Column manual

Metrosep A Supp 19 (6.01034.4x0)

Manual

8.0107.8013EN / v1 / 2023-03-08

Metrohm

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Technical Communication
Metrohm AG
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Disclaimer

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1 General information

The Metrosep A Supp 19 is a high-performance separation column and is especially suitable for the determination of inorganic anions and low-molecular organic anions with chemical and sequential suppression. Due to its high capacity, the Metrosep A Supp 19 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
6.01034.410	Metrosep A Supp 19 - 100/4.0
6.01034.420	Metrosep A Supp 19 - 150/4.0
6.01034.430	Metrosep A Supp 19 - 250/4.0

Table 2 Guard column

Order number	Designation
6.01034.500	Metrosep A Supp 19 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
6.01034.410	100 x 4.0 mm
6.01034.420	150 x 4.0 mm
6.01034.430	250 x 4.0 mm

pH range eluent 0–14

ph range sample 0–14

Temperature range 10–70 °C

Recommended standard temperature

25 °C

Maximum pressure

Order number	Maximum pressure
6.01034.410	20 MPa (200 bar)
6.01034.420	25 MPa (250 bar)
6.01034.430	25 MPa (250 bar)

Flow rate

Order number	Recommended flow rate	Maximum flow rate
6.01034.410	0.7 mL/min	1.3 mL/min
6.01034.420	0.7 mL/min	1.2 mL/min
6.01034.430	0.7 mL/min	1.0 mL/min

Standard eluent

8.0 mmol/L sodium carbonate (Na_2CO_3) and 0.25 mmol/L sodiumhydrogencarbonate ($NaHCO_3$)

Permitted organic additives

0–100% acetonitrile, acetone and methanol

Capacity

Order number	Capacity
6.01034.410	94 µmol (Cl^-)
6.01034.420	140 µmol (Cl^-)
6.01034.430	234 µmol (Cl^-)

Preparation

Use a flow gradient to set the column to the standard flow within 5 minutes. Then wait until the baseline is given.

Storage

Store the column in the standard eluent and at 4 to 30 °C.

Typical pressure

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

Order number	Typical pressure
6.01034.410	11 MPa
6.01034.420	14 MPa
6.01034.430	18 MPa

Column housing

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

Application

Determination of inorganic anions and low-molecular organic anions 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 Production of standard eluent

Proceed as follows to produce 2 L of standard eluent with 0.25 mmol/L of sodium hydrogen carbonate and 8.0 mmol/L of sodium carbonate:

Producing 2 L of standard eluent

Accessories

- Eluent bottle (6.1608.120)
- Cover (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 Degas the ultrapure water.

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 42.0 mg of sodium hydrogen carbonate.
- Weigh 1,695.8 mg of sodium carbonate.
- Add the weighed amounts of sodium hydrogen carbonate and sodium carbonate to the ultrapure water.

5 Rinse the column with eluent for 1 h.

This eluent (0.25 mmol/L of sodium hydrogen carbonate and 8.0 mmol/L of sodium carbonate) and chemical suppression can be used to achieve background conductivity of less than 23 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.

NOTICE

Metrohm recommends always working with guard columns. Guard columns 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 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).

NOTICE

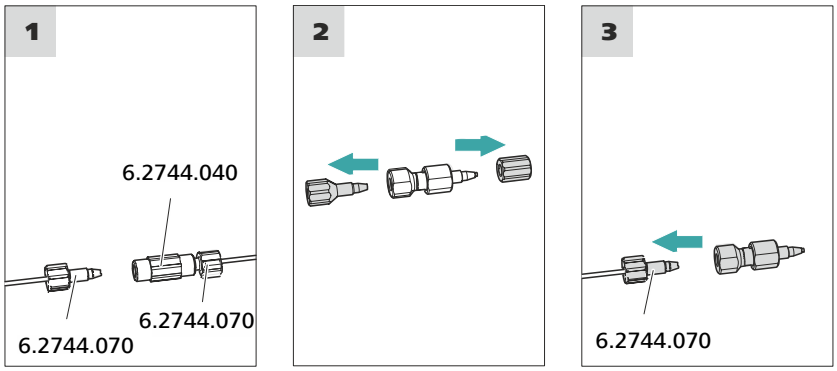
Do not connect the guard column until after the initial start-up of the instrument . Until then, replace the guard column and the separation column with couplings (6.2744.040).

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.

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 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.

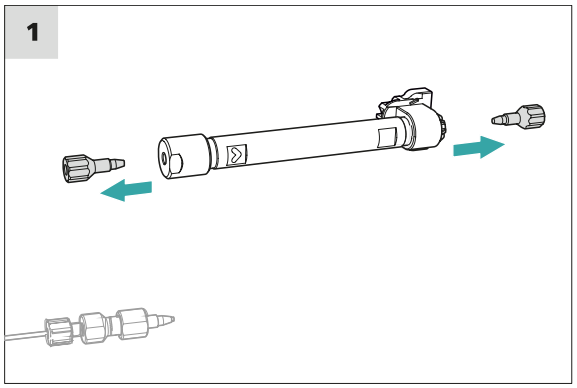
A test chromatogram accompanies every column. The column leaflet can be found online at *http://www.metrohm.com* with the corresponding article. Detailed information on special IC applications can be found in the corresponding **Application Bulletins** or **Application Notes**. You can find these online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

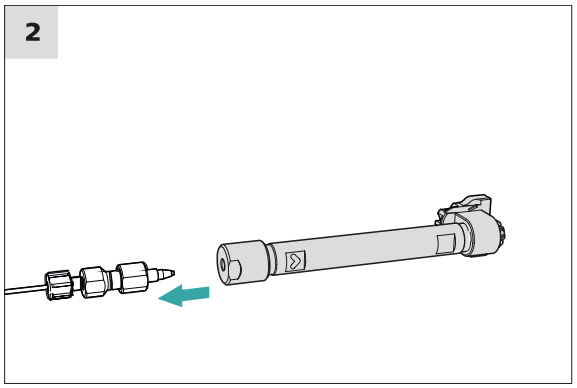
CAUTION

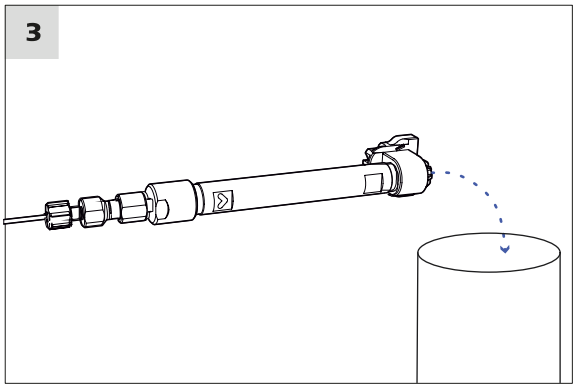
New IC Columns are filled with solution and sealed with stoppers on both sides. Before inserting the column, ensure that this solution can be mixed with the eluent being used (follow the information provided by the manufacturer).

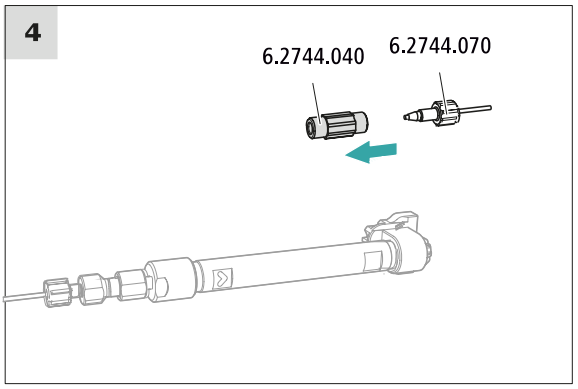
NOTICE

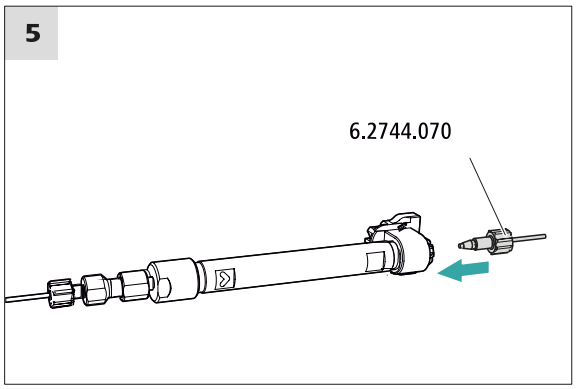
Connect the separation column only after the initial start-up of the instrument. Until that point, insert a coupling (6.2744.040) instead of the guard column and separation column.

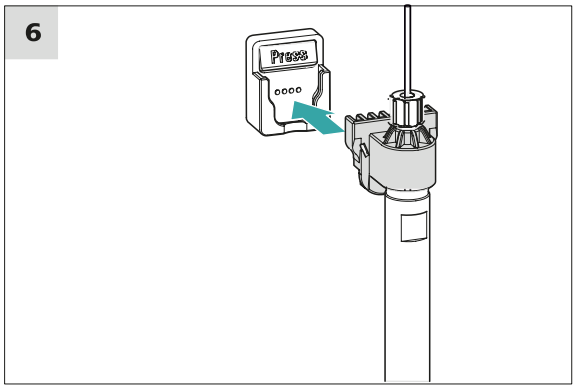












Connecting the separation column

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.
  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

Columns:
- Metrosep A Supp 19 - 100/4.0
- Metrosep A Supp 19 - 150/4.0
- Metrosep A Supp 19 - 250/4.0

Sample preparation: –

Detection: Conductivity

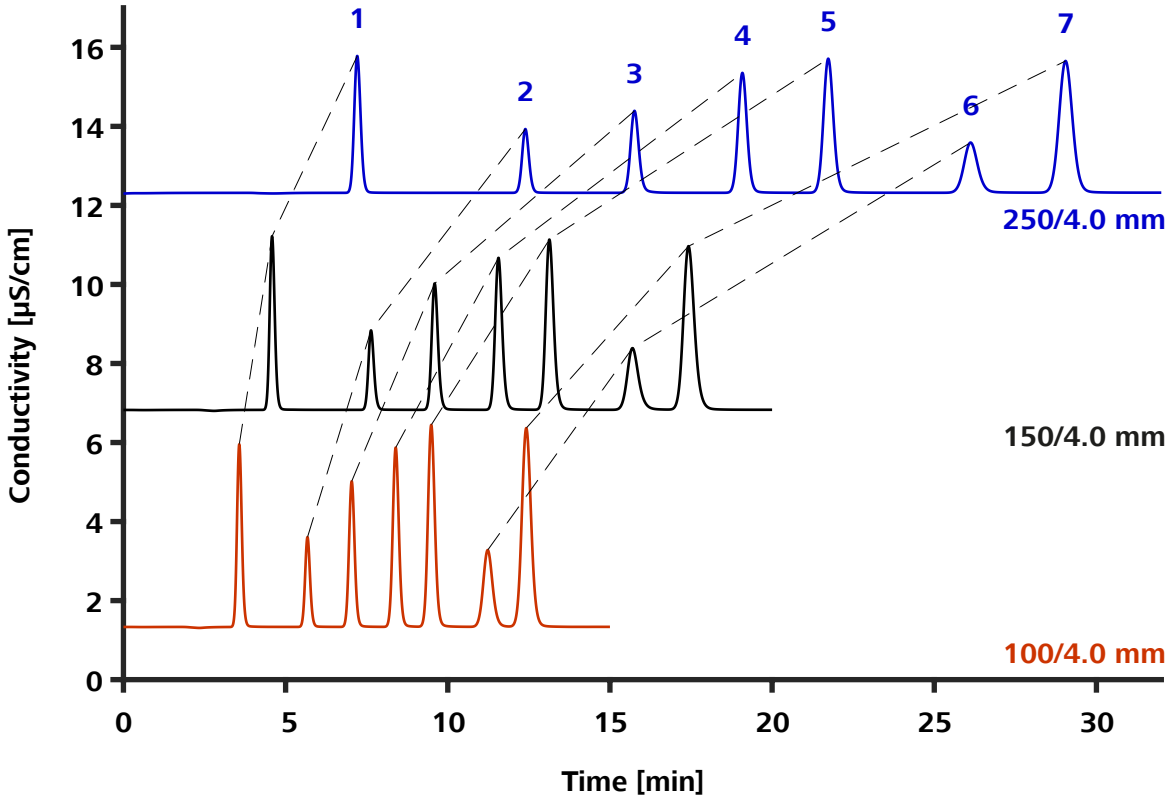
Suppression: Sequential suppression with MSM and MCS

Temperature: 25 °C

Loop: 20 µL

Flow rate: 0.7 mL/min

Eluent: 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3



	Metrosep A Supp 19 - XXX/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

5.2 Effects of temperature

Column: Metrosep A Supp 19 - 150/4.0

Sample preparation: –

Detection: Conductivity

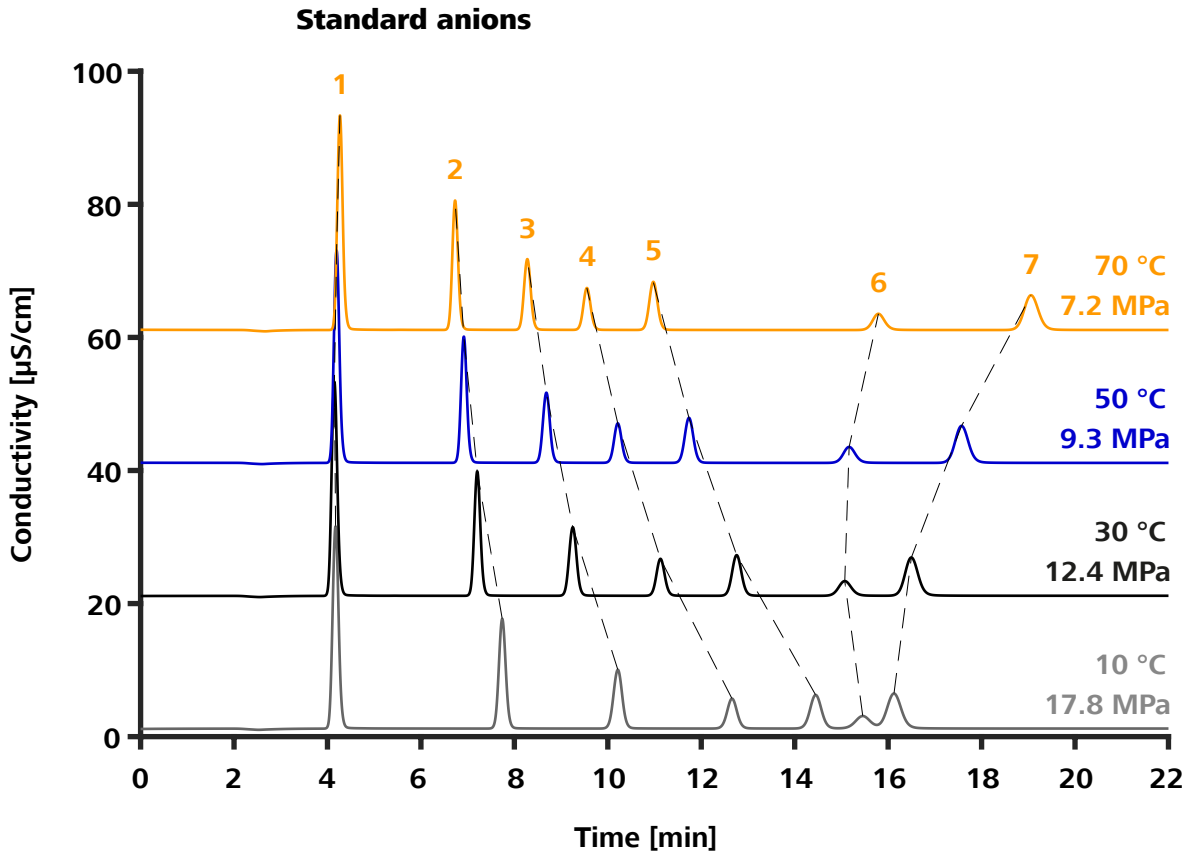
Suppression: Sequential suppression with MSM and MCS

