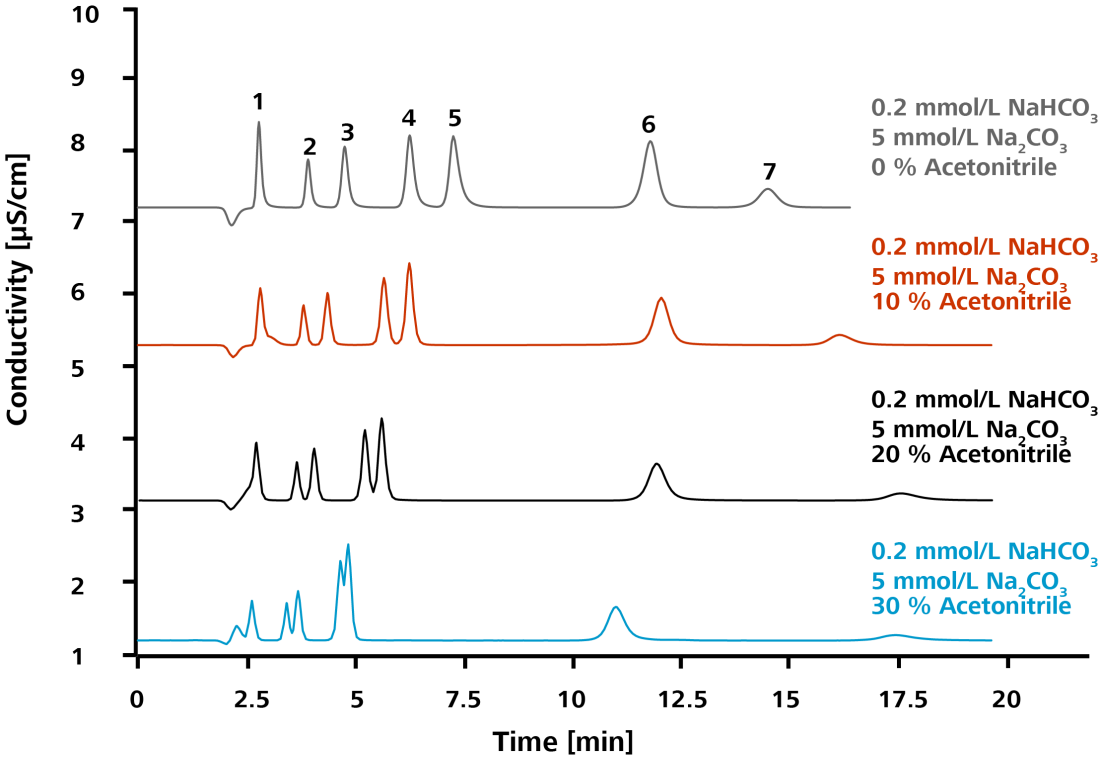
Flow rate: 0.6 mL/min

Eluent: A) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$, 0% acetonitrile

B) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$, 10% acetonitrile

C) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$, 20% acetonitrile

D) 5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$, 30% acetonitrile



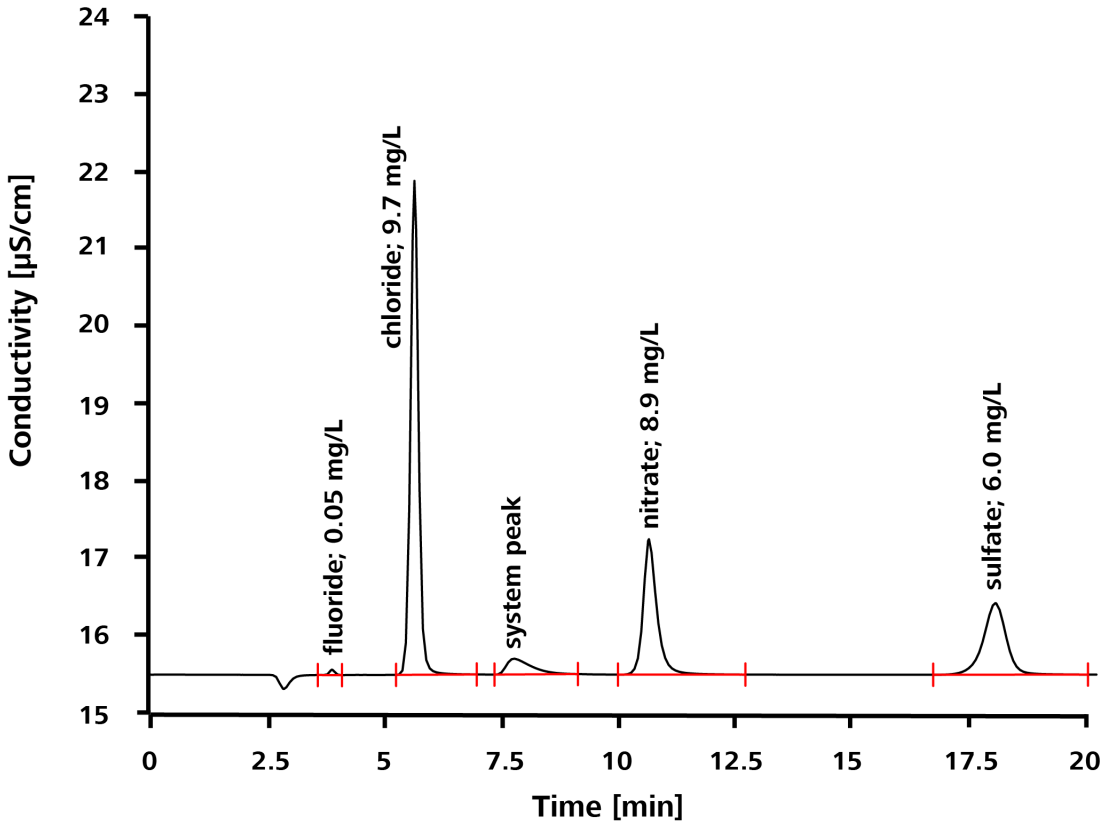
Metrosep A Supp 17 - 100/4.0		mg/L
1	Fluoride	2
2	Chloride	2
3	Nitrite	5
4	Bromide	10
5	Nitrate	10
6	Sulfate	10
7	Phosphate	10

The backpressure increases with increasing acetonitrile content. The pressure is 8.6 MPa at 10%. The retention time of phosphate increases. The resolution between bromide and nitrate as well as between chloride and nitrite decreases with an increasing acetonitrile content. An interference peak forms at the injection peak with increasing solvent content.

