Column manual

Metrosep A Supp 7 (6.1006.6X0)

Manual

8.107.8046EN / v2 / 2024-05-01

Metrohm

Metrohm AG
CH-9100 Herisau
Switzerland
Phone +41 71 353 85 85
Fax +41 71 353 89 01
info@metrohm.com
www.metrohm.com

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Technische Dokumentation
Metrohm AG
CH-9100 Herisau
techdoc@metrohm.com

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 suitable for determining the byproducts from water treatment in particular. The oxyhalides can be determined simultaneously with the standard anions using sequential suppression followed by conductivity detection. The column is also suitable for the separation of organic acids such as glycolate, acetate and formate.

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
6.1006.620	Metrosep A Supp 7 - 150/4.0
6.1006.630	Metrosep A Supp 7 - 250/4.0

Table 2 2-mm columns

Order number	Designation
6.1006.640	Metrosep A Supp 7 - 150/2.0
6.1006.650	Metrosep A Supp 7 - 250/2.0

Table 3 4-mm guard columns

Order number	Designation
6.1006.500	Metrosep A Supp 4/5 Guard/4.0
6.1006.540	Metrosep A Supp 4/5 S-Guard/4.0
6.1011.030	Metrosep RP 2 Guard/3.5
6.1031.500	Metrosep A Supp 16 Guard/4.0
6.1031.510	Metrosep A Supp 16 S-Guard/4.0

Table 4 2-mm guard columns

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

Order number	Designation
6.1031.600	Metrosep A Supp 16 Guard/2.0
6.1031.610	Metrosep A Supp 16 S-Guard/2.0

1.2 Technical specifications

Column material Polyvinyl alcohol with quaternary ammonium groups

Particle size 5 µm

Dimensions

Order number	Dimensions
6.1006.620	150 x 4.0 mm
6.1006.630	250 x 4.0 mm
6.1006.640	150 x 2.0 mm
6.1006.650	250 x 2.0 mm

pH range 3 to 12

Temperature range 20 to 60 °C

Recommended standard temperature 45 °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
6.1006.620	0.7 mL/min	1.0 mL/min
6.1006.630	0.7 mL/min	1.0 mL/min
6.1006.640	0.2 mL/min	0.6 mL/min
6.1006.650	0.2 mL/min	0.4 mL/min

Standard eluent 3.6 mmol/L sodium carbonate

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
6.1006.620	76 µmol (Cl^-)
6.1006.630	110 µmol (Cl^-)
6.1006.640	18 µmol (Cl^-)
6.1006.650	27 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 at 4 to 8 °C.

Typical pressure

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

Order number	Typical pressure
6.1006.620	5.8 ± 2 MPa
6.1006.630	9.6 ± 2 MPa
6.1006.640	5.2 ± 2 MPa
6.1006.650	9.0 ± 2 MPa

Column body

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

Application

Determination of inorganic anions and oxyhalides including dichloroacetic acid 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 or acetonitrile to the eluent.

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.

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 sodium hydroxide eluents. The eluent develops a strong elution strength 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 humidity and carbon dioxide from getting into the eluent. The adsorber

tube is usually filled with a 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, 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 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 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 the guard columns, which should always be used with each 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 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 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 rinsed properly 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 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 up 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

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 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 also be insufficient. 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 resistance 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
  Merck order number: 1.06393.1000
- 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 3.6 mmol/L sodium carbonate, the following steps must be carried out:

Producing 2 L of standard eluent

Required accessories

- Eluent bottle (6.1608.120)
- Cap (6.1602.200) equipped with CO_2 adsorber
- Ultrapure water
- Sodium 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 763 mg sodium carbonate.

This eluent (3.6 mmol/L of sodium carbonate) and sequential suppression can be used to achieve a background conductivity of < 1 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.

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

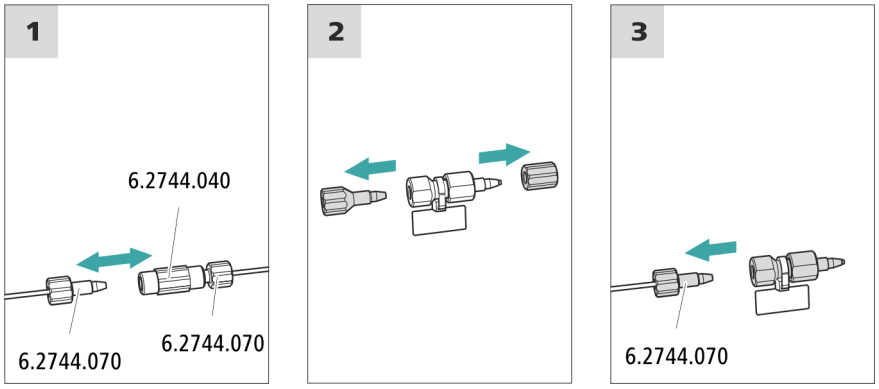
The guard column may not be connected until after the instrument has already been put into operation once . The guard column and the separation column have to be replaced by a coupling (6.2744.040) until then.

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.

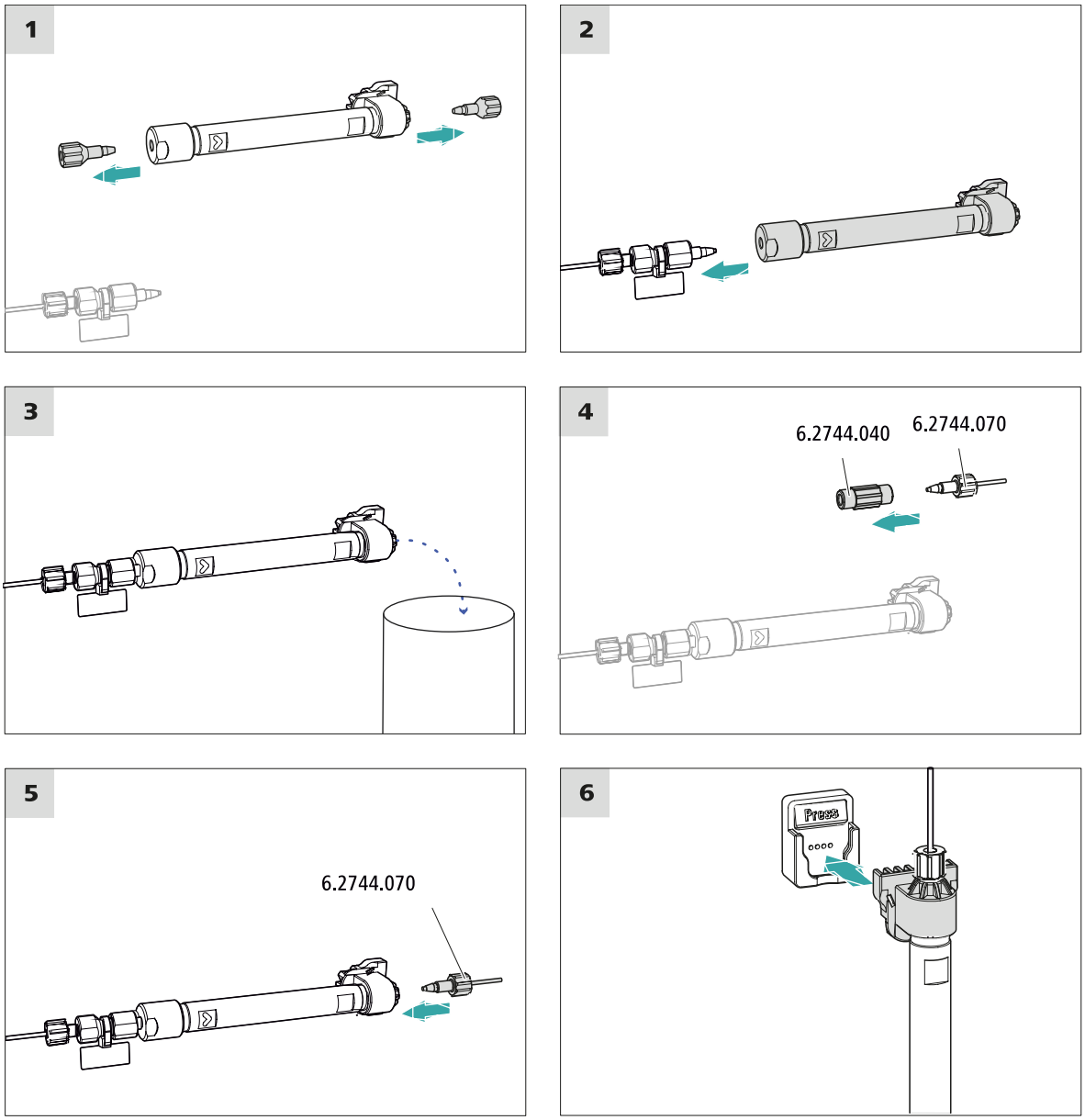
A test chromatogram and a leaflet accompanies every column. Detailed information on special IC applications can be found in the corresponding "**Application Bulletins**" or "**Application Notes**". You can find these online at *http://www.metrohm.com* in the Applications area or request them from your 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: 45 °C