Temperature: 10–70 °C

Loop: 20 µL

Flow rate: 0.7 mL/min

Eluent: 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3

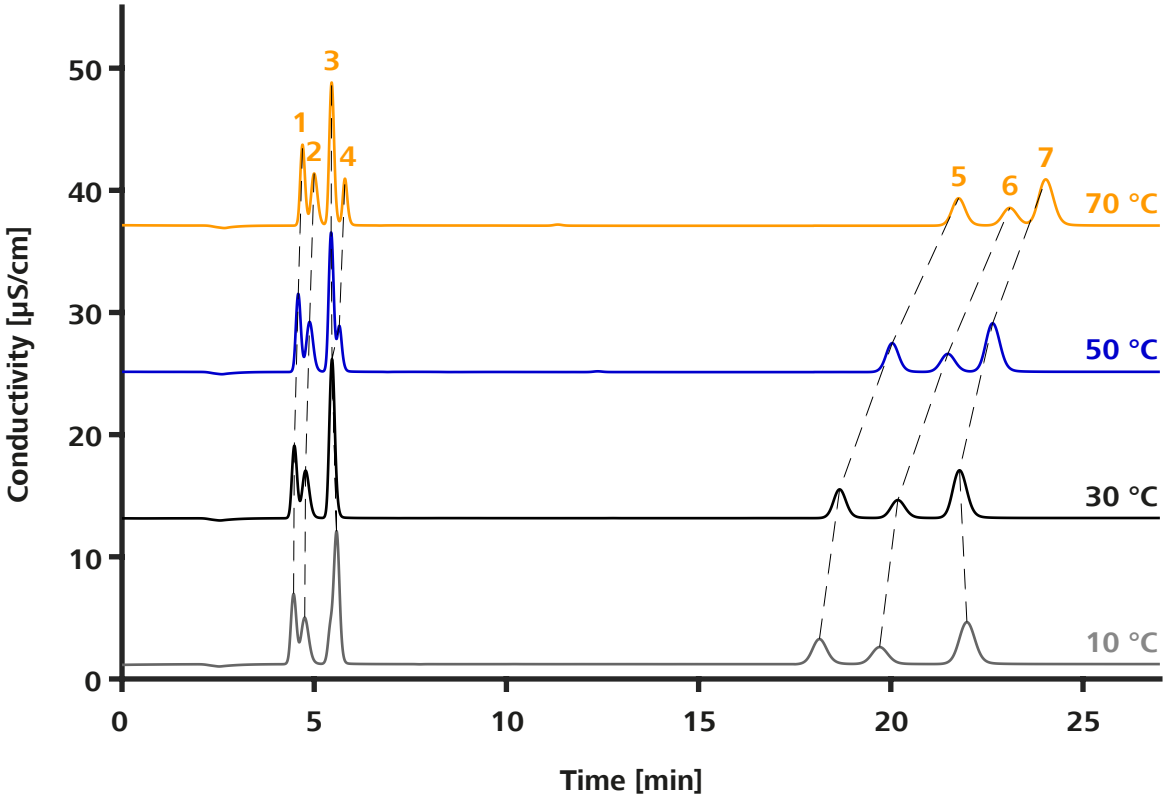


	Metrosep A Supp 19 - 150/4.0	mg/L
1	Fluoride	10
2	Chloride	10
3	Nitrite	10
4	Bromide	10
5	Nitrate	10
6	phosphate	10
7	Sulfate	10

The Metrosep A Supp 19 can be used at temperatures from 10 to 70 °C. The retention times of the monovalent anions decrease with increases in temperature. Especially the polarizable ions nitrite, bromide and nitrate are strongly accelerated at higher temperature. At the same time, the peaks become somewhat sharper at higher temperatures. The retention time of phosphate increases slightly when the temperature increases. The retention time of sulfate increases significantly with increasing temperatures. At 10 °C, phosphate and sulfate are not completely separated.

Increasing the temperature also causes the column backpressure to decrease considerably. At 70 °C, the backpressure is only approx. 7.2 MPa, whereas the column backpressure at 10 °C is more than twice as high (about 17.8 MPa). Due to the high backpressure at low temperatures, standard flow measurements at 10 °C are not possible on the long Metrosep A Supp 19 - 250/4.0 column.

Organic acids

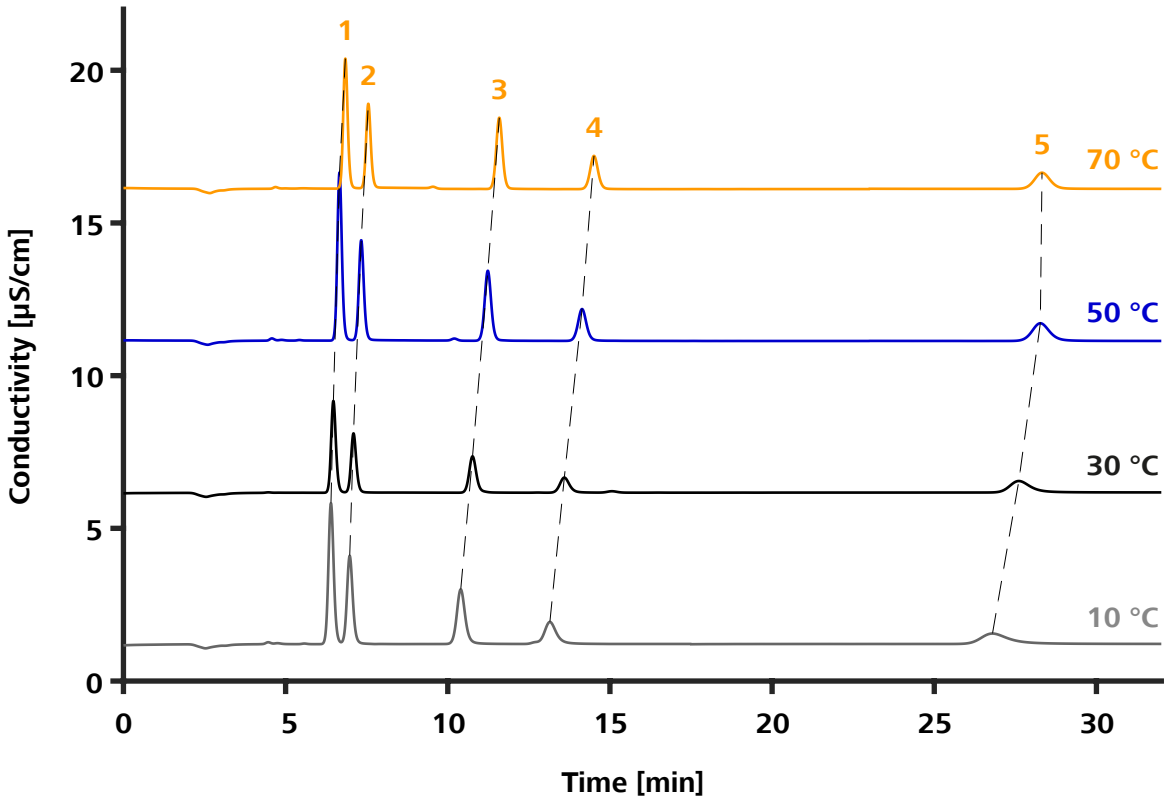


	Metrosep A Supp 19 - 150/4.0	mg/L
1	Glycolate	10
2	Acetate	10
3	Formate	10
4	Pyruvate	10
5	Tartrate	10
6	Succinate	10
7	Oxalate	10

The monovalent organic acids such as glycolate, acetate, formate and pyruvate elute between fluoride and chloride. Glycolate, acetate and formate are largely separated from one another at 30 °C. Their retention times are affected only slightly by temperature. The retention time of pyruvate, on

the other hand, increases with increasing temperature. At 10 °C, formate and pyruvate coelute. At 70 °C, pyruvate is separated from formate and elutes after formate. The divalent organic acids, such as tartrate, succinate and oxalate, behave similarly to the divalent inorganic anions, such as sulfate: With increasing temperature, the retention times also increase.

Haloacetic acids



	Metrosep A Supp 19 - 150/4.0	mg/L
1	Monochloroacetate	10
2	Monobromoacetate	10
3	Dichloracetate	10
4	Dibromacetate	10
5	Trichloroacetate	10

The haloacetic acids are another class of components that are often analyzed in ion chromatography. Although haloacetic acids are monovalent ions, their reaction to temperature influences differs from that of inorganic anions. When the temperature increases, the retention times of all ions increase. It should be noted that haloacetic acids degrade at higher temperatures, making them difficult to analyze.

5.3 Variation of the eluent flow rate

Column: Metrosep A Supp 19 - 150/4.0

Sample preparation: –

Detection: Conductivity

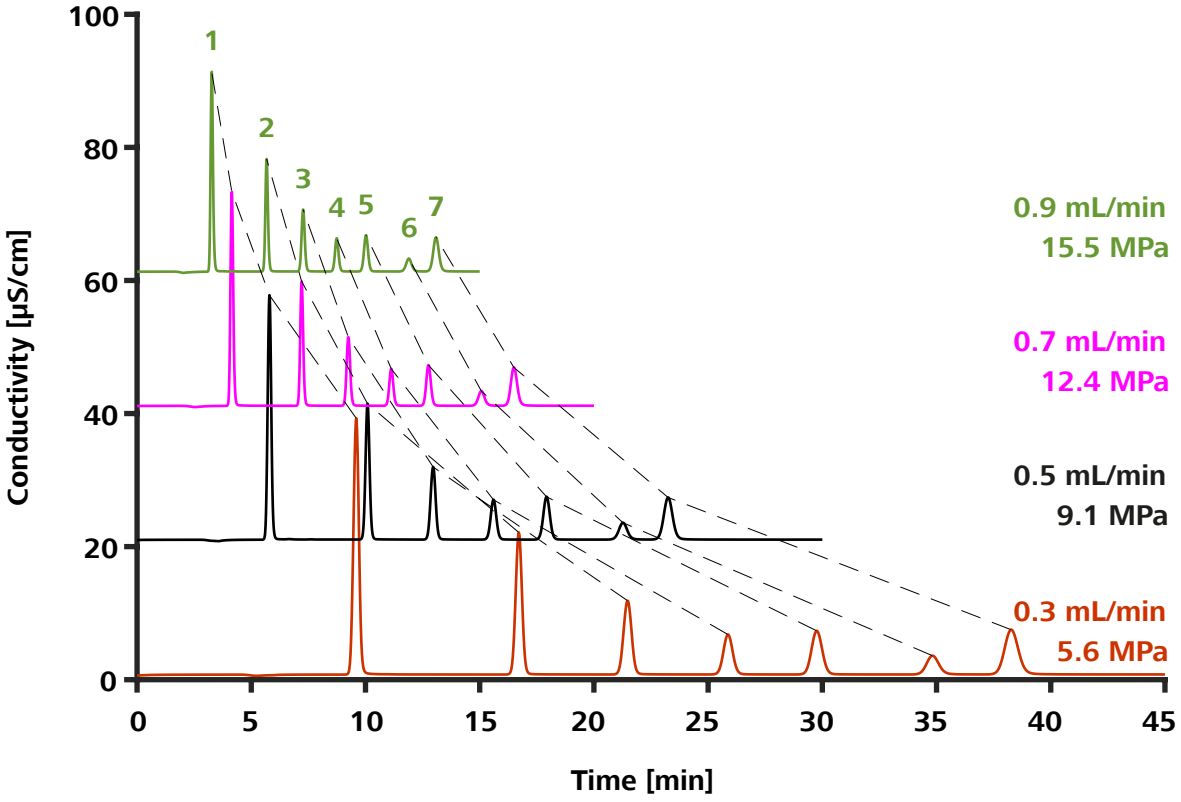
Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.3–0.9 mL/min

Eluent: 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3



	Metrosep A Supp 19 - 150/4.0	mg/L
1	Fluoride	10
2	Chloride	10
3	Nitrite	10
4	Bromide	10
5	Nitrate	10
6	phosphate	10
7	Sulfate	10

The Metrosep A Supp 19 - 150/4.0 can be operated with a flow of up to 1.2 mL/min. As the flow rate increases, all ions are accelerated uniformly, which means that sulfate elutes in less than 14 minutes at 0.9 mL/min. The pressure increases almost proportionally to the flow. Due to the higher flow rate, the dwell time of the analytes in the detector is reduced, resulting in smaller peak areas. The long Metrosep A Supp 19 - 250/4.0 can be operated with a maximum flow of 1.0 mL/min. The temperature must be increased in the event of high flow rates on the long column. If not, there will be an overpressure on the column.

5.4 Variation of the eluent

5.4.1 Constant Na_2CO_3/$NaHCO_3$ ratio

Column: Metrosep A Supp 19 - 150/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.7 mL/min

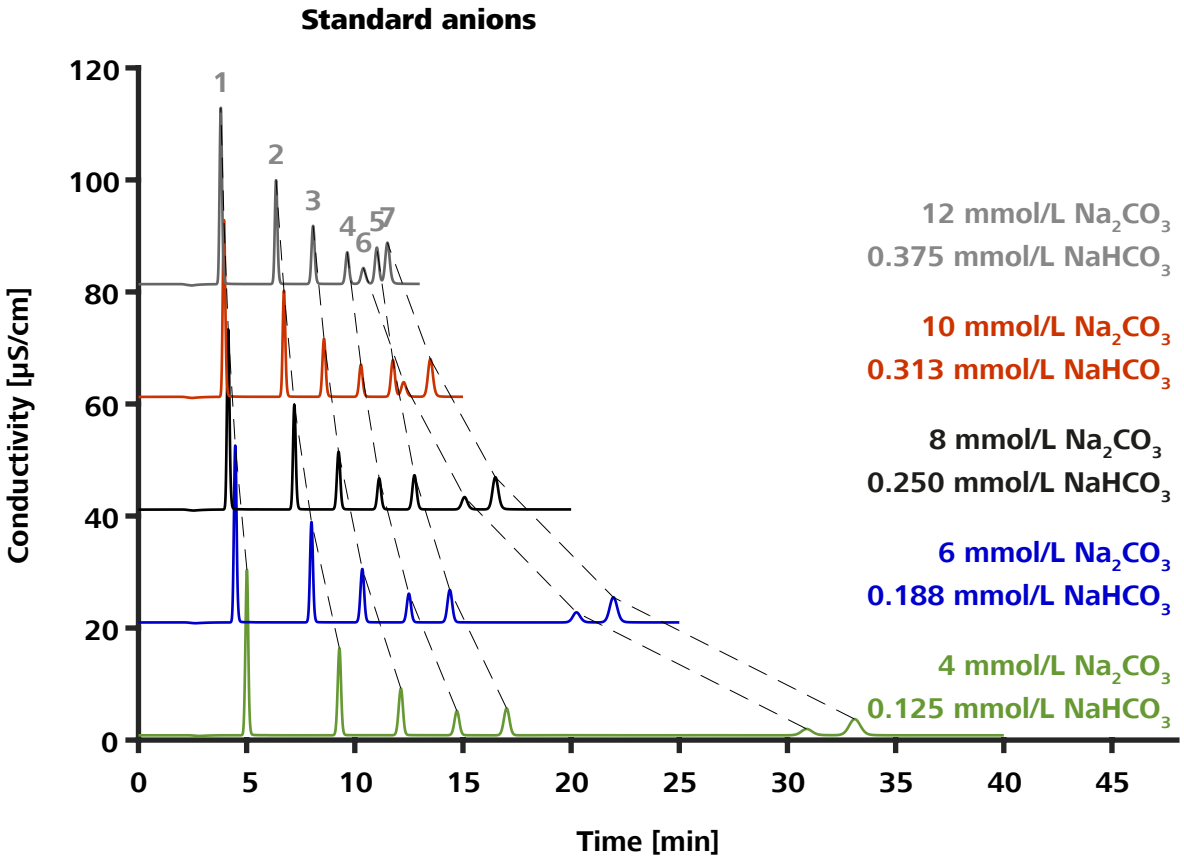
Eluent: A) 0.125 mmol/L $NaHCO_3$, 4.0 mmol/L Na_2CO_3