5.5 Water analysis

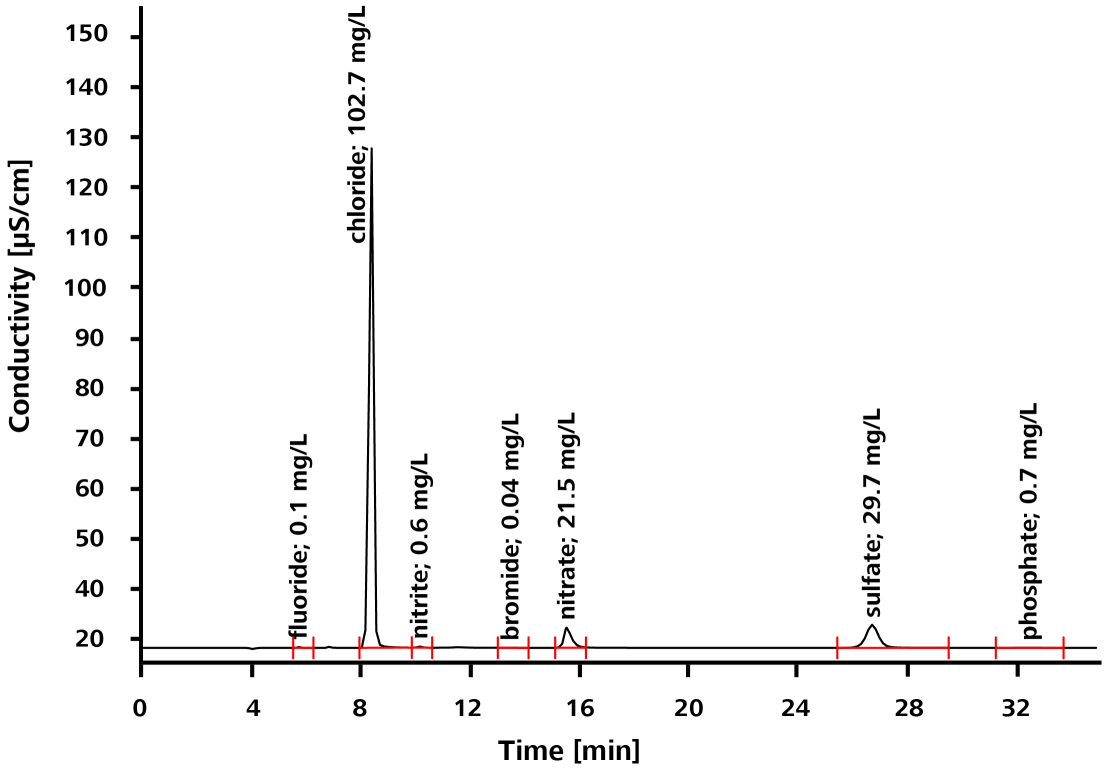
Drinking water analysis

Column:	Metrosep A Supp 17 - 150/4.0
Sample preparation:	Metrohm Inline Ultrafiltration
Detection:	Conductivity
Suppression:	Chemical suppression with MSM A
Temperature:	25 °C
Loop:	10 µL
Flow rate:	0.6 mL/min
Eluent:	5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$



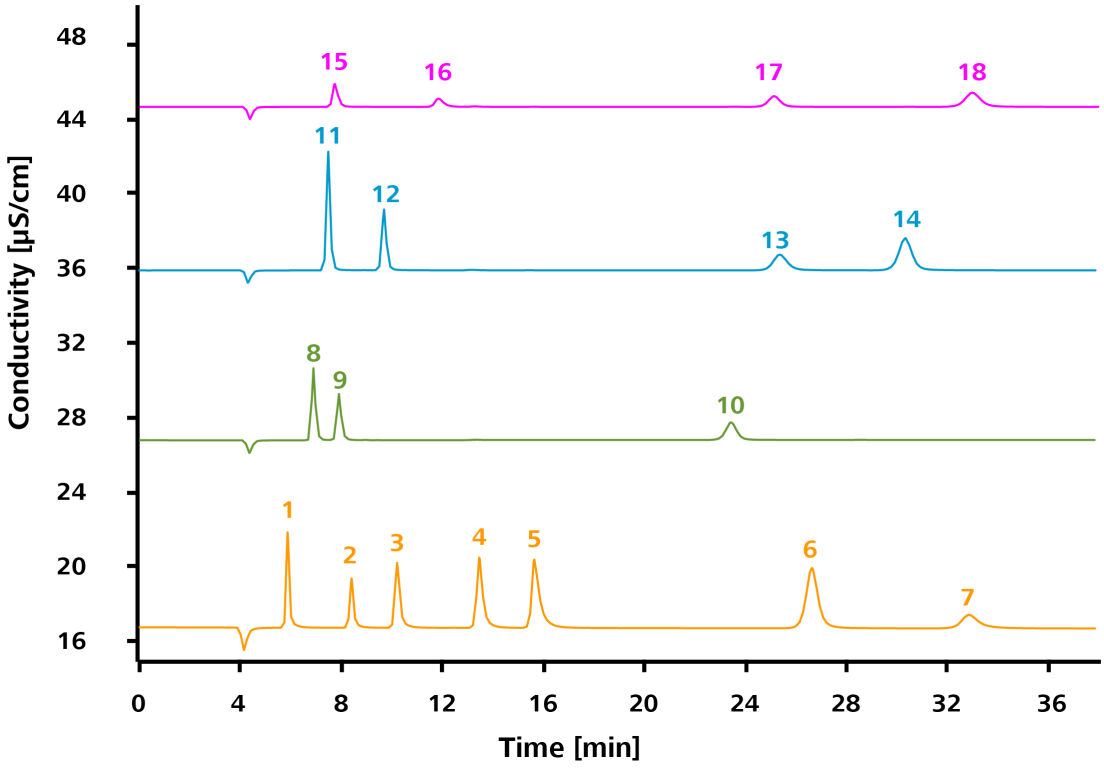
Wastewater analysis

Column:	Metrosep A Supp 17 - 250/4.0
Sample preparation:	Metrohm Inline Ultrafiltration
Detection:	Conductivity
Suppression:	Chemical suppression with MSM A
Temperature:	25 °C
Loop:	10 µL
Flow rate:	0.6 mL/min
Eluent:	5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$



5.6 Organic acids

Column:	Metrosep A Supp 17 - 250/4.0
Sample preparation:	-
Detection:	Conductivity
Suppression:	Chemical suppression with MSM A
Temperature:	25 °C
Loop:	10 µL
Flow rate:	0.6 mL/min
Eluent:	5 mmol/L of Na_2CO_3, 0.2 mmol/L of $NaHCO_3$



Metrosep A Supp 17 - 250/4.0		mg/L
1	Fluoride	2
2	Chloride	2
3	Nitrite	5
4	Bromide	10
5	Nitrate	10
6	Sulfate	10
7	Phosphate	10
8	Glycolate	20
9	Lactate	20
10	Malate	20
11	Formate	20
12	Methanesulfonic acid	20
13	Tartrate	20
14	Oxalate	20
15	Acetate	20
16	Propionate	20

Metrosep A Supp 17 - 250/4.0		mg/L
17	Succinate	20
18	Maleate	20

Glycolate shows a good separation from fluoride as well as from acetate/formate. The separation of acetate/formate is not sufficient. Methanesulfonic acid elutes a bit earlier than nitrite. Propionate elutes between nitrite and bromide. At the back, oxalate elutes nicely between sulfate and phosphate.