Flow rate: 4 mm: 0.7 mL/min
2 mm: 0.2 mL/min

Loop: 4 mm: 20 µL
2 mm: 10 µL

Eluent: 3.6 mmol/L Na_2CO_3

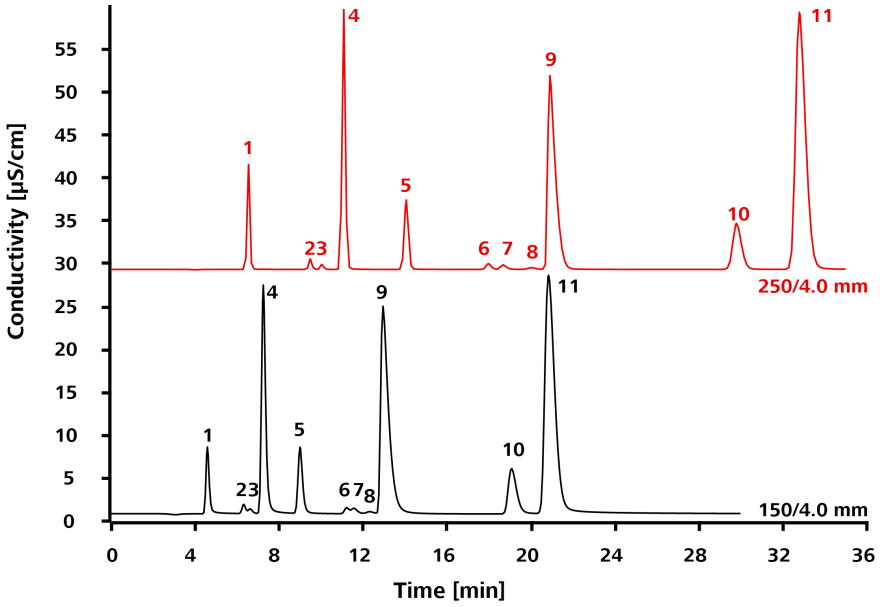


Figure 1 Metrosep A Supp 7 - X50/4.0

	Metrosep A Supp 7 x50/4.0	mg/L
1	Fluoride	2
2	Chlorite	1

	Metrosep A Supp 7 x50/4.0	mg/L
3	Bromate	1
4	Chloride	10
5	Nitrite	5
6	Bromide	1
7	Chlorate	1
8	Dichloroacetic acid	1
9	Nitrate	30
10	Phosphate	15
11	Sulfate	40

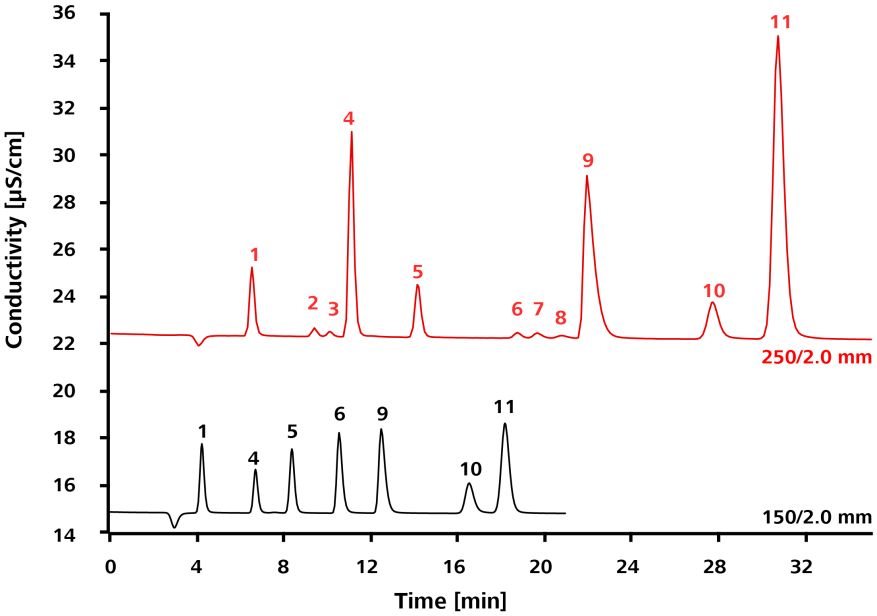


Figure 2 Metrosep A Supp 7 - X50/2.0

	Metrosep A Supp 7 x50/2.0	mg/L
1	Fluoride	2
2	Chlorite	1
3	Bromate	1
4	Chloride	10
5	Nitrite	5
6	Bromide	1
7	Chlorate	1
8	Dichloroacetic acid	1
9	Nitrate	30
10	Phosphate	15

	Metrosep A Supp 7 x50/2.0	mg/L
11	Sulfate	40

5.2 Effects of temperature

Column: Metrosep A Supp 7 - 250/4.0

Sample preparation: -

Detection: Conductivity

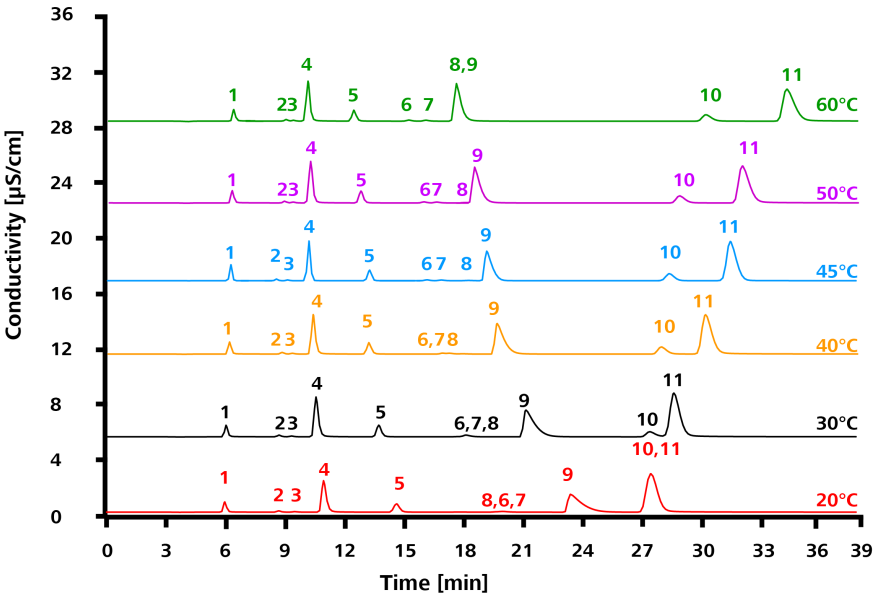
Suppression: Sequential suppression with MSM and MCS