B) 0.188 mmol/L $NaHCO_3$, 6.0 mmol/L Na_2CO_3

C) 0.250 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3

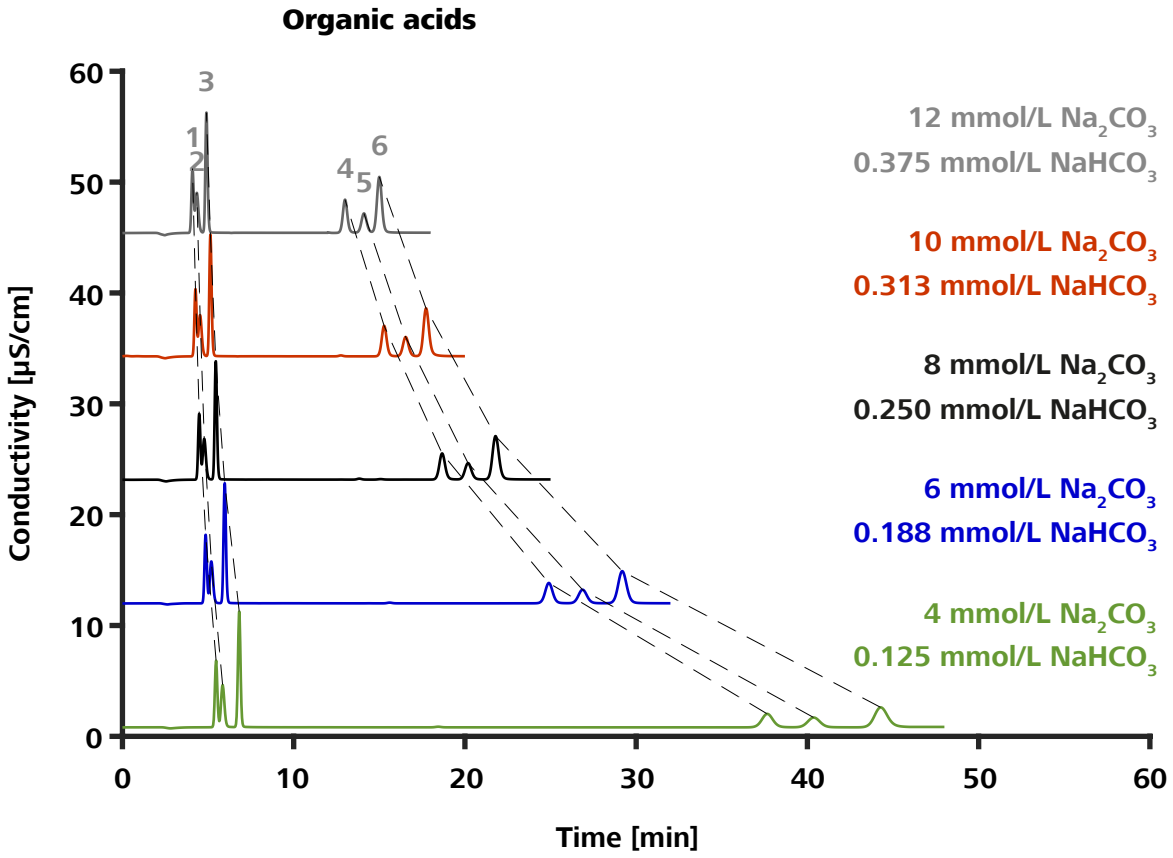
D) 0.313 mmol/L $NaHCO_3$, 10.0 mmol/L Na_2CO_3

E) 0.375 mmol/L $NaHCO_3$, 12.0 mmol/L Na_2CO_3



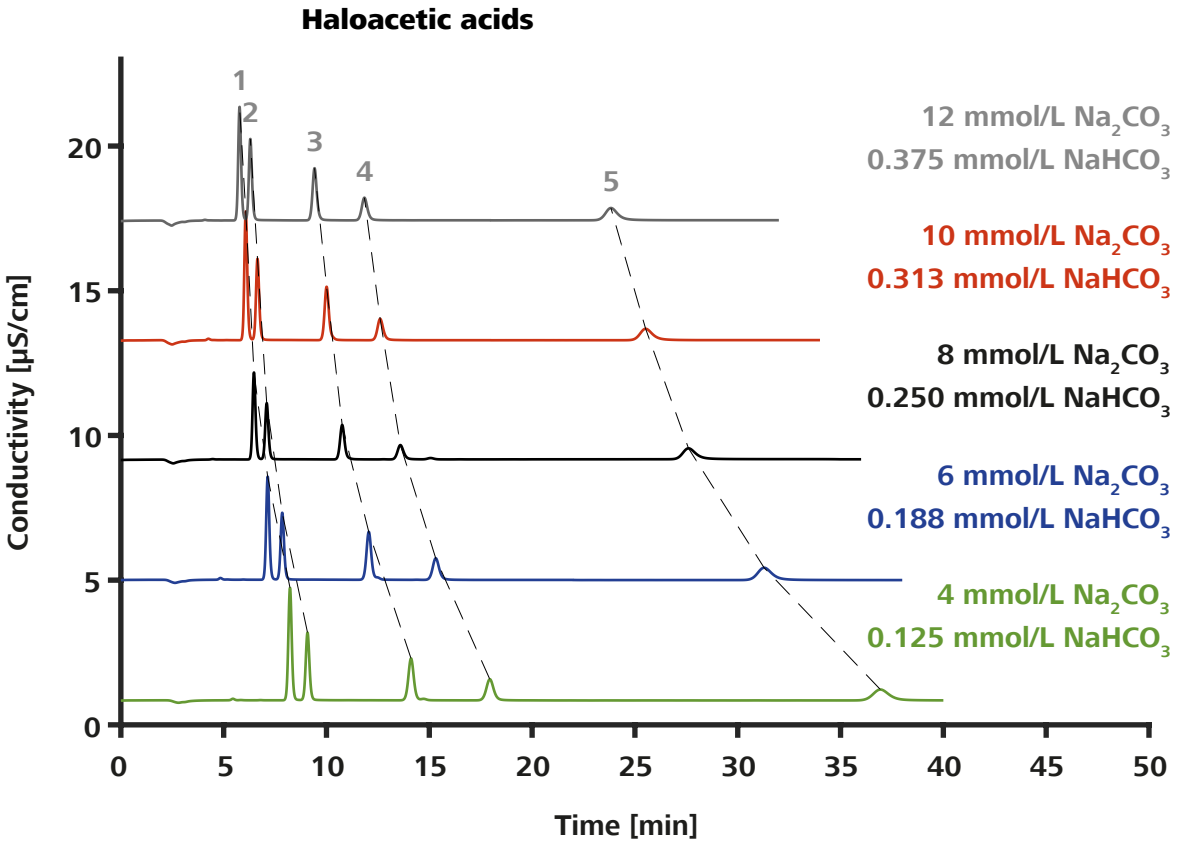
	Metrosep A Supp 19 - 150/4.0	mg/L
1	Fluoride	10
2	Chloride	10
3	Nitrite	10
4	Bromide	10
5	Nitrate	10
6	phosphate	10
7	Sulfate	10

With increasing eluent concentration, all anions are significantly accelerated. In this process, the multivalent anions phosphate and sulfate are accelerated faster than the monovalent anions. With a stronger eluent, the peaks are sharper and correspondingly higher. For a strong eluent (0.313 mmol/L $NaHCO_3$, 10 mmol/L Na_2CO_3), nitrate coelutes with phosphate. For an even stronger eluent (0.375 mmol/L $NaHCO_3$, 12 mmol/L Na_2CO_3), the elution order is phosphate, nitrate, sulfate. The separation between nitrate and sulfate is not optimal.



	Metrosep A Supp 19 - 150/4.0	mg/L
1	Glycolate	10
2	Acetate	10
3	Formate	10
4	Tartrate	10
5	Succinate	10
6	Oxalate	10

The organic acids are greatly accelerated with increasing eluent concentration. The monovalent organic acids such as glycolate, acetate and formate are accelerated to an extent similar to that of fluoride or chloride. The divalent organic acids such as tartrate, succinate and oxalate, on the other hand, behave like sulfate and are accelerated to a much greater extent. As the eluent concentration increases, the peaks become sharper and higher. This is particularly visible with divalent organic acids.



	Metrosep A Supp 19 - 150/4.0	mg/L
1	Monochloroacetate	10
2	Monobromoacetate	10
3	Dichloracetate	10
4	Dibromacetate	10
5	Trichloroacetate	10

The haloacetic acids react strongly on the Metrosep A Supp 19 to the eluent strength. With increasing eluent strength, all haloacetic acids are greatly accelerated.

5.4.2 $NaHCO_3$ variation at constant Na_2CO_3

Column: Metrosep A Supp 19 - 150/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.7 mL/min

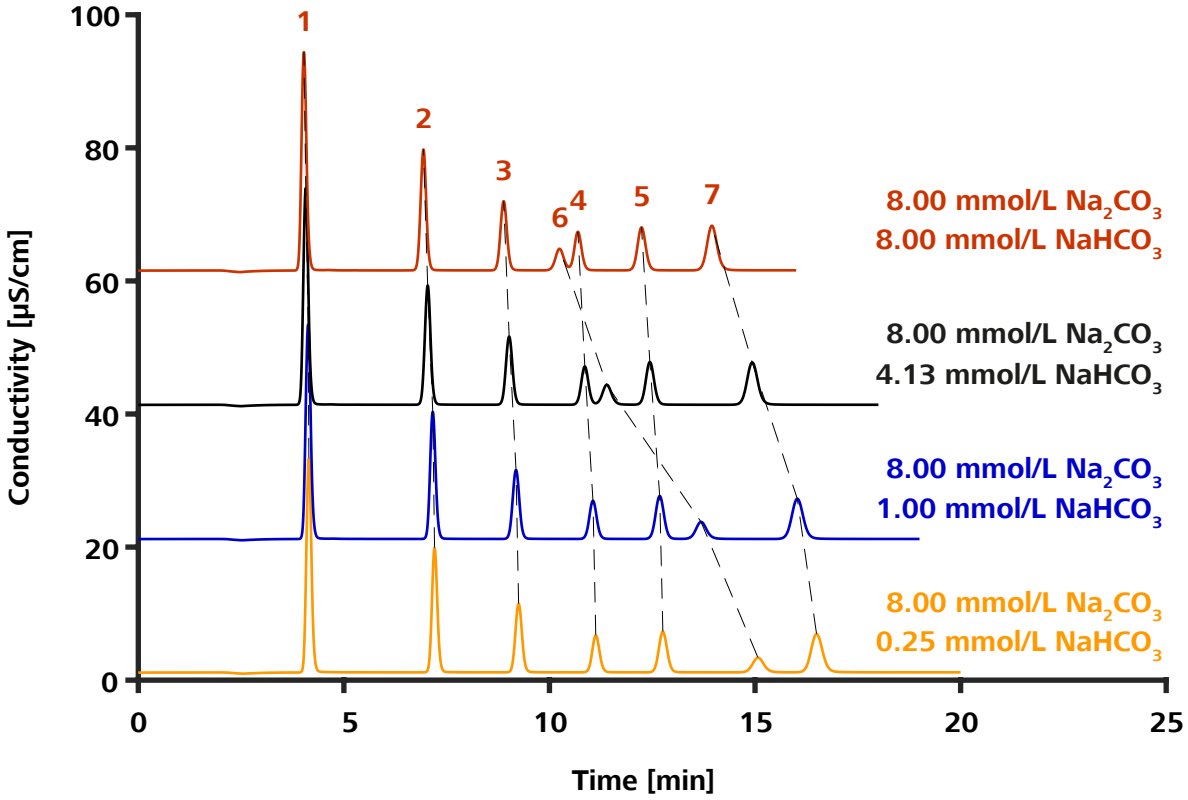
Eluent: A) 0.250 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3

B) 1.0 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3

C) 4.125 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3

D) 8.0 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3

Standard anions

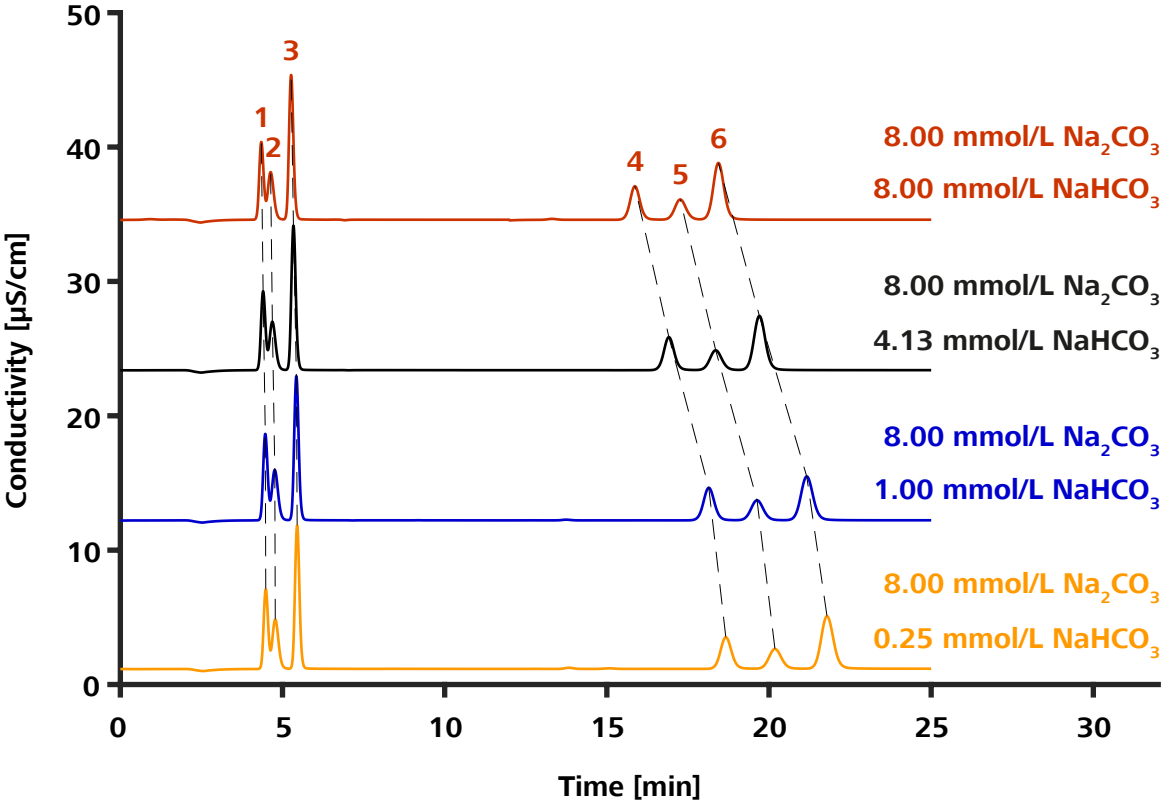


	Metrosep A Supp 19 - 150/4.0	mg/L
1	Fluoride	10
2	Chloride	10
3	Nitrite	10
4	Bromide	10
5	Nitrate	10
6	phosphate	10

	Metrosep A Supp 19 - 150/4.0	mg/L
7	Sulfate	10

Sodium hydrogen carbonate influences the eluent strength much less than sodium carbonate. The retention times of the anions are therefore only slightly shortened by increasing the content of sodium hydrogen carbonate in the eluent. Only the retention time of phosphate is shortened to a significantly greater extent, because the pH value of the eluent changes significantly, which means that the effective charge of the phosphate ion is also reduced. The retention time of sulfate also shortens as the hydrogen carbonate content increases. No significant change in peak heights was observed in the tested range of sodium hydrogen carbonate. In an eluent composition of 4.125–8.000 mmol/L $NaHCO_3$ and 8.0 mmol/L Na_2CO_3, bromide and phosphate are not optimally separated.