5.7 Alternative eluent for standard anions

Column: Metrosep A Supp 17 - 100/4.0

Sample preparation: -

Detection: Conductivity

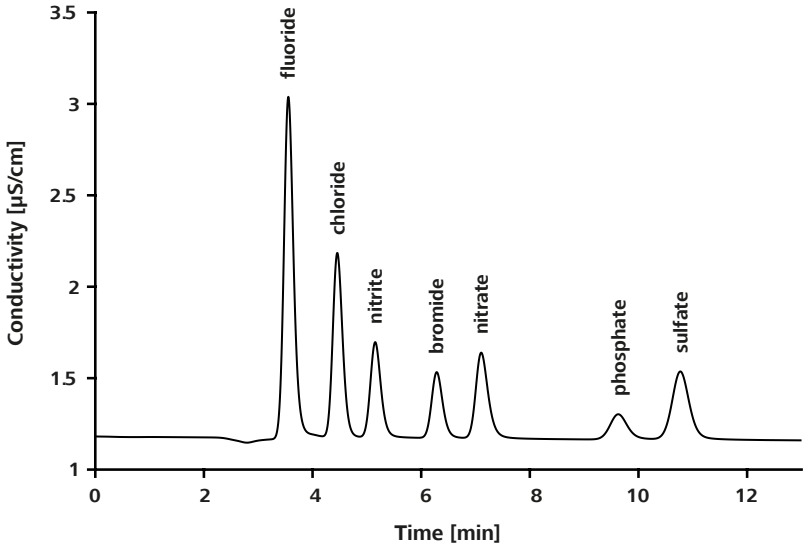
Suppression: Sequential suppression with MSM A and MCS

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.6 mL/min

Eluent: 5 mmol/L of Na_2CO_3, 5 mmol/L of $NaHCO_3$



Metrosep A Supp 17 - 250/4.0		mg/L
1	Fluoride	1
2	Chloride	1
3	Nitrite	1
4	Bromide	1
5	Nitrate	1
6	Phosphate	1
7	Sulfate	1

Combining the Metrosep A Supp 10 standard eluent with the Metrosep A Supp 17 column reduces the chromatogram duration by shifting the phosphate peak between the nitrate peak and the sulfate peak. Under these conditions, the carbonate peak shifts to the same retention time as chloride. Take this into account particularly when analyzing samples with a high carbonate content. For the Metrosep A Supp 17 - 100/4.0 and the Metrosep A Supp 17 - 150/4.0, the capacity of the MSM A is sufficient for the entire determination. For the Metrosep A Supp 17 - 250/4.0, use an MSM-HC A.

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

- 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. Only regenerate the separation column as described below if this measure does not help.

NOTICE

Ensure that the maximum pressure is never exceeded during regeneration.

If the pressure becomes too high, reduce the flow rate.

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:

- Inorganic contamination *(see table 3, page 37)*
- Organic contamination *(see table 4, page 37)*

Table 3 Inorganic contamination

		Duration	Flow rate 4 mm
1.	Rinse with ultrapure water	20 min	0.3 mL/min
2.	Rinse with 10x concentrated eluent	120 min	0.3 mL/min
3.	Rinse with ultrapure water	20 min	0.3 mL/min
4.	Rinse with eluent	120 min	0.3 mL/min

Table 4 Organic contamination

		Duration	Flow rate 4 mm
1.	Rinse with 70% methanol	16 h	0.3 mL/min
2.	Rinse with eluent	120 min	0.3 mL/min

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 a 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 they do not, replace those capillaries with thinner capillaries. ▪ Check that all of the 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 / 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. Backpressure should 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/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 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:

- Application Note S-353 Drinking water analysis using the Eco IC and the Metrosep A Supp 17 - 150/4.0
- Application Note S-354 Waste water analysis using the Eco IC and the Metrosep A Supp 17 - 250/4.0
- Monograph: Analysis of water samples and water constituents with Metrohm instruments, page 73ff. (8.038.5003)
- Column catalog, 8.000.5194

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