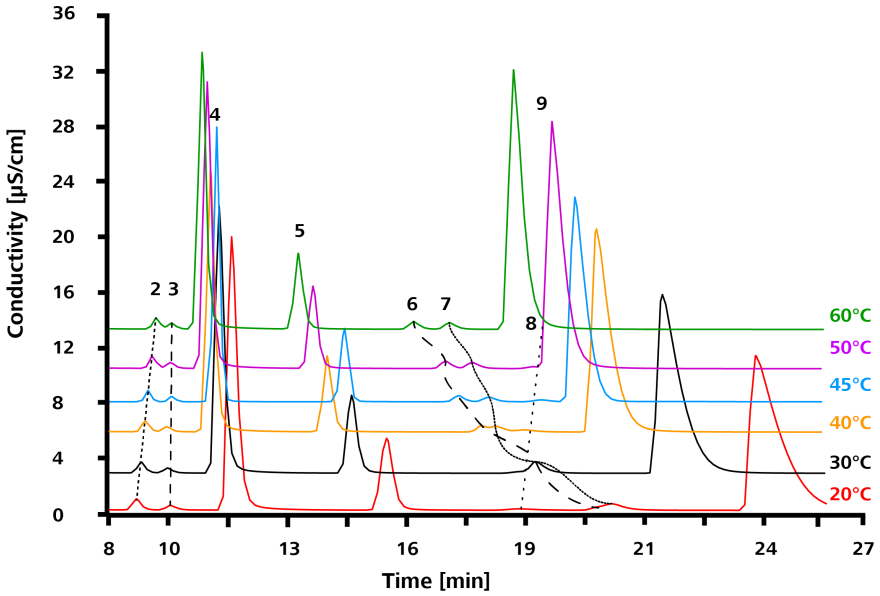
Temperature: 20 to 60 °C

Loop: 50 µL

Flow rate: 0.7 mL/min

Eluent: 3.6 mmol/L Na_2CO_3





	Metrosep A Supp 7 250/4.0	mg/L
1	Fluoride	2
2	Chlorite	1
3	Bromate	1
4	Chloride	10
5	Nitrite	5
6	Bromide	1
7	Chlorate	1
8	Dichloroacetic acid	1
9	Nitrate	30
10	Phosphate	15
11	Sulfate	40

An increase in temperature results in longer retention times of polyvalent ions such as phosphate (10) and sulfate (11). The retention time of the other standard anions is slightly shorter. Nitrate (9) is an exception as its retention time shortens strongly. At the same time, the peak symmetry of Nitrate improves.

The separation between Chlorite (2) and Bromate (3) is bigger at cooler temperatures. The separation between Bromide (6) and Chlorate (7), however, is better at higher temperatures. Dichloroacetic acid (8) is barely influenced by temperature.

5.3 Eluent flow rate variation

Column: Metrosep A Supp 7 - 150/4.0

Sample preparation: -

Detection: Conductivity

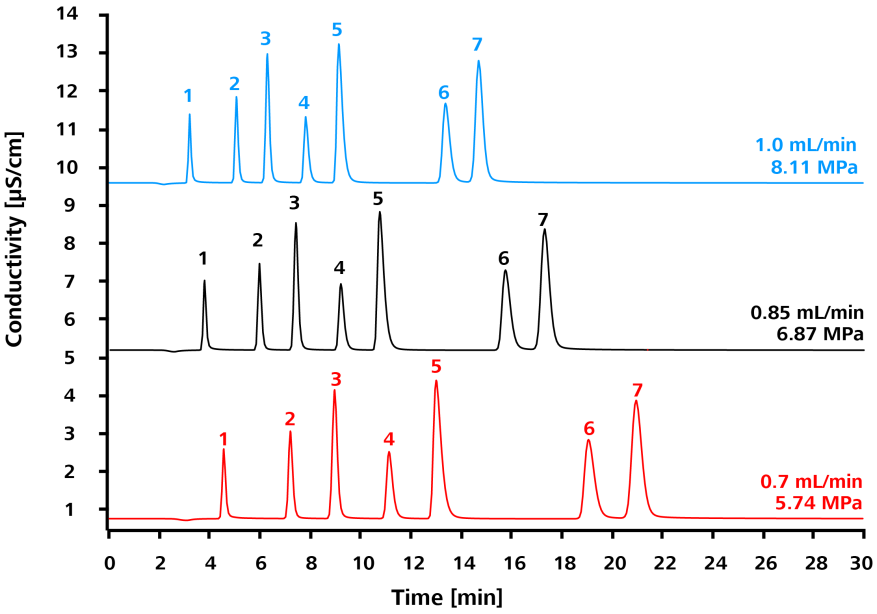
Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 50 µL

Flow rate: 0.7 mL/min, 0.85 mL/min, 1.0 mL/min

Eluent: 3.6 mmol/L Na_2CO_3



	Metrosep A Supp 7 - 150/4.0	mg/L
1	Fluoride	1
2	Chloride	2
3	Nitrite	5
4	Bromide	5
5	Nitrate	10
6	Phosphate	15
7	Sulfate	10

5.4 Variation of the eluent

Variation with constant Na_2CO_3/$NaHCO_3$ ratio

Column: Metrosep A Supp 7 - 150/4.0

Sample preparation: -

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 20 µL

Flow rate: 0.7 mL/min

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

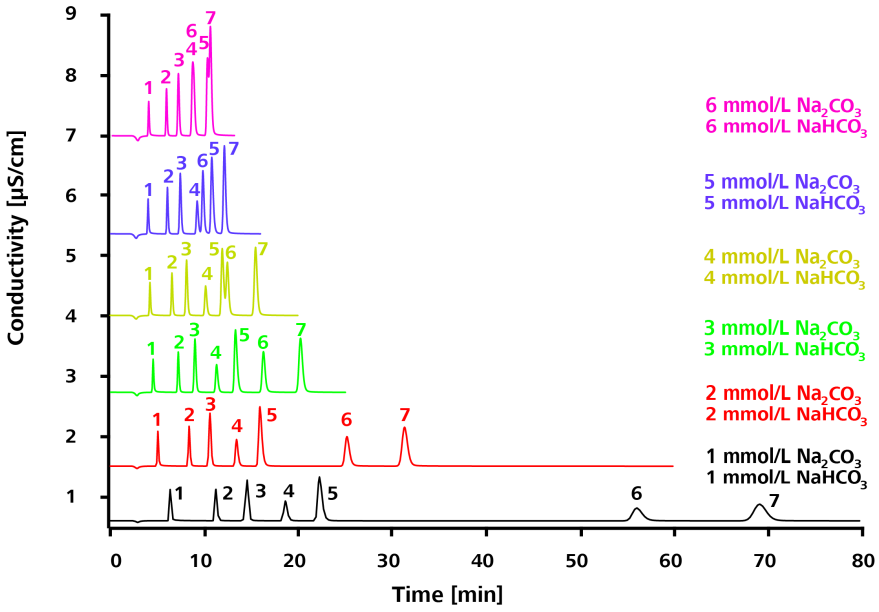
B) 2 mmol/L of Na_2CO_3, 2 mmol/L of $NaHCO_3$

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

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

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

F) 6 mmol/L of Na_2CO_3, 6 mmol/L of $NaHCO_3$



	Metrosep A Supp 7 - 150/4.0	mg/L
1	Fluoride	1
2	Chloride	2
3	Nitrite	5
4	Bromide	5
5	Nitrate	10
6	Phosphate	15
7	Sulfate	10

The retention time becomes shorter with increasing Na_2CO_3/$NaHCO_3$ concentration. Those for the polyvalent anions phosphate and sulfate are strongly shortened in particular. With 5 mmol/L Na_2CO_3 / 5 mmol/L $NaHCO_3$, phosphate (6) elutes nicely between bromide (4) and nitrate (5). With 6 mmol/L Na_2CO_3 / 6 mmol/L $NaHCO_3$, bromide (4) co-elutes with phosphate (6) and nitrate (5) with sulfate (7).