Organic acids

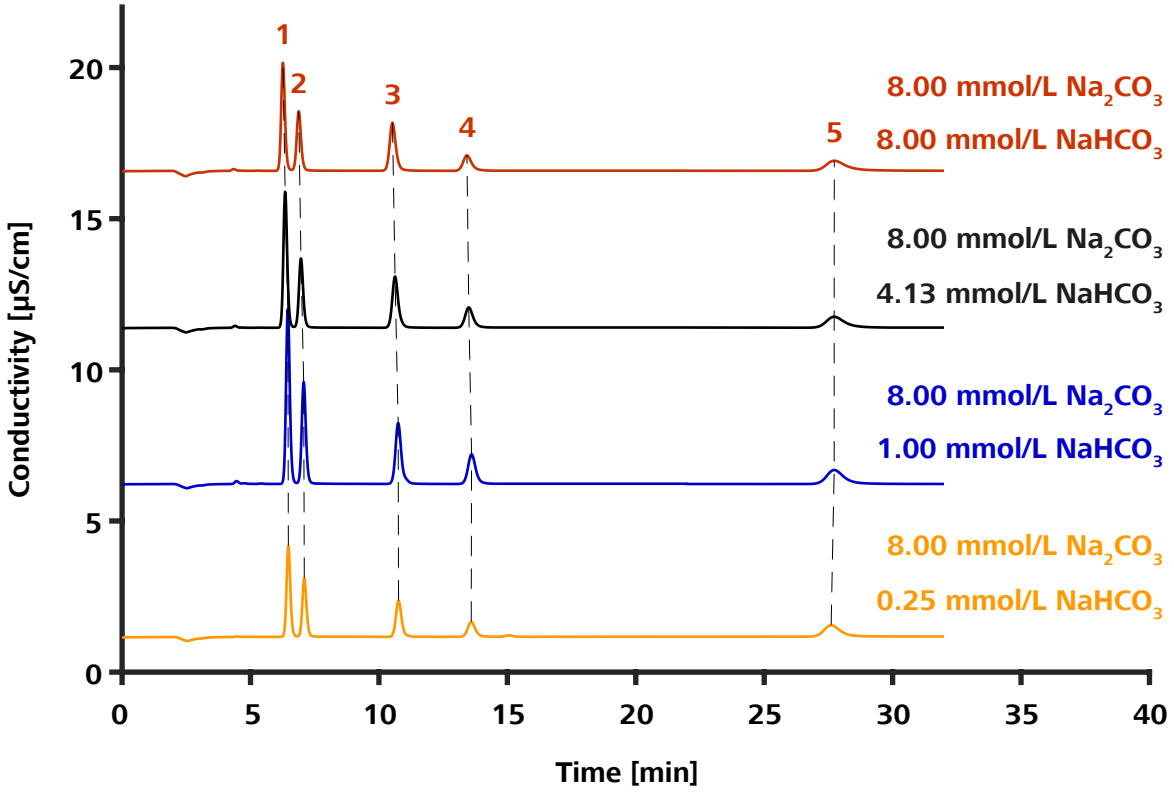


	Metrosep A Supp 19 - 150/4.0	mg/L
1	Glycolate	10
2	Acetate	10
3	Formate	10

	Metrosep A Supp 19 - 150/4.0	mg/L
4	Tartrate	10
5	Succinate	10
6	Oxalate	10

The weak elution strength of hydrogen carbonate is also evident with organic acids. The monovalent organic acids are hardly affected by the hydrogen carbonate content. The divalent organic acids are moved to slightly earlier retention times when the hydrogen carbonate content is high. Nevertheless, the effect is relatively weak.

Haloacetic acids



	Metrosep A Supp 19 - 150/4.0	mg/L
1	Monochloroacetate	10
2	Monobromoacetate	10
3	Dichloracetate	10
4	Dibromacetate	10
5	Trichloroacetate	10

The hydrogen carbonate content in the eluent does not affect the haloacetic acids. The retention times and shapes of the peaks of these analytes remain constant and are not affected by hydrogen carbonate.

5.4.3 Na_2CO_3 variation at constant $NaHCO_3$

Column: Metrosep A Supp 19 - 150/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.7 mL/min

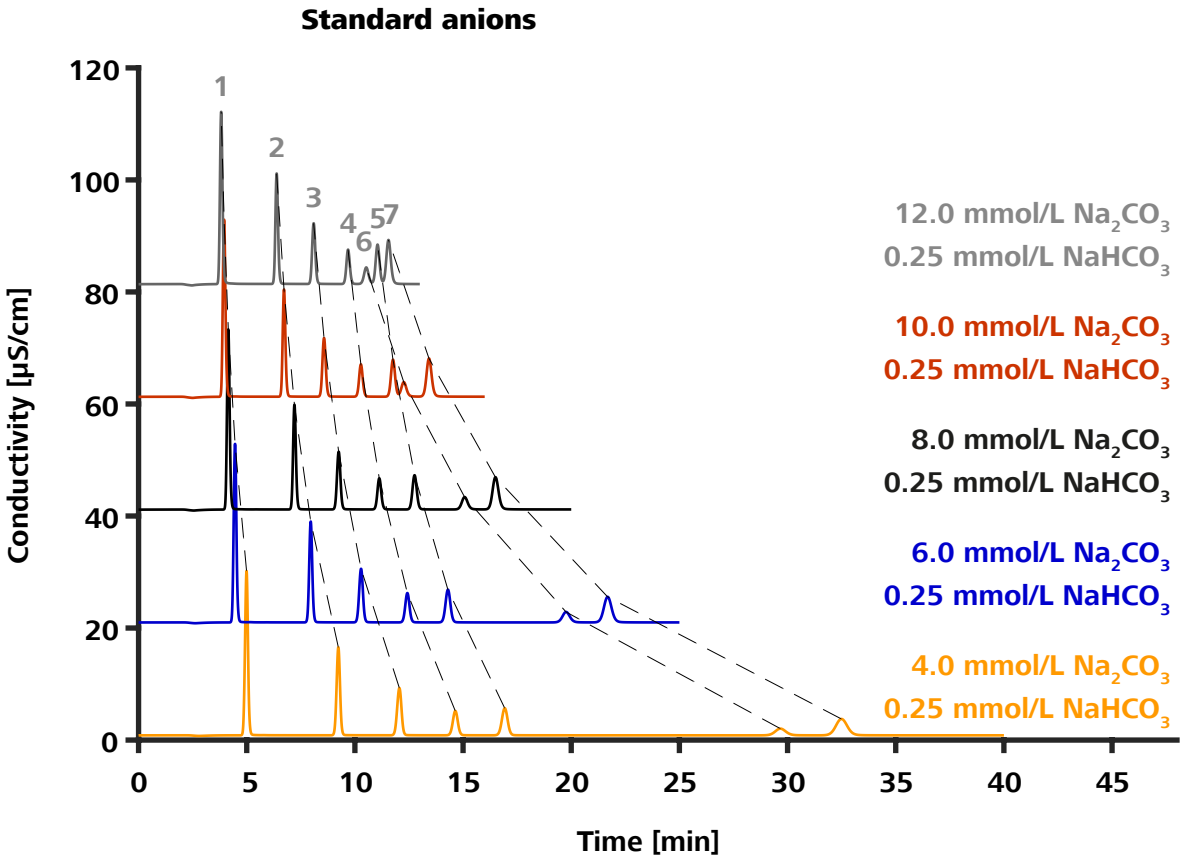
Eluent: A) 0.25 mmol/L $NaHCO_3$, 4.0 mmol/L Na_2CO_3

B) 0.25 mmol/L $NaHCO_3$, 6.0 mmol/L Na_2CO_3

C) 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3

D) 0.25 mmol/L $NaHCO_3$, 10.0 mmol/L Na_2CO_3

E) 0.25 mmol/L $NaHCO_3$, 12.0 mmol/L Na_2CO_3



	Metrosep A Supp 19 - 150/4.0	mg/L
1	Fluoride	10
2	Chloride	10
3	Nitrite	10
4	Bromide	10
5	Nitrate	10
6	phosphate	10
7	Sulfate	10

Due to the strong elution strength of sodium carbonate, the influence of the sodium carbonate content in the eluent is much stronger than the influence of the sodium hydrogen carbonate content. Increasing the sodium carbonate content in the eluent significantly reduces the retention times of all anions. The multivalent anions phosphate and sulfate are accelerated the most here. In a weak eluent (0.25 mmol/L $NaHCO_3$ and 4.0 mmol/L Na_2CO_3), phosphate and sulfate elute late after nitrate. In a concentrated eluent (0.25 mmol/L $NaHCO_3$ and 10.0 mmol/L Na_2CO_3), the baseline separation between nitrate and sulfate is no longer present. In a very strong eluent (0.25 mmol/L $NaHCO_3$ and 12.0 mmol/L Na_2CO_3), phos-

phate elutes before nitrate. The acceleration of the anions due to the use of a stronger eluent also causes higher peaks.

5.5 Variation with organic modifier

5.5.1 Variation of the acetone concentration

Column: Metrosep A Supp 19 - 150/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

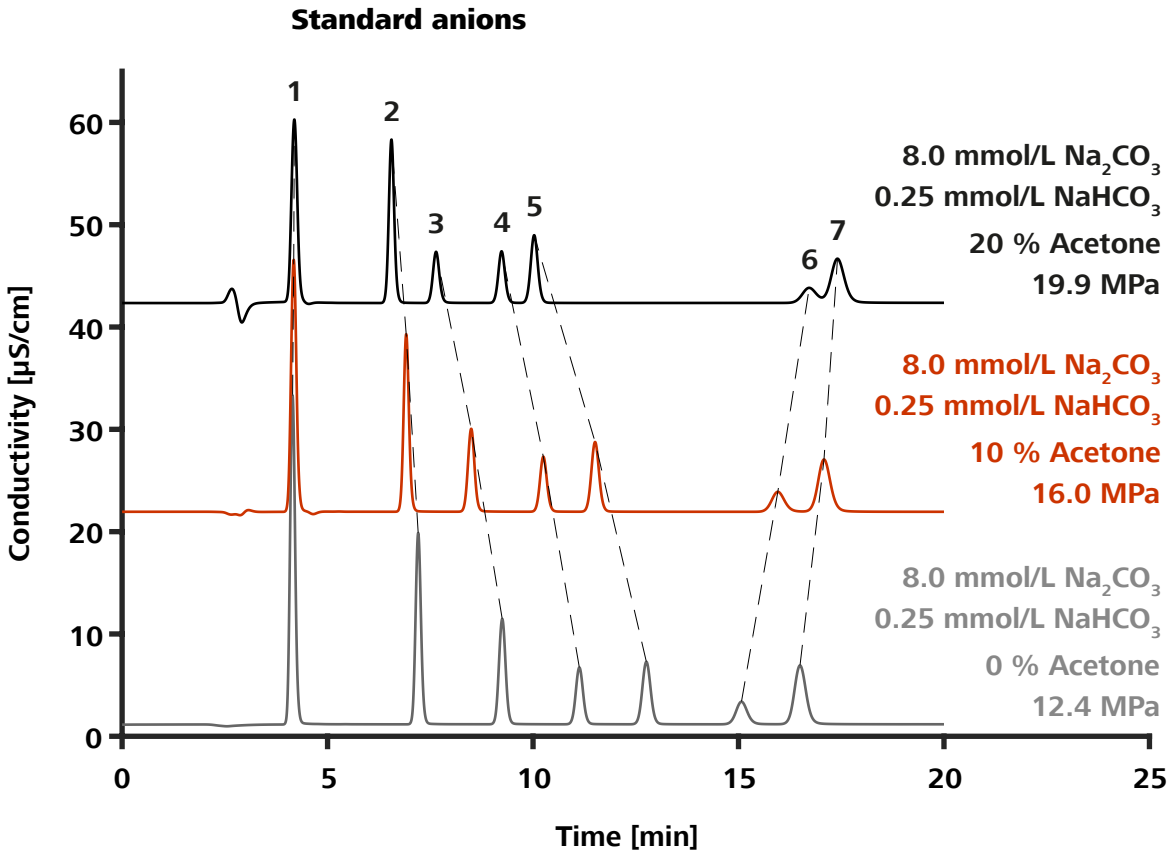
Loop: 20 µL

Flow rate: 0.7 mL/min

Eluent: A) 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3, 0% acetone

B) 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3, 10% acetone

C) 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3, 20% acetone

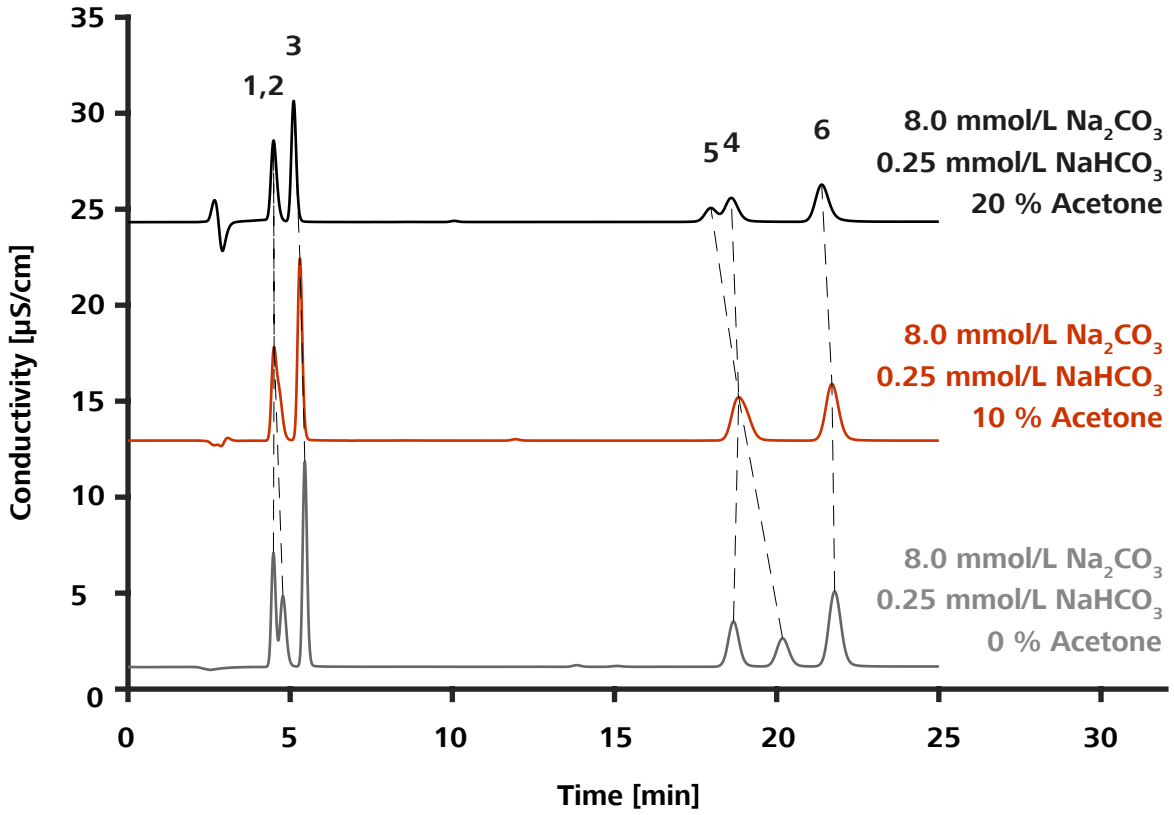


	Metrosep A Supp 19 - 150/4.0	mg/L
1	Fluoride	10
2	Chloride	10
3	Nitrite	10
4	Bromide	10
5	Nitrate	10
6	phosphate	10
7	Sulfate	10

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 acetone content in the eluent accelerates the elution of chloride, nitrite, bromide and nitrate. Phosphate and sulfate, on the other hand, are retarded. Adding acetone to the eluent tends to cause deterioration of the shapes of the peaks and thus also the peak heights.