$NaHCO_3$ variation with constant Na_2CO_3

Column: Metrosep A Supp 7 - 150/4.0

Sample preparation: -

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 20 µL

Flow rate: 0.7 mL/min

Eluent:

A) 1 mmol/L of Na_2CO_3, 1 mmol/L of $NaHCO_3$

B) 1 mmol/L of Na_2CO_3, 2 mmol/L of $NaHCO_3$

C) 1 mmol/L of Na_2CO_3, 3 mmol/L of $NaHCO_3$

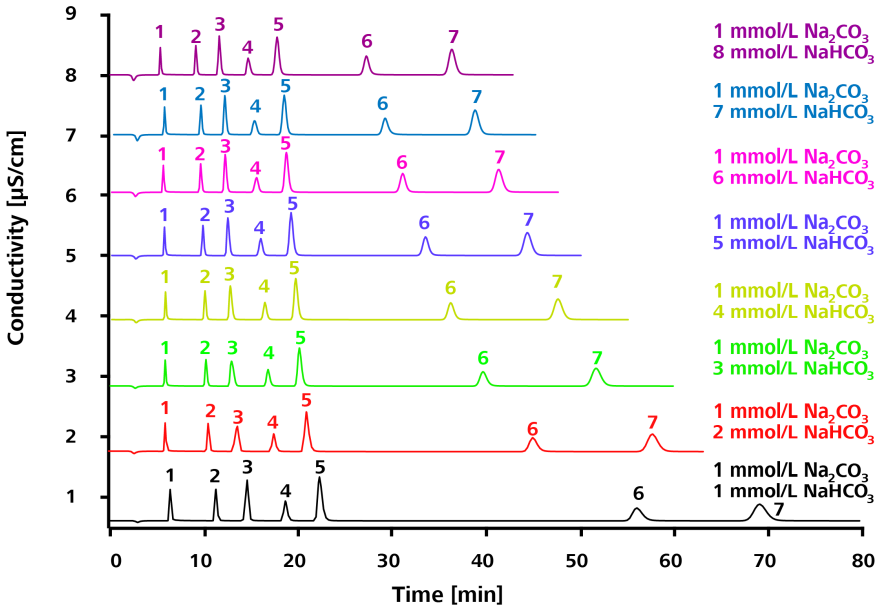
D) 1 mmol/L of Na_2CO_3, 4 mmol/L of $NaHCO_3$

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

F) 1 mmol/L of Na_2CO_3, 6 mmol/L of $NaHCO_3$

G) 1 mmol/L of Na_2CO_3, 7 mmol/L of $NaHCO_3$

H) 1 mmol/L of Na_2CO_3, 8 mmol/L of $NaHCO_3$



	Metrosep A Supp 7 - 150/4.0	mg/L
1	Fluoride	1
2	Chloride	2
3	Nitrite	5
4	Bromide	5
5	Nitrate	10
6	Phosphate	15
7	Sulfate	10

The retention times of monovalent anions decrease only slightly. For phosphate (6) and sulfate (7), however, the retention decreases evidently. This effect weakens with increasing $NaHCO_3$ concentration.

Na_2CO_3 variation with constant $NaHCO_3$

Column: Metrosep A Supp 7 - 150/4.0

Sample preparation: -

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 20 µL

Flow rate: 0.7 mL/min

Eluent:

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

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

C) 3 mmol/L of Na_2CO_3, 5 mmol/L of $NaHCO_3$

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

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

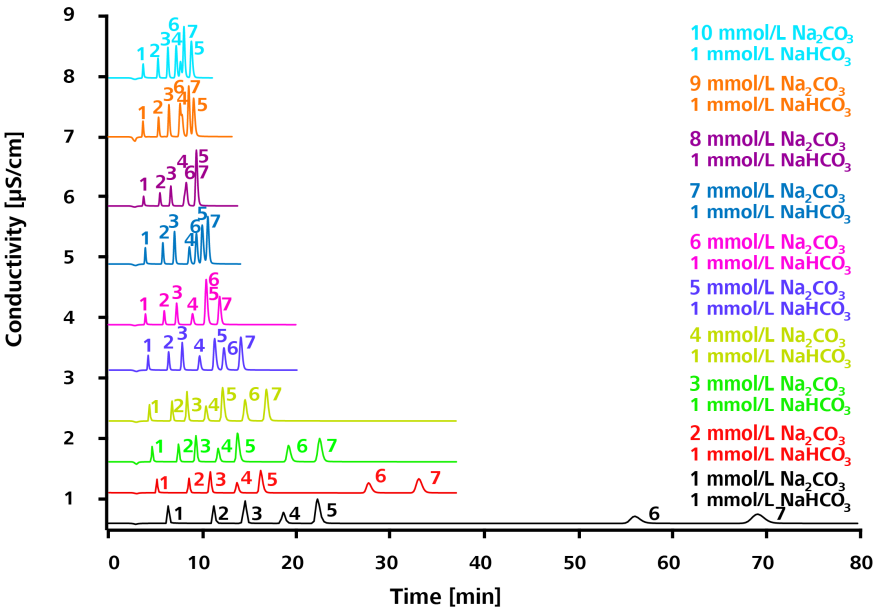
F) 6 mmol/L of Na_2CO_3, 5 mmol/L of $NaHCO_3$

G) 7 mmol/L of Na_2CO_3, 5 mmol/L of $NaHCO_3$

H) 8 mmol/L of Na_2CO_3, 5 mmol/L of $NaHCO_3$

I) 9 mmol/L of Na_2CO_3, 5 mmol/L of $NaHCO_3$

J) 10 mmol/L of Na_2CO_3, 5 mmol/L of $NaHCO_3$



	Metrosep A Supp 7 - 150/4.0	mg/L
1	Fluoride	1
2	Chloride	2
3	Nitrite	5
4	Bromide	5
5	Nitrate	10
6	Phosphate	15
7	Sulfate	10

The retention times of phosphate (6) and sulfate (7) can be shortened disproportionately by increasing the Na_2CO_3 concentration. At 10 mmol/L Na_2CO_3, nitrate (5) elutes even after phosphate (6) and sulfate (7).