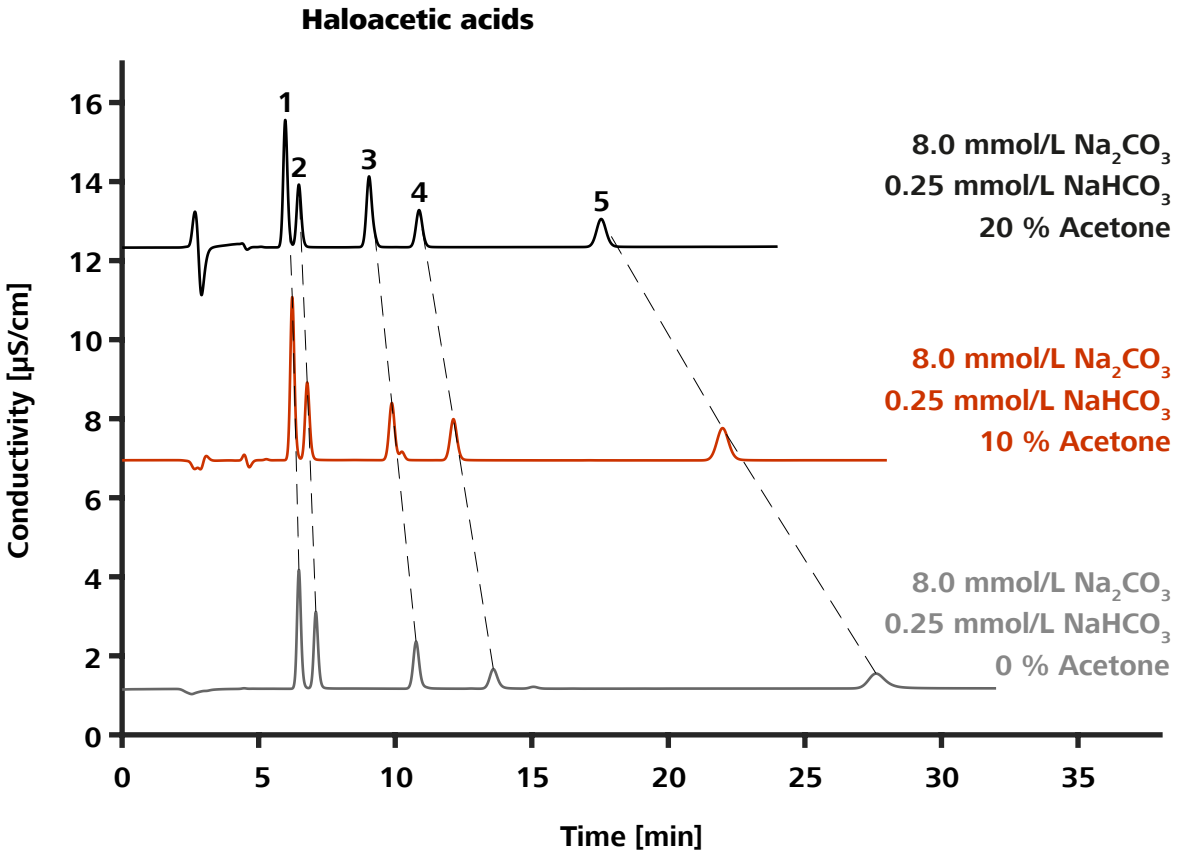
The column backpressure increases with increasing acetone content in the eluent due to the increasing viscosity of the eluent mixture. Without acetone in the eluent, the pressure is approx. 12 MPa. With 20% acetone in the eluent, it is already approx. 20 MPa.

Organic acids



	Metrosep A Supp 19 - 150/4.0	mg/L
1	Glycolate	10
2	Acetate	10
3	Formate	10
4	Tartrate	10
5	Succinate	10
6	Oxalate	10

The reactions of the different organic acids vary greatly when acetone is added to the eluent. Glycolate, formate, tartrate and oxalate remain almost unchanged with 0 to 20% acetone in the eluent. Acetate and succinate, on the other hand, are accelerated by the addition of acetone. Glycolate and acetate as well as tartrate and succinate coelute with 10% acetone. With 20% acetone, succinate overtakes tartrate.



	Metrosep A Supp 19 - 150/4.0	mg/L
1	Monochloroacetate	10
2	Monobromoacetate	10
3	Dichloracetate	10
4	Dibromacetate	10
5	Trichloroacetate	10

Acetone in the eluent accelerates the haloacetic acids. The retention times of all haloacetic acids shorten uniformly and linearly with increasing acetone content.

5.5.2 Variation of the methanol concentration

Column: Metrosep A Supp 19 - 150/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

Loop: 20 µL

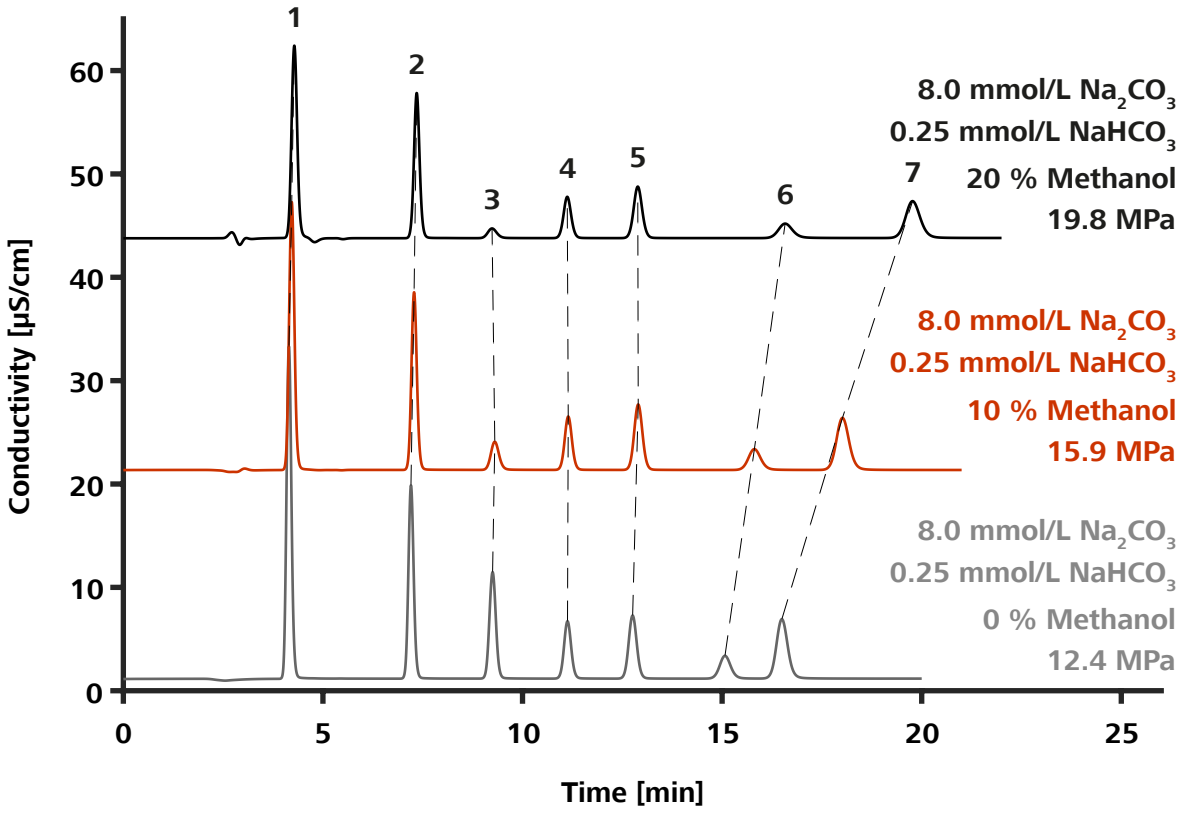
Flow rate: 0.7 mL/min

Eluent: A) 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3, 0% methanol

B) 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3, 10% methanol

C) 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3, 20% methanol

Standard anions

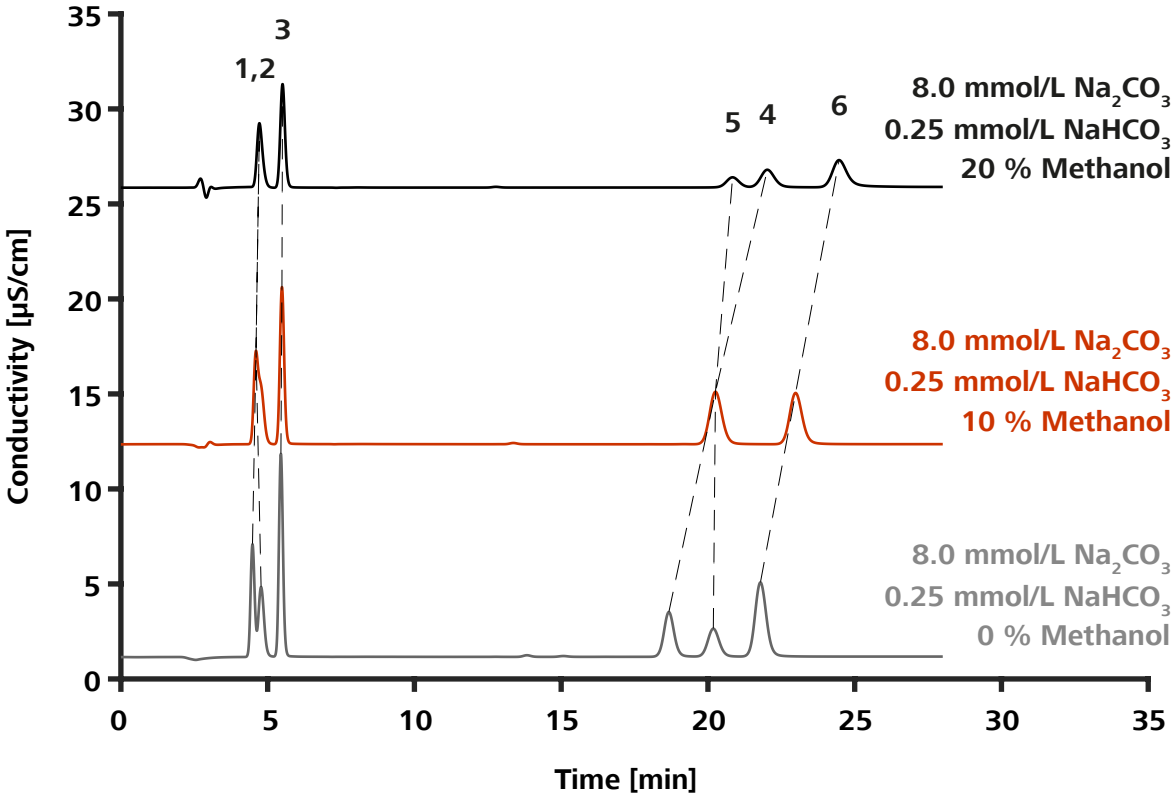


	Metrosep A Supp 19 - 150/4.0	mg/L
1	Fluoride	10
2	Chloride	10
3	Nitrite	10
4	Bromide	10
5	Nitrate	10
6	phosphate	10
7	Sulfate	10

Methanol is also commonly used as an organic modifier. The retention times of the anions fluoride, chloride, nitrite, bromide and nitrate do not change with the addition of methanol. In contrast, the retention times of phosphate and sulfate increase with increasing methanol content. The elution order of the standard anions does not change due to methanol in the eluent. In contrast, the peak areas and peak heights of nitrite and phosphate in particular become significantly smaller with increasing methanol content.

Similar to acetone, methanol contributes to an increase in eluent viscosity. As a result, the column backpressure increases significantly, from approx. 12 MPa with 0% methanol in the eluent to approx. 20 MPa with 20% methanol.

Organic acids

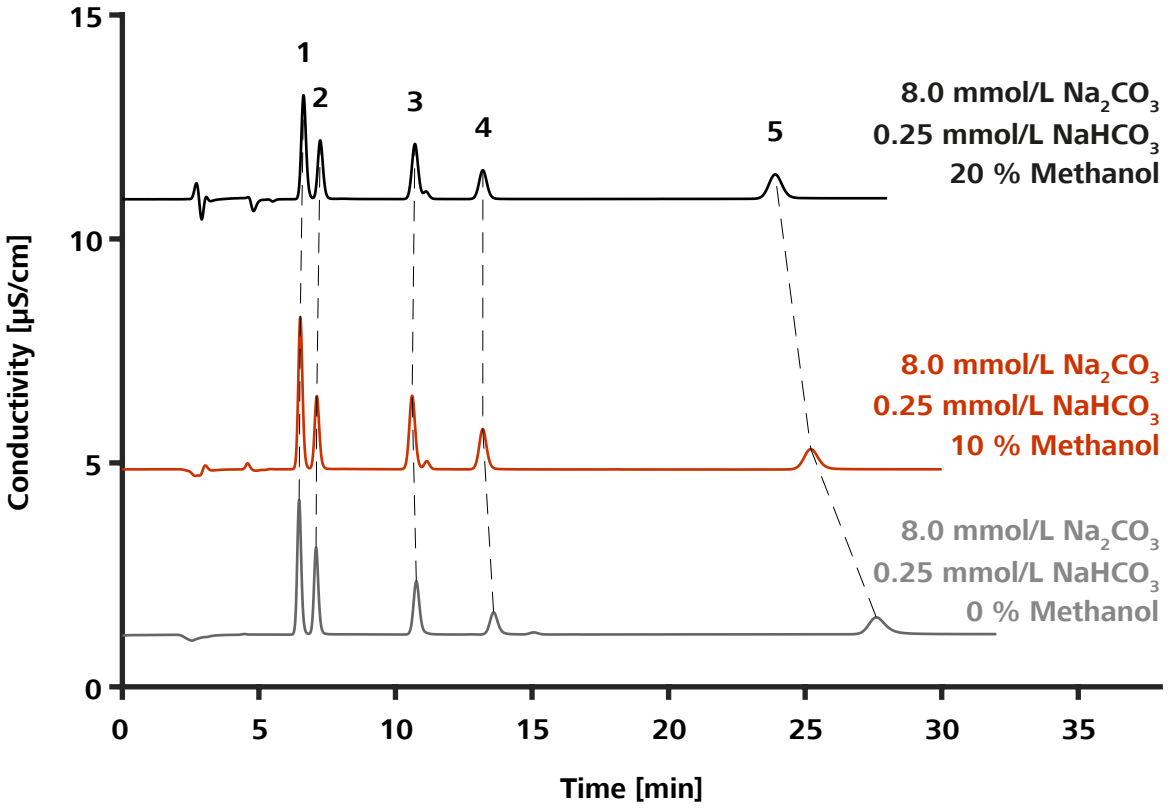


	Metrosep A Supp 19 - 150/4.0	mg/L
1	Glycolate	10
2	Acetate	10
3	Formate	10
4	Tartrate	10
5	Succinate	10

	Metrosep A Supp 19 - 150/4.0	mg/L
6	Oxalate	10

The monovalent organic acids, similar to the monovalent inorganic anions, react hardly at all to the addition of methanol to the eluent. Only the shapes of the peak are affected by the addition of methanol to the eluent. Glycolate and acetate coelute starting with 10% methanol in the eluent. In the case of multivalent organic acids, succinate behaves differently than tartrate and oxalate. Succinate undergoes minimal additional retardation with increasing methanol content. Tartrate and oxalate, by contrast, exhibit a significant shift in retention time. This reverses the elution order between succinate and tartrate.

Haloacetic acids



	Metrosep A Supp 19 - 150/4.0	mg/L
1	Monochloroacetate	10
2	Monobromoacetate	10
3	Dichloracetate	10
4	Dibromacetate	10
5	Trichloroacetate	10

The smaller haloacetic acids react hardly at all to the addition of methanol to the eluent. Only the retention time of trichloroacetate decreases with increasing methanol content.