Variation of organic modifier: Acetone

Column: Metrosep A Supp 7 - 150/4.0

Sample preparation: -

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 10 µL

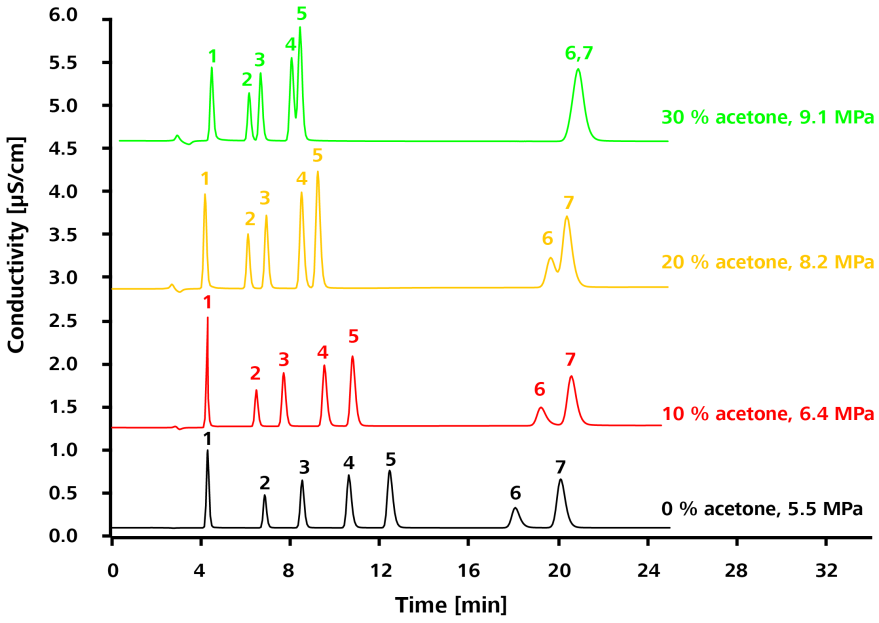
Flow rate: 0.7 mL/min

Eluent: A) 3.6 mmol/L of Na_2CO_3, 0% acetone

B) 3.6 mmol/L of Na_2CO_3, 10% acetone

C) 3.6 mmol/L of Na_2CO_3, 20% acetone

D) 3.6 mmol/L of Na_2CO_3, 30% acetone



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

	Metrosep A Supp 7 - 150/4.0	mg/L
6	Phosphate	10
7	Sulfate	10

The monovalent ions move in the opposite direction of phosphate (6) and sulfate (7) as acetone content increases. Phosphate (6) and sulfate (7) have longer retention times as acetone content increases. With 30% acetone, phosphate (6) co-elutes with sulfate (7).

The backpressure increases gradually as acetone content increases.

Variation of organic modifier: Methanol

Column: Metrosep A Supp 7 - 150/4.0

Sample preparation: -

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 10 µL

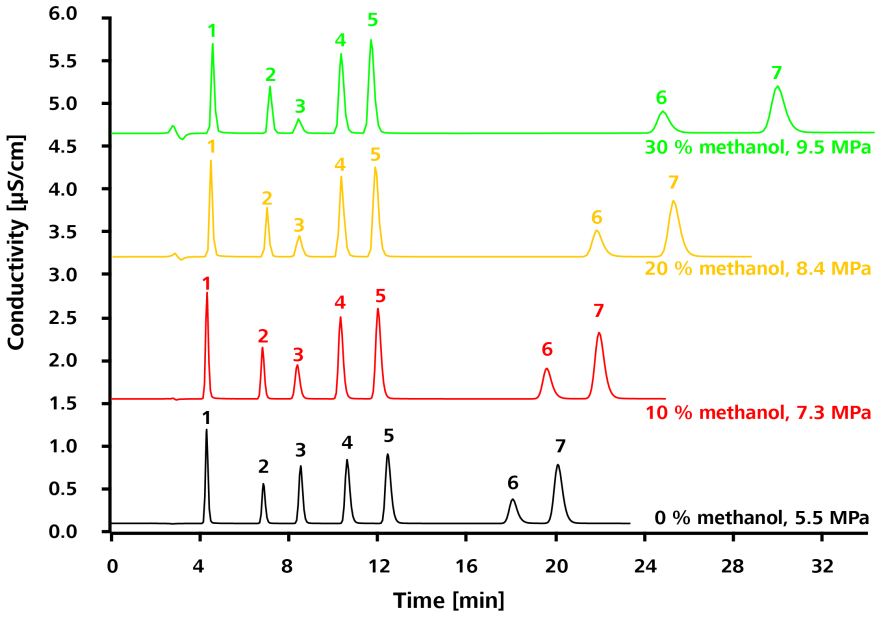
Flow rate: 0.7 mL/min

Eluent: A) 3.6 mmol/L of Na_2CO_3, 0% methanol

B) 3.6 mmol/L of Na_2CO_3, 10% methanol

C) 3.6 mmol/L of Na_2CO_3, 20% methanol

D) 3.6 mmol/L of Na_2CO_3, 30% methanol



	Metrosep A Supp 7 - 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 monovalent ions fluoride (1), chloride (2), nitrite (3), bromide (4), nitrate (5) are only minimally influenced by methanol to shorter retention times. Phosphate (6) and sulfate (7) move to later retention times with increasing methanol content. In contrast to acetone, the separation between phosphate (6) and sulfate (7) remains as methanol content increases.

The sensitivity of the ions decreases with increasing methanol content. This can be observed for nitrite (3) in particular.

The backpressure increases gradually as methanol content increases.

Variation of organic modifier: Acetonitrile

Column: Metrosep A Supp 7 - 150/4.0

Sample preparation: -

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 10 µL

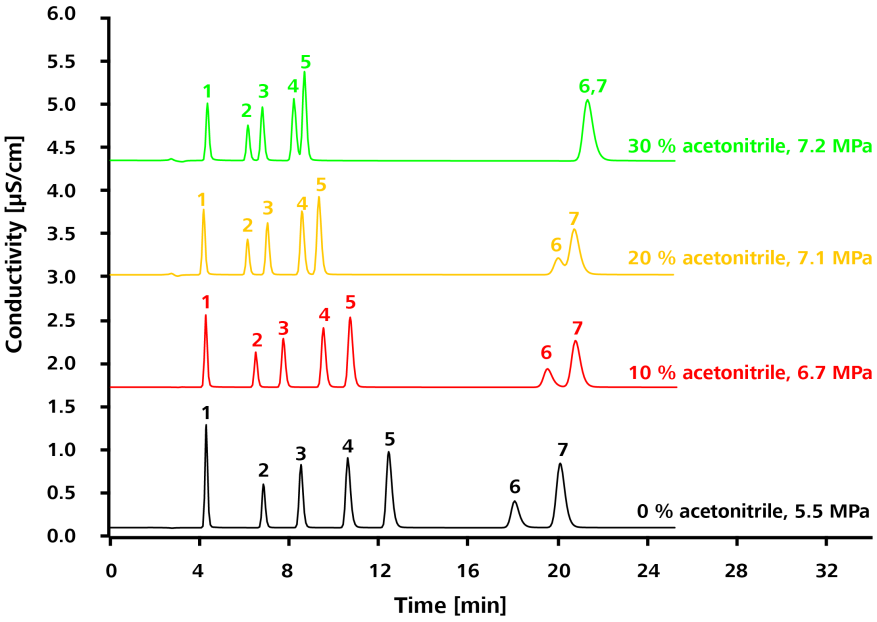
Flow rate: 0.7 mL/min

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

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

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

D) 3.2 mmol/L of Na_2CO_3, 1.0 mmol/L of $NaHCO_3$, 30% acetonitrile



	Metrosep A Supp 7 - 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

A similar behavior to acetone can be seen with acetonitrile as organic modifier. The monovalent ions move in the opposite direction of phosphate (6) and sulfate (7) as acetonitrile content increases. Phosphate (6)

and sulfate (7) have longer retention times as acetonitrile content increases. With 30% acetonitrile, phosphate (6) co-elutes with sulfate (7).

An increasing acetonitrile content as organic modifier has the least effect on backpressure increase.