5.5.3 Variation of the acetonitrile concentration

Column: Metrosep A Supp 19 - 150/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

Loop: 20 µL

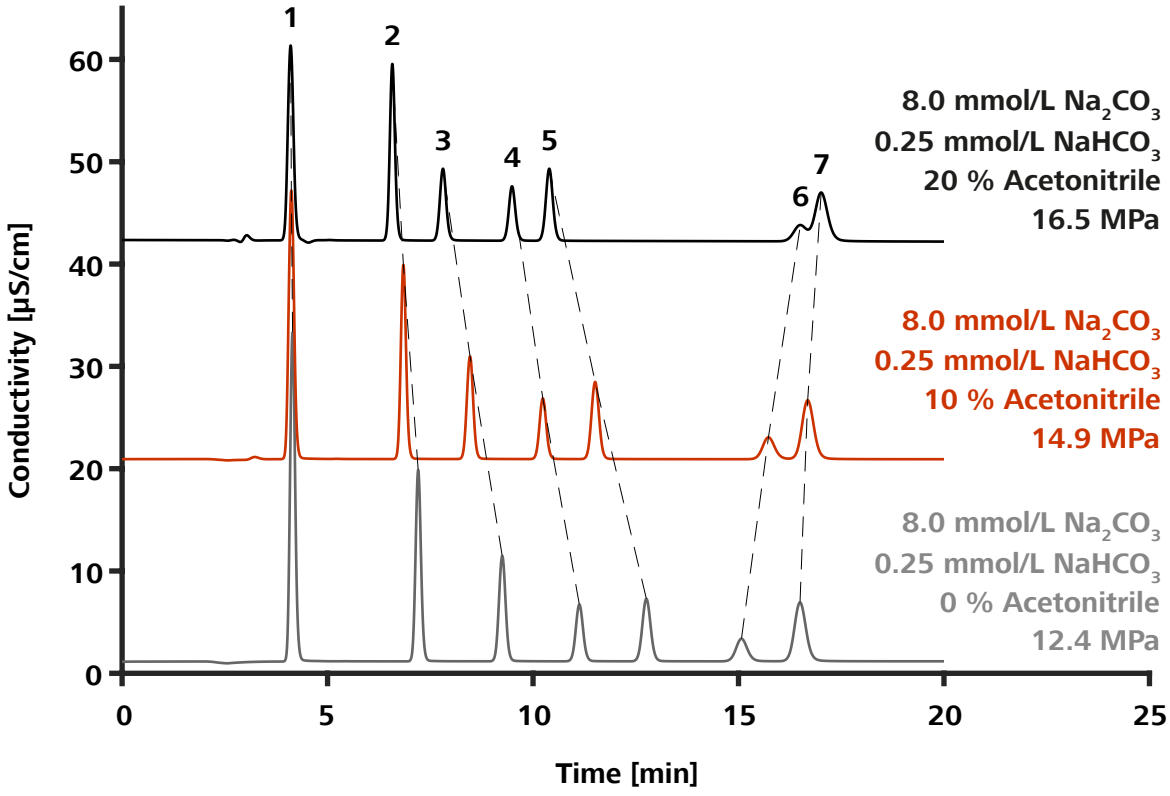
Flow rate: 0.7 mL/min

Eluent: A) 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3, 0% acetonitrile

B) 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3, 10% acetonitrile

C) 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3, 20% acetonitrile

Standard anions

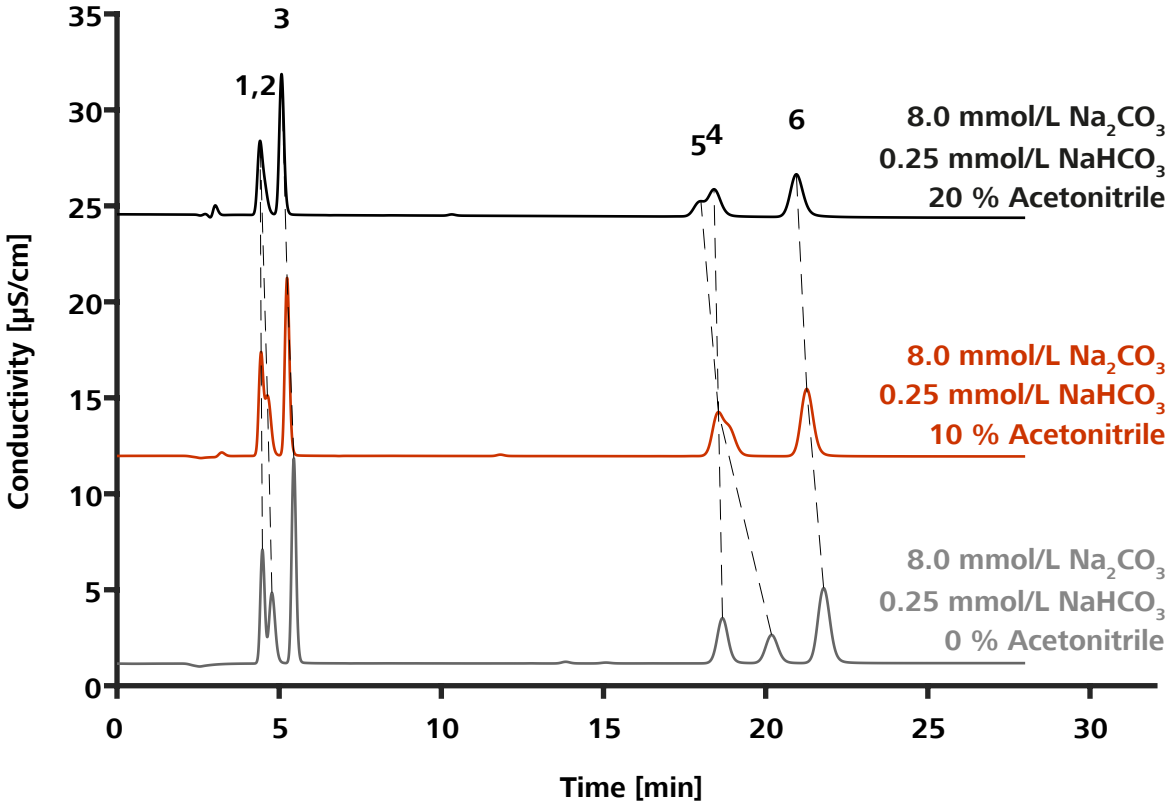


	Metrosep A Supp 19 - 150/4.0	mg/L
1	Fluoride	10
2	Chloride	10
3	Nitrite	10
4	Bromide	10
5	Nitrate	10
6	phosphate	10
7	Sulfate	10

The addition of acetonitrile as an organic modifier contributes significantly less to the pressure increase than does the addition of acetone or methanol. Even with 20% acetonitrile in the eluent, the column backpressure increases by only approx. 33%.

The effect of acetonitrile on retention times is very similar to the effect of acetone: Fluoride, chloride, nitrite, bromide and nitrate elute earlier as acetonitrile content increases. The retention times of phosphate and sulfate are prolonged. In the process, the peak heights and peak areas become only slightly smaller. With 20% acetonitrile, phosphate and sulfate coelute.

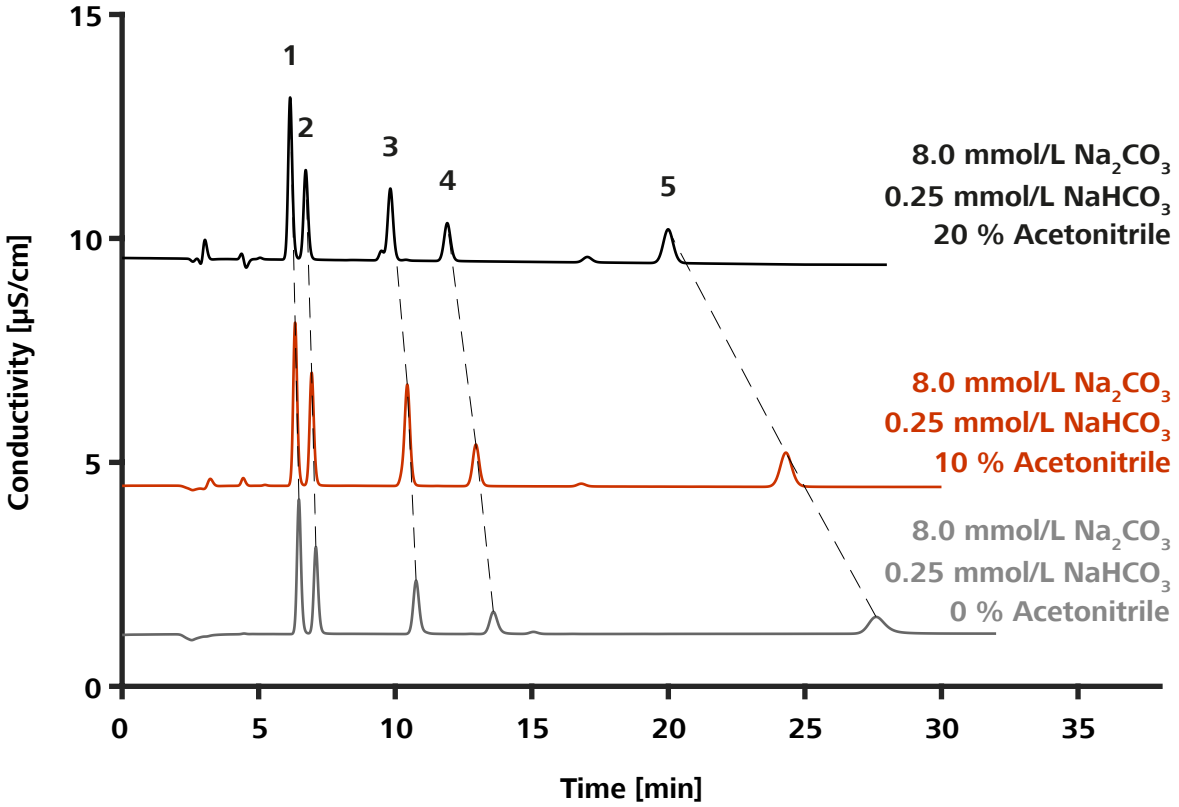
Organic acids



	Metrosep A Supp 19 - 150/4.0	mg/L
1	Glycolate	10
2	Acetate	10
3	Formate	10
4	Tartrate	10
5	Succinate	10
6	Oxalate	10

The retention times of the monovalent organic acids glycolate, acetate and formate and the divalent acids tartrate and oxalate are only slightly affected by the addition of acetonitrile. The peaks become broader with increasing acetonitrile content, causing glycolate and acetate to co-elute. Only the retention time of succinate is strongly dependent on the acetonitrile content. The more acetonitrile in the eluent, the shorter the retention time. With 20% acetonitrile in the eluent, succinate overtakes tartrate.

Haloacetic acids



	Metrosep A Supp 19 - 150/4.0	mg/L
1	Monochloroacetate	10
2	Monobromoacetate	10
3	Dichloracetate	10
4	Dibromacetate	10
5	Trichloroacetate	10

Acetonitrile has only a minimal effect on the retention times of monochloroacetate and monobromoacetate. In contrast to this, the haloacetic acids dichloroacetate, dibromoacetate, and trichloroacetate are significantly accelerated with the addition of acetonitrile.

5.5.4 Variation of the ethanol concentration

Column: Metrosep A Supp 19 - 150/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

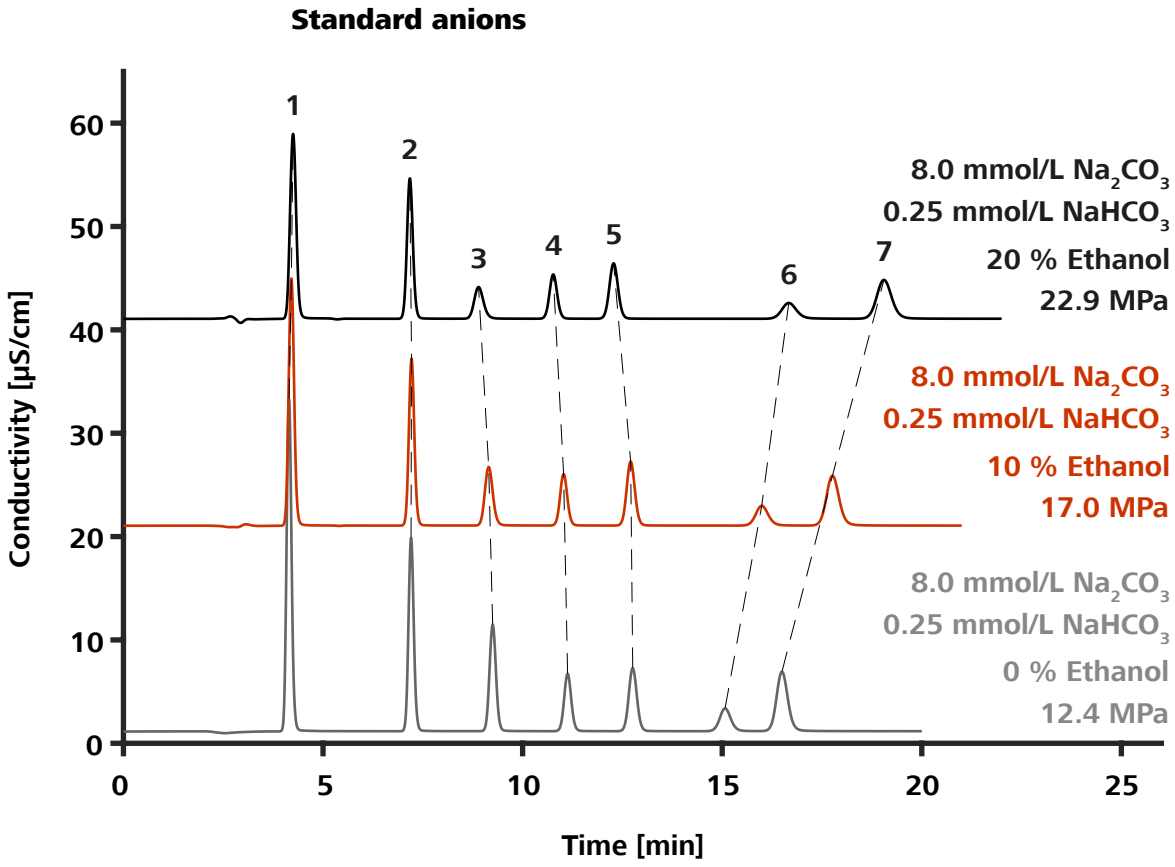
Loop: 20 µL

Flow rate: 0.7 mL/min

Eluent: A) 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3, 0 % ethanol

B) 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3, 10% ethanol

C) 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3, 20% ethanol



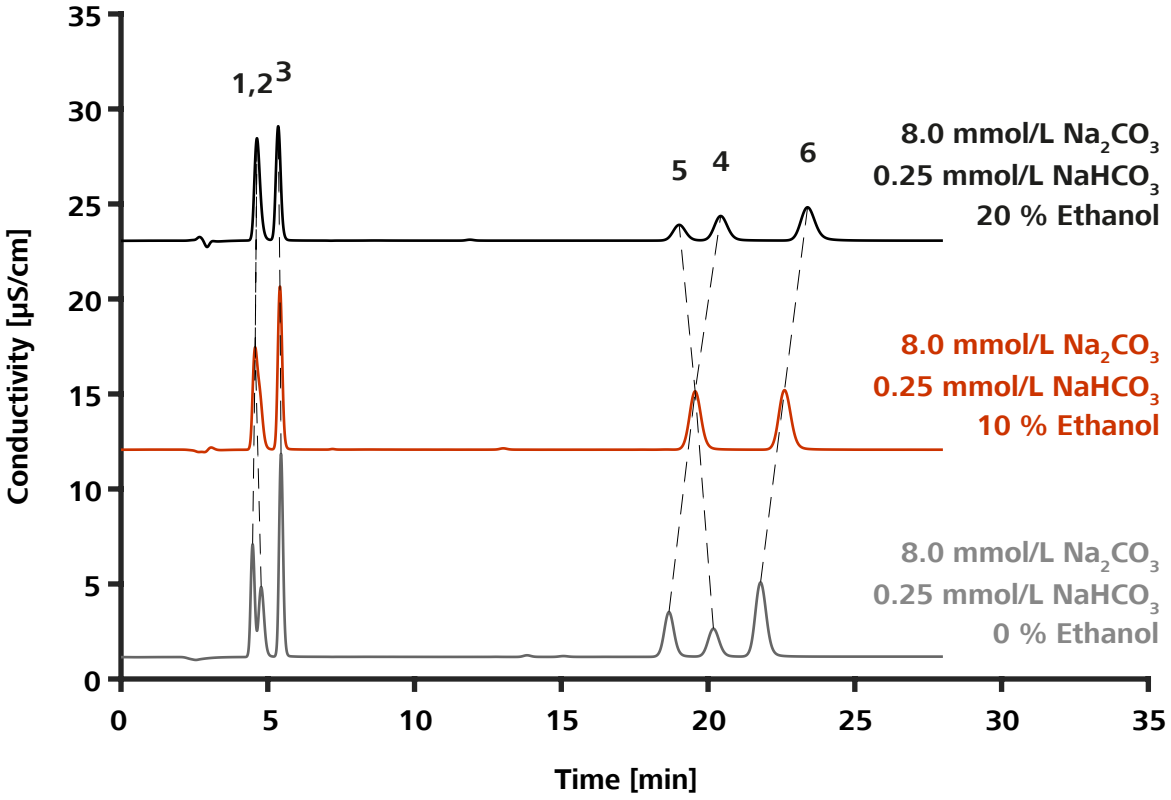
	Metrosep A Supp 19 - 150/4.0	mg/L
1	Fluoride	10
2	Chloride	10
3	Nitrite	10
4	Bromide	10
5	Nitrate	10
6	phosphate	10
7	Sulfate	10

Ethanol is also used as an organic modifier in isolated cases. Ethanol increases the eluent viscosity by far the most, which is then reflected in the column backpressure. Adding 20% ethanol almost doubles the pressure.