Eluent variation: Metrosep A Supp 5 eluent

Column: Metrosep A Supp 7 - 250/4.0

Sample preparation: -

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

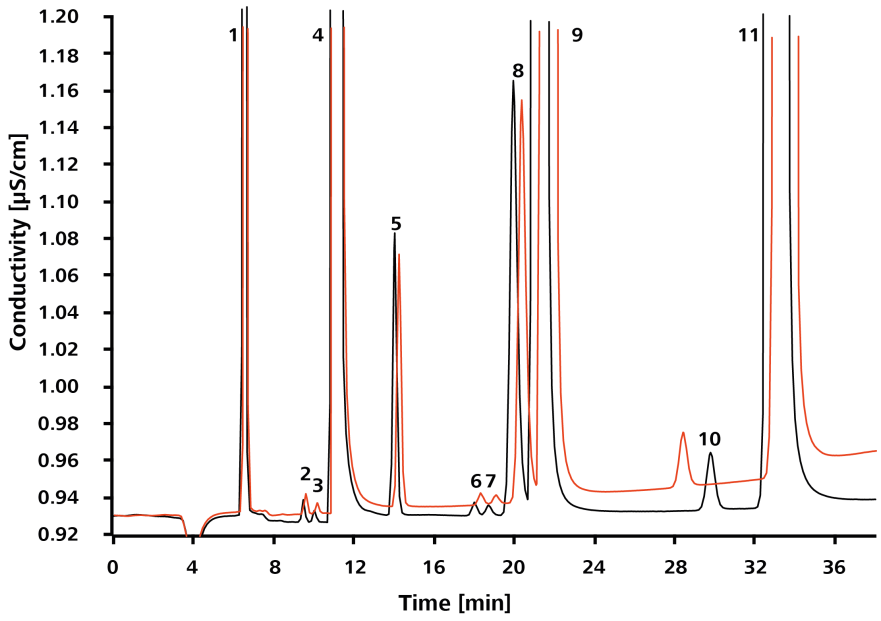
Temperature: 45 °C

Loop: 50 µL

Flow rate: 0.7 mL/min

Eluent: A) 3.2 mmol/L of Na_2CO_3 / 1.0 mmol/L of $NaHCO_3$ (red)

B) 3.6 mmol/L Na_2CO_3 (black)



	Metrosep A Supp 7 250/4.0	mg/L
1	Fluoride	0.1
2	Chlorite	0.01
3	Bromate	0.01

	Metrosep A Supp 7 250/4.0	mg/L
4	Chloride	10
5	Nitrite	0.1
6	Bromide	0.1
7	Chlorate	0.01
8	Dichloroacetic acid	1
9	Nitrate	10
10	Phosphate	0.1
11	Sulfate	10

The Metrosep A Supp 7 cannot only be used with the standard eluent (black) but also with the eluent of the Metrosep A Supp 5 (red), that is buffered. There cannot be observed any influence on the oxyhalides. Phosphate (10) moves slightly due to the pH change.

5.5 Separation of oxyhalides according to EPA 300.1

Column: Metrosep A Supp 7 - 250/4.0

Sample preparation: Inline Ultrafiltration

Detection: Conductivity

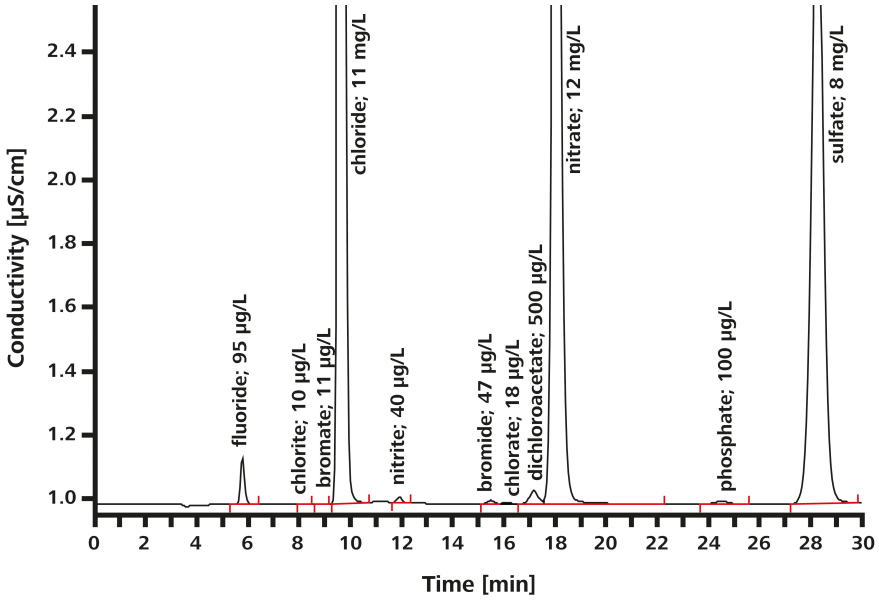
Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 20 µL

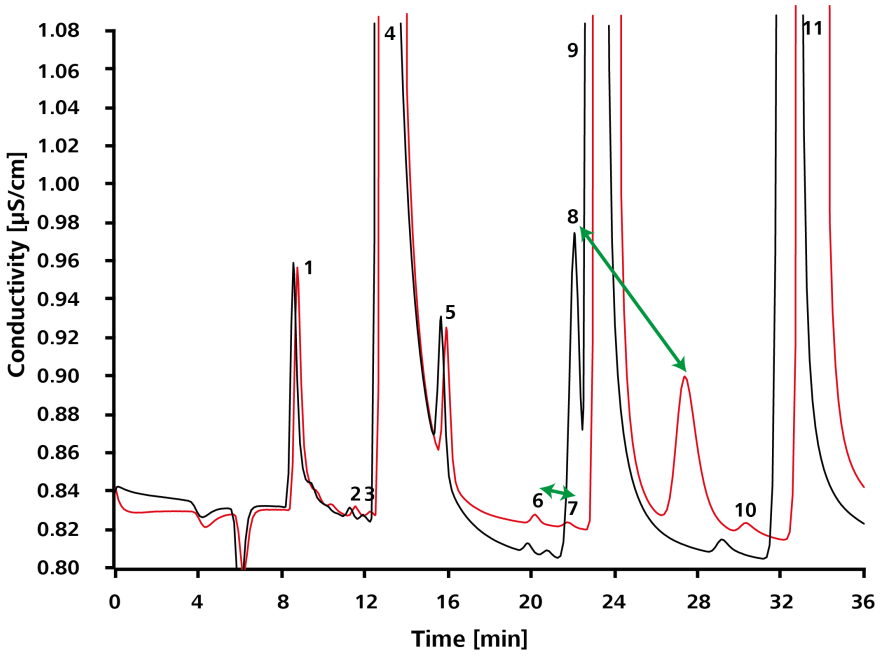
Flow rate: 0.7 mL/min

Eluent: 3.6 mmol/L Na_2CO_3



5.6 Improvement of bromide/chlorate/DCA separation with A Supp 16 Guard (EPA 300.1)

Column:	Metrosep A Supp 7 - 250/2.0
Sample preparation:	-
Detection:	Conductivity
Suppression:	Sequential suppression with MSM and MCS
Temperature:	45 °C
Loop:	10 µL
Flow rate:	0.2 mL/min
Eluent:	3.6 mmol/L Na_2CO_3



	Metrosep A Supp 7 - 250/2.0	mg/L
1	Fluoride	0.1
2	Chlorite	0.01
3	Bromate	0.01
4	Chloride	10
5	Nitrite	0.1
6	Bromide	0.1
7	Chlorate	0.01
8	Dichloroacetic acid	1
9	Nitrate	10
10	Phosphate	0.1
11	Sulfate	10

A Metrosep A Supp 5 Guard/2.0 does not influence the separation of the oxyhalides. The Metrosep A Supp 16 Guard/2.0, however, does improve bromide/chlorate resolution. At the same time, DCA moves behind nitrate. The same effect can also be observed with the 4-mm columns.