The retention times of the monovalent anions are only slightly affected by the addition of ethanol. The peak height and peak area of nitrite and phosphate decrease with increasing ethanol content. The multivalent anions are retarded with the addition of ethanol. This improves the sepa-

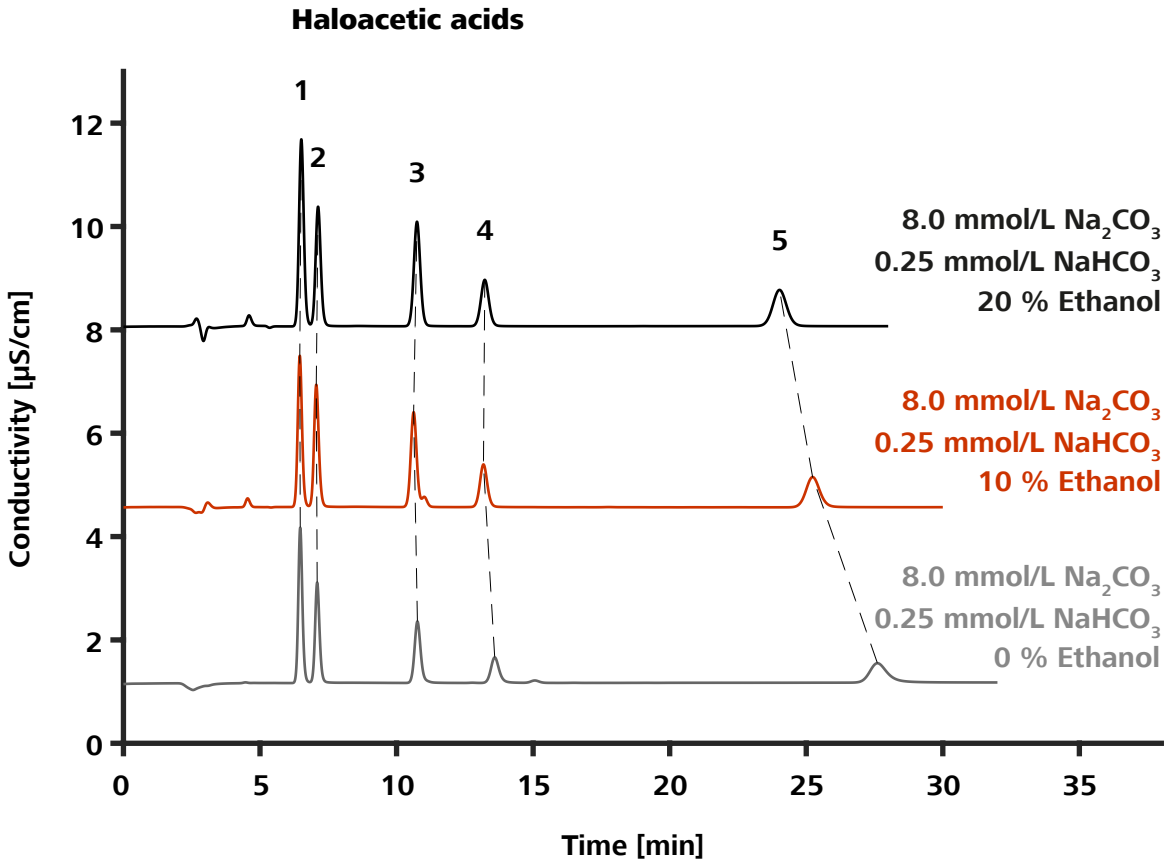
ration between phosphate and sulfate. The effect of ethanol on the separation behavior is comparable to that of methanol.

Organic acids



	Metrosep A Supp 19 - 150/4.0	mg/L
1	Glycolate	10
2	Acetate	10
3	Formate	10
4	Tartrate	10
5	Succinate	10
6	Oxalate	10

The retention times of the monovalent organic acids are not affected by the addition of ethanol to the eluent. Due to peak widening, glycolate coelutes with acetate starting with 10% ethanol. Succinate is slightly accelerated with the addition of ethanol, whereas tartate and oxalate become retarded with increasing ethanol content.



	Metrosep A Supp 19 - 150/4.0	mg/L
1	Monochloroacetate	10
2	Monobromoacetate	10
3	Dichloracetate	10
4	Dibromacetate	10
5	Trichloroacetate	10

Except for trichloroacetate, the haloacetic acids show no change following addition of ethanol to the eluent. Their retention time does not change, regardless of ethanol content. Trichloroacetate is slightly accelerated with increasing ethanol content.

5.6 Determination of standard anions in mineral water samples

Column: Metrosep A Supp 19 - 150/4.0

Sample preparation: –

Detection: Conductivity

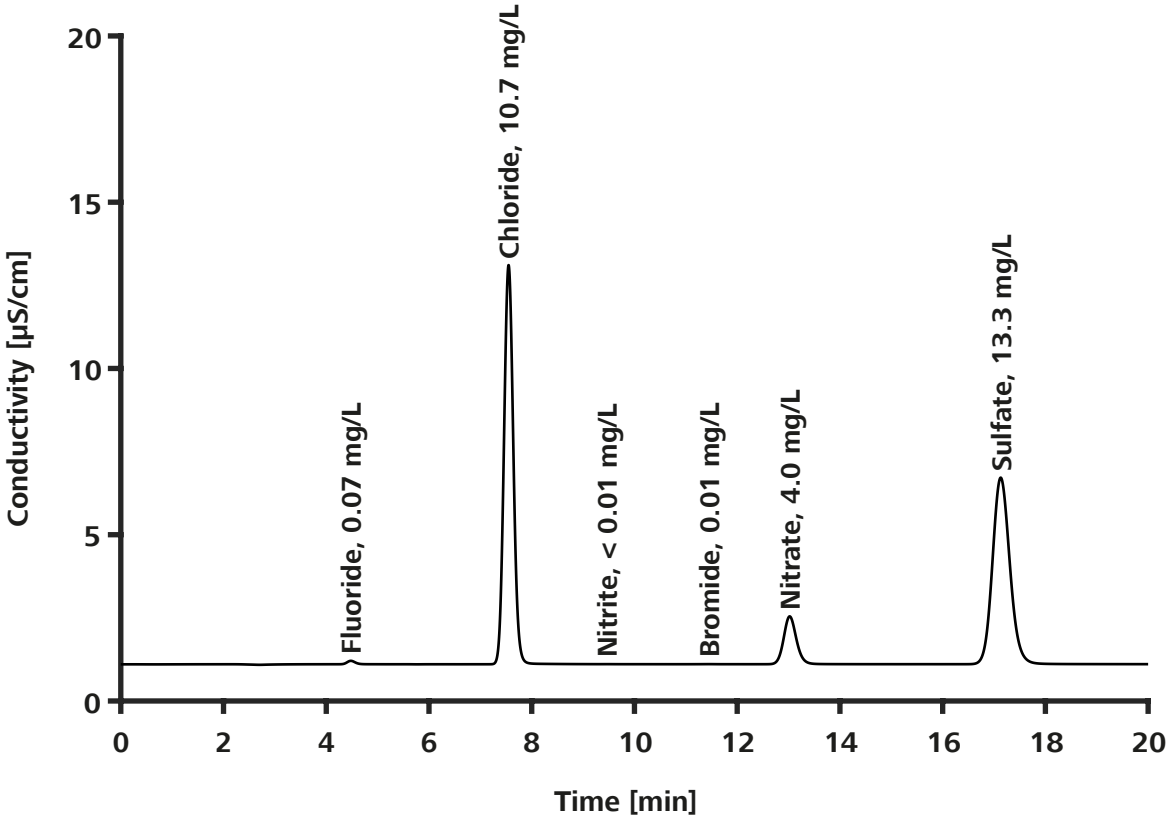
Suppression: Sequential suppression with MSM and MCS

Temperature: 25 °C

Loop: 20 µL

Flow rate: 0.7 mL/min

Eluent: 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3



5.7 Determination of standard anions and organic acids in boiler feed water of power plants

Column: Metrosep A Supp 19 - 250/4.0

Sample preparation: –

Detection: Conductivity

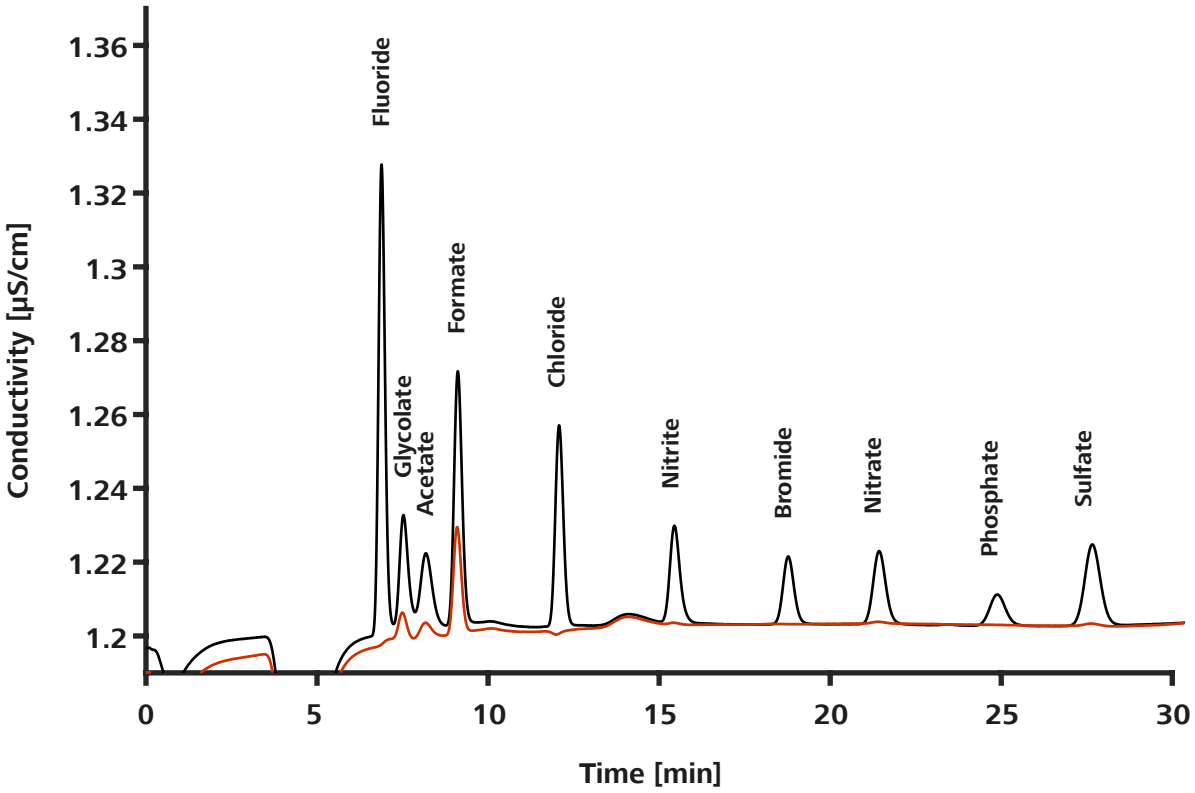
Suppression: Sequential suppression with MSM and MCS

Temperature: 25 °C

Loop: 1,000 µL (preconcentration)

Flow rate: 0.75 mL/min

Eluent: 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3



The red chromatogram corresponds to boiler feed water with 4.0 mg/L ethanolamine and 0.4 mg/L ammonia. The black chromatogram shows the same sample to which 2 µg/L of the standard anions and of the organic acids glycolate, acetate and formate were added.

5.8 Direct determination of standard anions in bioethanol

Column: Metrosep A Supp 19 - 150/4.0

Sample preparation: –

Detection: Conductivity

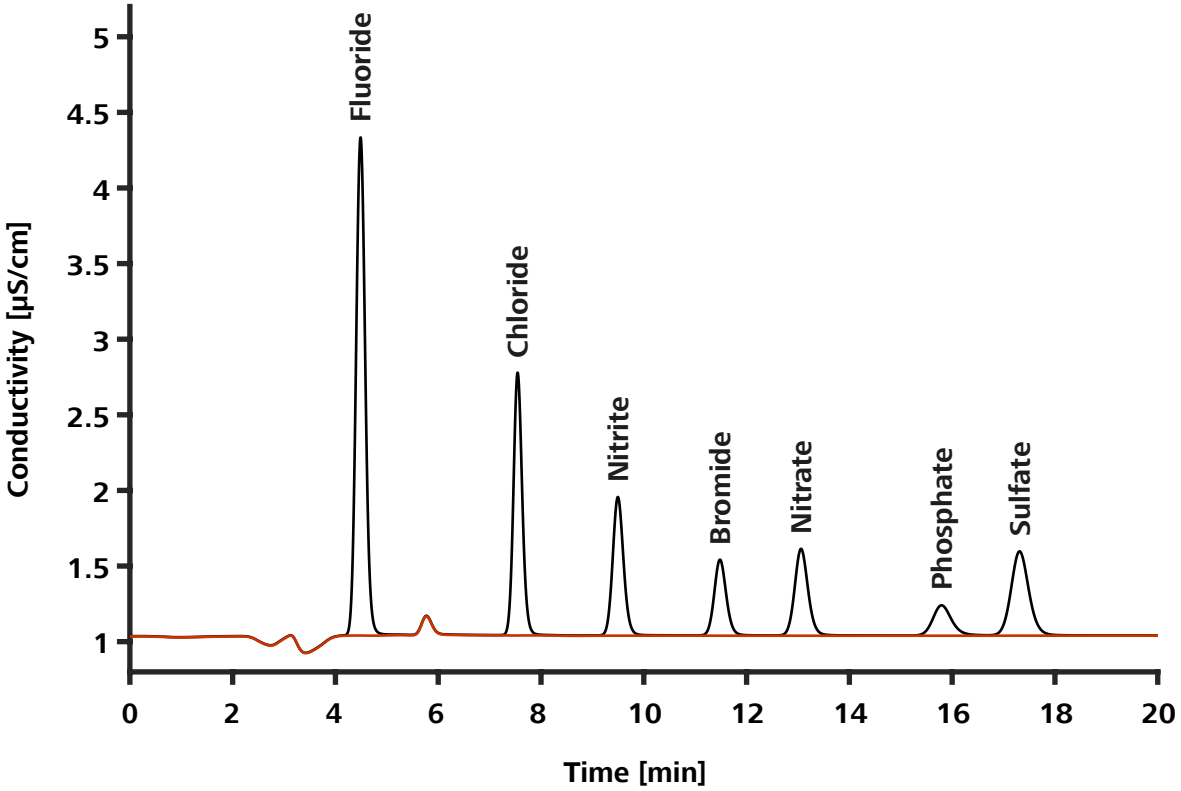
Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.7 mL/min

Eluent: 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3



The red chromatogram corresponds to the direct injection of the ethanol sample onto the column. The Metrosep A Supp 19 - 150/4.0 is inert when injecting organic solvents, such as ethanol. The black chromatogram

shows the same sample to which 2 µg/L of the standard anions were added.