5.7 Multi-component analysis with Dose-in Gradient

Column: Metrosep A Supp 7 - 250/4.0

Sample preparation: -

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 20 µL

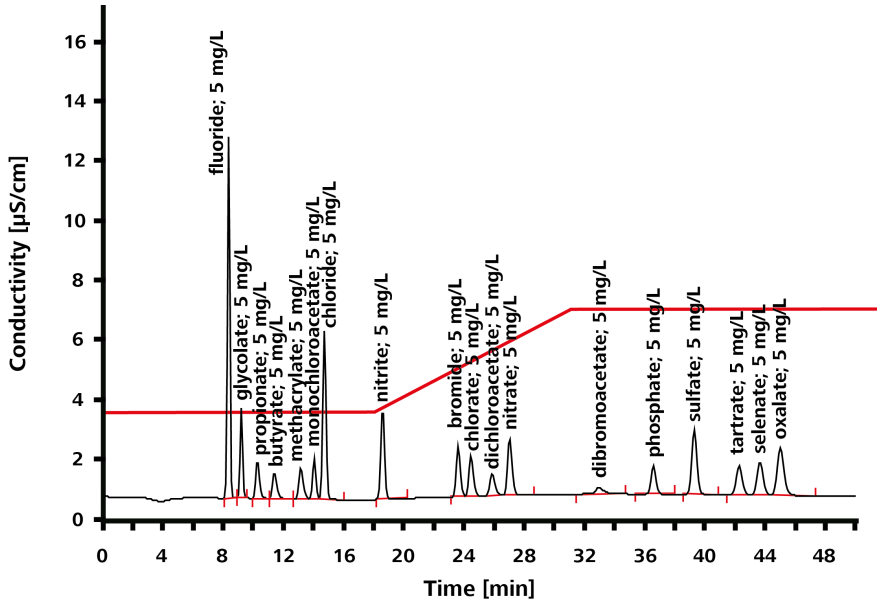
Flow rate: 0.7 mL/min

Eluent: Eluent A: 9.0 mmol/L Na_2CO_3

Eluent B: 1.8 mmol/L Na_2CO_3

Linear gradient with 10 mL Dosing Unit as follows:

- 0-15 min: A: 0%, B: 100%
- 15-30 min: A: 0-37.5%, B: 100-62.5%
- 30-50 min: A: 37.5%, B: 62.5%
- 50-60 min: A: 0%, B: 100%



5.8 Primary circuit of a pressurized water reactor

Column: Metrosep A Supp 7 - 250/4.0

Sample preparation: Inline Preconcentration with Inline Neutralization of 3.3 mg/L of lithium hydroxide and Matrix Elimination of 2 g/L of boric acid (MiPCT-NE-ME)

Detection: Conductivity

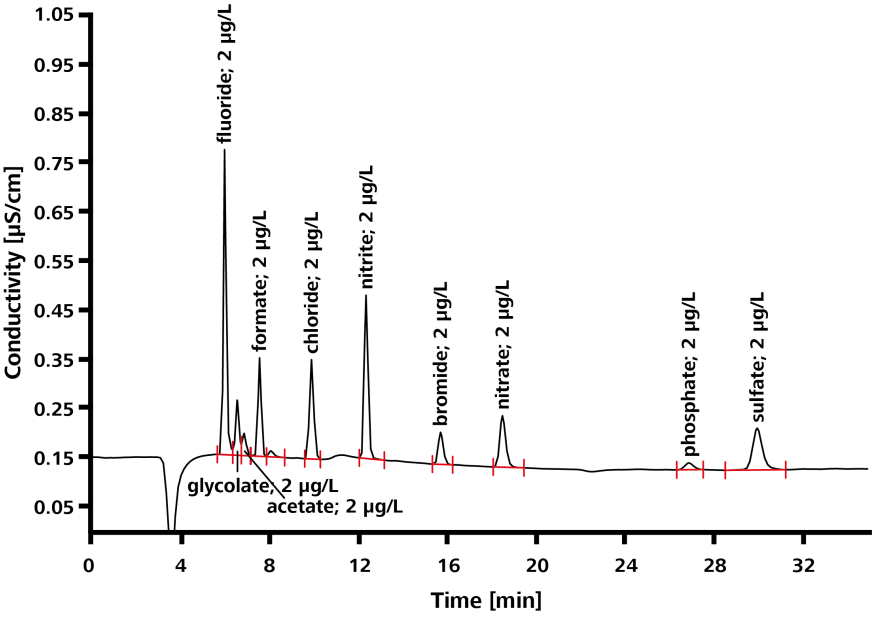
Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 2000 µL with preconcentration on Metrosep A PCC 2 HC/4.0

Flow rate: 0.7 mL/min

Eluent: 3.6 mmol/L Na_2CO_3



5.9 Ethanol E85 with 85% ethanol and 15% gasoline

Column: Metrosep A Supp 7 - 250/4.0

Sample preparation: Inline Matrix Elimination and Inline Preconcentration with 7.5% acetone as transfer solution

Detection: Conductivity

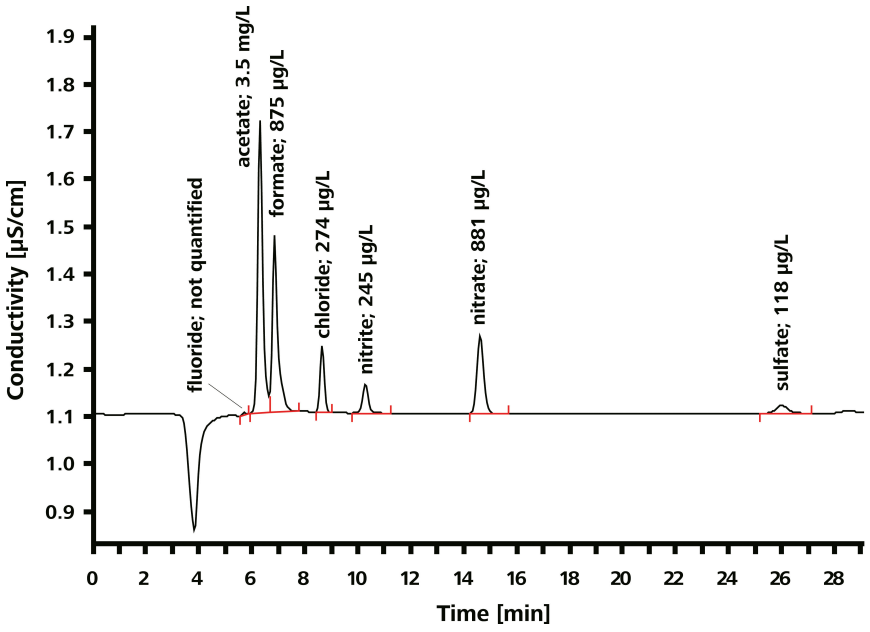
Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 10 µL with preconcentration on Metrosep A PCC 2 HC/4.0

Flow rate: 0.8 mL/min

Eluent: 3.6 mmol/L of Na_2CO_3, 7.5% acetone



5.10 Multi-component analysis with organic acids

Column: Metrosep A Supp 7 - 250/2.0

Sample preparation: -

Detection: Conductivity

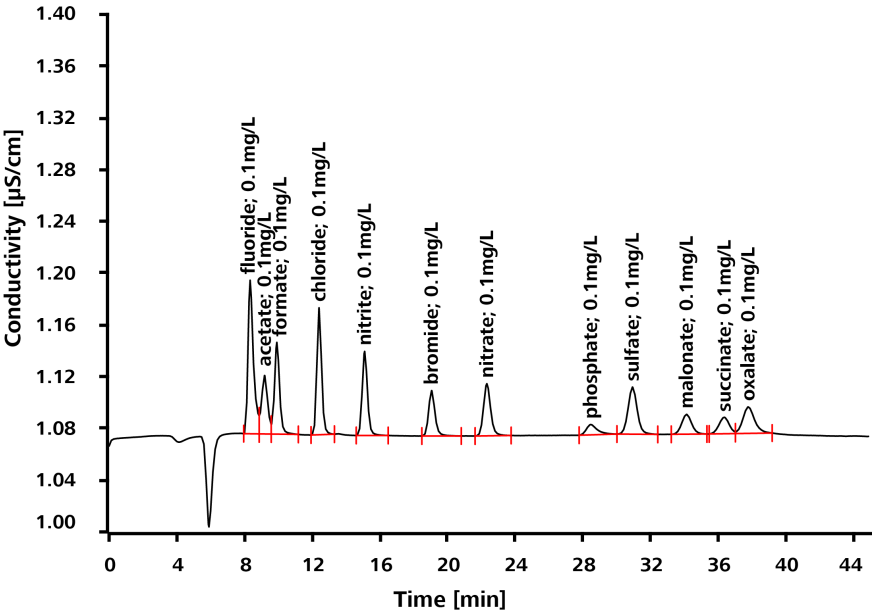
Suppression: Sequential suppression with MSM-LC and MCS

Temperature: 45 °C

Loop: 10 µL

Flow rate: 0.2 mL/min

Eluent: 3.6 mmol/L Na_2CO_3



5.11 Rapid analysis for late-eluting ions

Column: Metrosep A Supp 7 - 150/2.0

Sample preparation: -

Detection: Conductivity

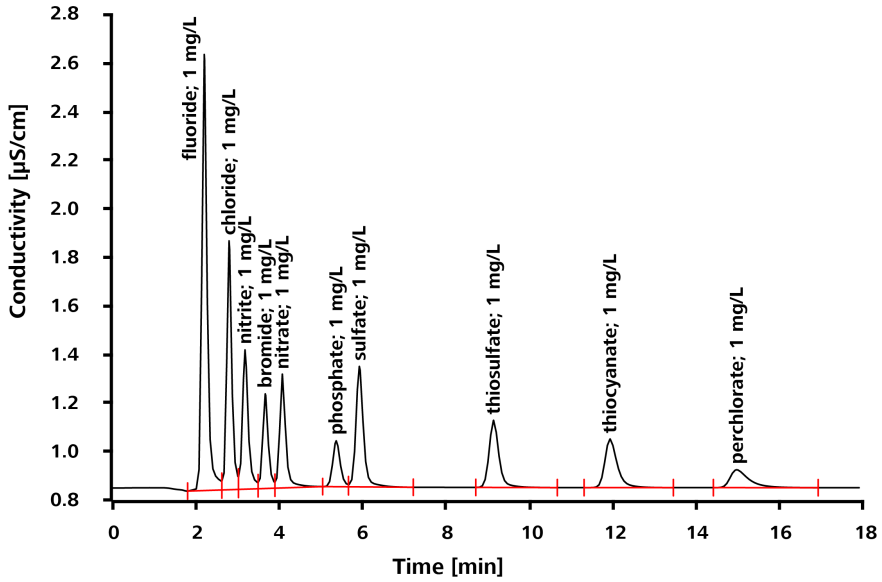
Suppression: Sequential suppression with MSM and MCS

Temperature: 55 °C

Loop: 10 µL

Flow rate: 0.6 mL/min (backpressure: approx. 18 MPa)

Eluent: 4.0 mmol/L Na_2CO_3



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

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.

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 5, page 38)*
- Contamination with high-valence hydrophobic ions or organic contamination *(see table 6, page 39)*

Table 5 Contamination with low-valence hydrophilic ions

		Duration in minutes	Flow rate 2 mm	Flow rate 4 mm
1.	Rinse with ultrapure water	25	0.1 mL/min	0.3 mL/min

		Duration in minutes	Flow rate 2 mm	Flow rate 4 mm
2.	Rinse with 10x concentrated eluent	100	0.1 mL/min	0.3 mL/min
3.	Rinse with ultrapure water	25	0.1 mL/min	0.3 mL/min
4.	Rinse with eluent	100	0.1 mL/min	0.3 mL/min

Table 6 Contamination with high-valence hydrophobic ions or organic contamination

		Duration in minutes	Flow rate 2 mm	Flow rate 4 mm
1.	Rinse with ultrapure water	25	0.1 mL/min	0.3 mL/min
2.	Rinse with 100% acetonitrile	20	0.1 mL/min	0.3 mL/min
3.	Rinse with ultrapure water	25	0.1 mL/min	0.3 mL/min
4.	Rinse with 10x concentrated eluent	100	0.1 mL/min	0.3 mL/min
5.	Rinse with ultrapure water	25	0.1 mL/min	0.3 mL/min
6.	Rinse with eluent	100	0.1 mL/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 was 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 smaller 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/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 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

We recommend the following literature for more detailed information:

- Application Note S-229 Oxohalides and monovalent organic acids in the presence of standard anions
- Application Note S-235 Determination of Anions and Oxohalides by US EPA method 300.1 A and B in a single analysis (Standard solution)
- Application Note S-236 Determination of Anions and Oxohalides by US EPA method 300.1 A and B in a single analysis (Sample)
- Application Note S-249 14 anions in an industrial process water
- Application Note S-252 Standard anions and organic acids in Bayer liquor applying Inline Neutralization
- Application Note S-255 Sixteen anions separated on the Metrosep A Supp 7 - 250 applying gradient elution
- Application Note S-256 Thirteen anions separated on the Metrosep A Supp 7 - 250
- Application Note S-267 Anions in an E85 blend (85% ethanol and 15% gasoline) applying Inline Matrix Elimination
- Application Note S-277 Hexafluorosilicate besides standard anions
- Application Note S-278 Anions in sodium tetraborate with Metrohm Inline Acidification, Inline Matrix Elimination and Inline Calibration
- Application Note U-48 Silicate and hexafluorosilicate
- Application Note S-305 Determination of chlorate, thiosulfate, thiocyanate, and perchlorate in the presence of standard anions using a Dose-in Gradient
- Application Note S-324 Traces of perchlorate in drinking water
- Application Note S-327 Shorter citrate retention times in beverage analysis using a step gradient
- Application Note S-339 15 organic acids on the Metrosep A Supp 7 - 250/4.0 using a high-pressure gradient
- Trace-level determination of anions in the primary circuit of a PWR-type nuclear power plant using ion chromatography after inline sample preparation, Poster Pittcon 2007, 8.000.6071
- Simultaneous Determination of Anions and Oxy-Halides (US EPA 300.1) by Sequential Suppressed Ion Chromatography in a Single Injection Analysis, p.23ff, The Application Notebook, 2007
- Direct Injection, Simple and Robust Analysis of Trace-Level Bromate and Bromide in Drinking Water by IC with Suppressed Conductivity Detection, p.537-543, Journal of separation Science, 48, 2010
- Physiochemical properties of carbonaceous aerosol from agricultural residue burning: Density, volatility, and hygroscopicity, p. 94-105, Atmospheric Environment, 140, 2016

- 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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