5.9 Determination of fluoride in dental gel according to USP

Column: Metrosep A Supp 19 - 150/4.0

Sample preparation: –

Detection: Conductivity

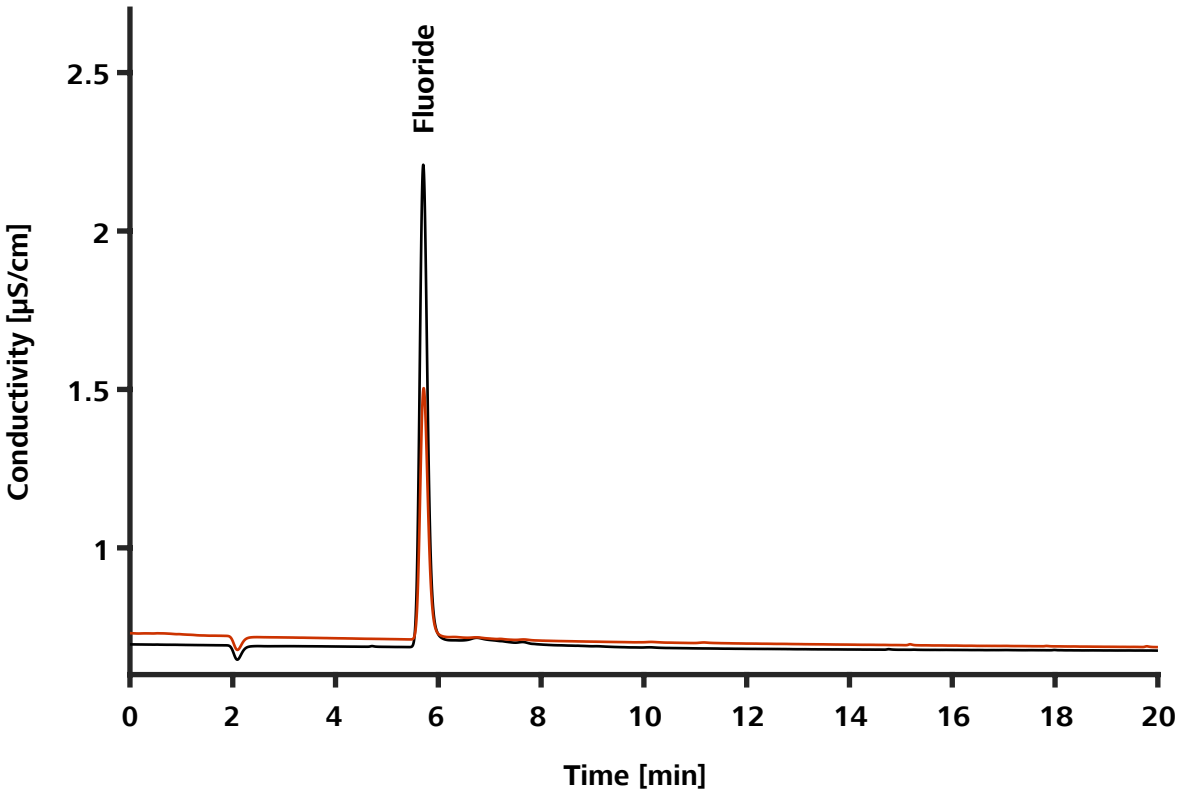
Suppression: Sequential suppression with MSM and MCS

Temperature: 40 °C

Loop: 20 µL

Flow rate: 1.0 mL/min

Eluent: 15.0 mmol/L KOH



In this method, the current monograph was repeated with the Metrosep A Supp 19. The red chromatogram corresponds to the dental

gel that contains a nominal 1 µg/mL NaF. The black chromatogram shows the same sample to which 1 µg/mL NaF was added. Thus, it shows that the Metrosep A Supp 19 is also suitable for USP methods.

5.10 Direct determination of standard anions in lactose-free milk

Column: Metrosep A Supp 19 - 150/4.0

Sample preparation: Dilution 1:50

Detection: Conductivity

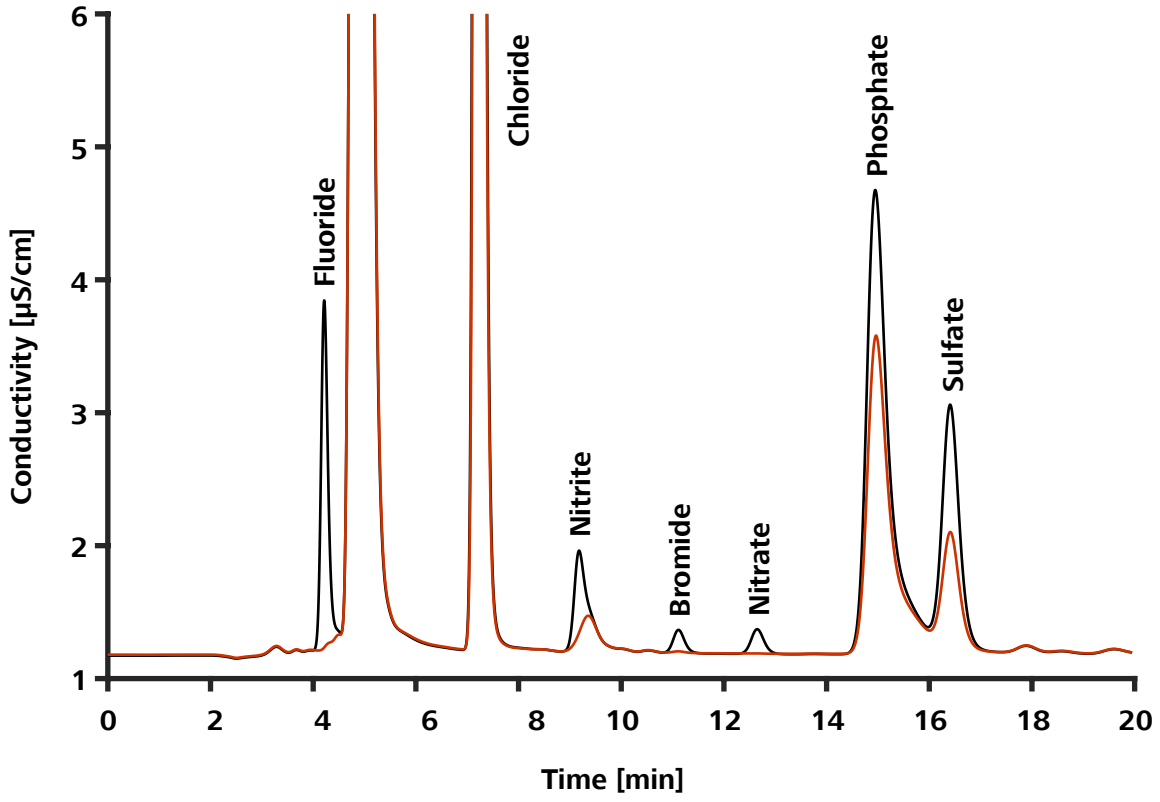
Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.75 mL/min

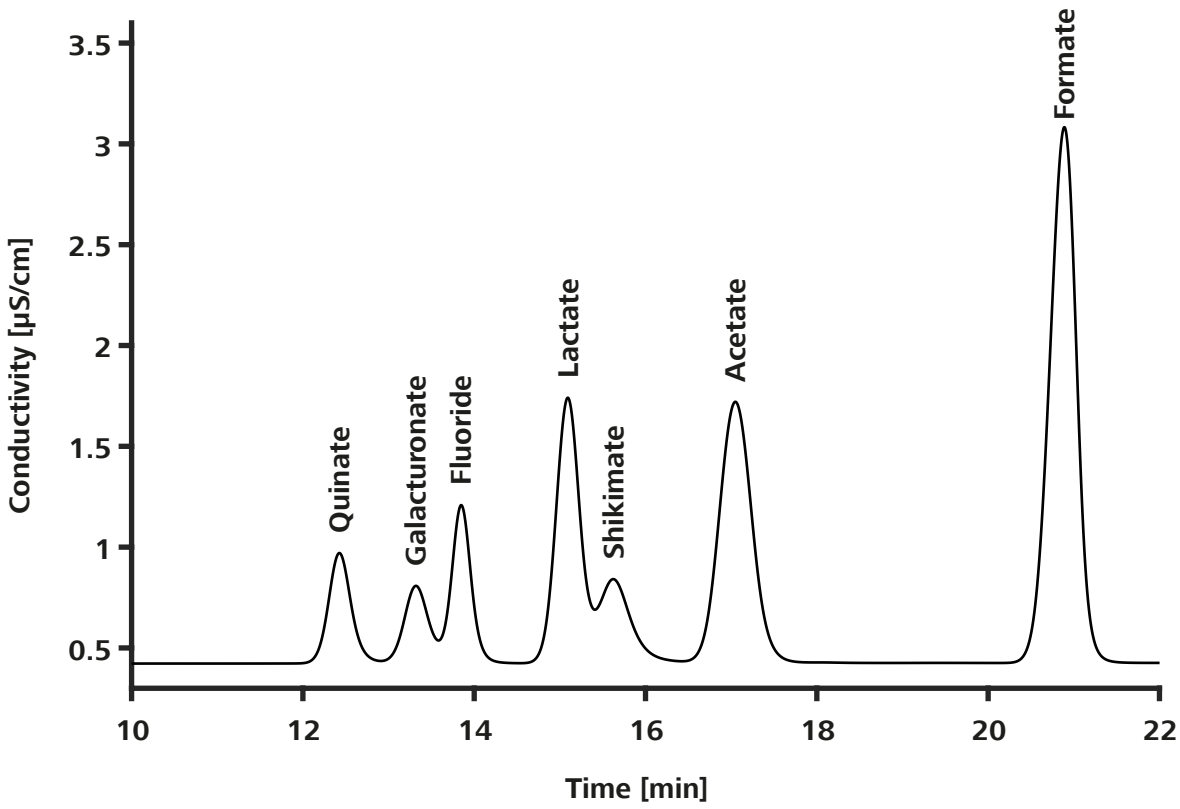
Eluent: 0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3



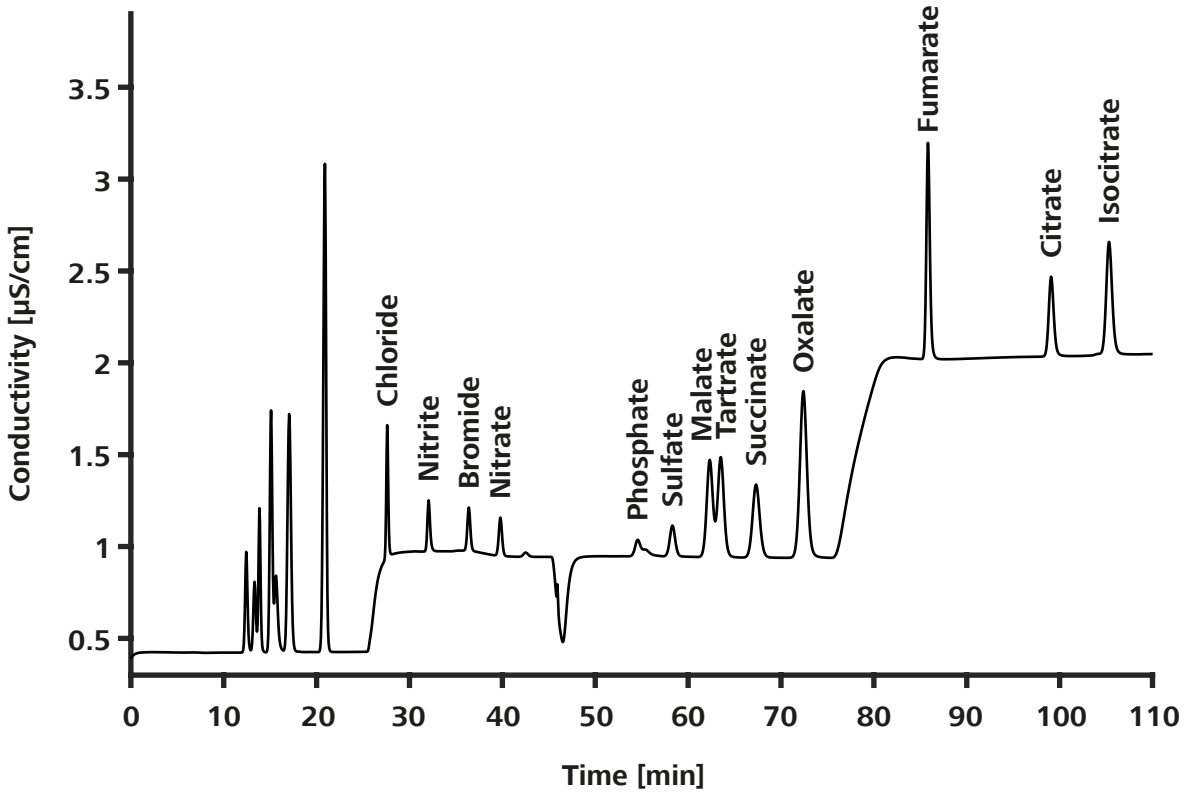
Lactose-free milk was analyzed with Metrosep A Supp 19 for standard anions (fluoride, chloride, nitrite, bromide, nitrate, phosphate and sulfate). For this purpose, the sample was diluted 1:50 in ultrapure water. The red chromatogram shows the original sample. The black chromatogram shows the same sample to which 1 µg/L of the standard anions was added. The standard anions can be readily determined under these conditions. An organic acid elutes between fluoride and chloride.

5.11 Determination of standard anions and 13 organic acids in food samples with conductivity

Column:	Metrosep A Supp 19 - 250/4.0
Sample preparation:	-
Detection:	Conductivity
Suppression:	Sequential suppression with MSM and MCS
Temperature:	25 °C
Loop:	20 µL
Flow rate:	0.7 mL/min
Eluent:	A) 1.0 mmol/L Na_2CO_3 B) 50.0 mmol/L Na_2CO_3 0–18 min: 100% A 18–20 min: 100–90% A 20–70 min: 90% A 70–75 min: 90–40% A 75–107 min: 40% A 107–120 min: 100% A



In addition to the determination of standard anions, the Metrosep A Supp 19 - 250/4.0 is particularly suitable for the determination of low-molecular-weight organic acids. Quinate, galacturonate, fluoride, lactate, shikimate, acetate and formate can be reliably resolved in the front part of the chromatogram with gradient elution. The column can thus be of excellent use for determinations in food samples.



In the back part of the chromatogram, higher eluent concentrations are used to elute the multivalent analytes. Organic acids are also dissolved after sulfate.

	Metrosep A Supp 19 - 250/4.0	mg/L
1	Quinate	2.5
2	Galacturonate	2.5
3	Fluoride	2.5
4	Lactate	2.5
5	Shikimate	2.5
6	Acetate	2.5
7	Formate	2.5
8	Chloride	1
9	Nitrite	1
10	Bromide	1
11	Nitrate	1
12	phosphate	1
13	Sulfate	1

	Metrosep A Supp 19 - 250/4.0	mg/L
14	Malate	5
15	Tartrate	5
16	Succinate	5
17	Oxalate	5
18	Fumarate	5
19	Citrate	5
20	Isocitrate	10

5.12 Determination of standard anions and 16 organic acids with IC-MS

Column: Metrosep A Supp 19 - 150/4.0

Sample preparation: -

Detection: Mass spectrometry

Suppression: Sequential suppression with MSM and MCS

Temperature: 60 °C

Loop: 10 µL

Flow rate: 0.75 mL/min

Eluent: High-pressure gradient

A) 8.0 mmol/L Na_2CO_3, 0.25 mmol/L $NaHCO_3$, 10% MeOH (v/v)

B) 80.0 mmol/L Na_2CO_3, 2.5 mmol/L $NaHCO_3$, 10% MeOH (v/v)

0–8 min: 99% A

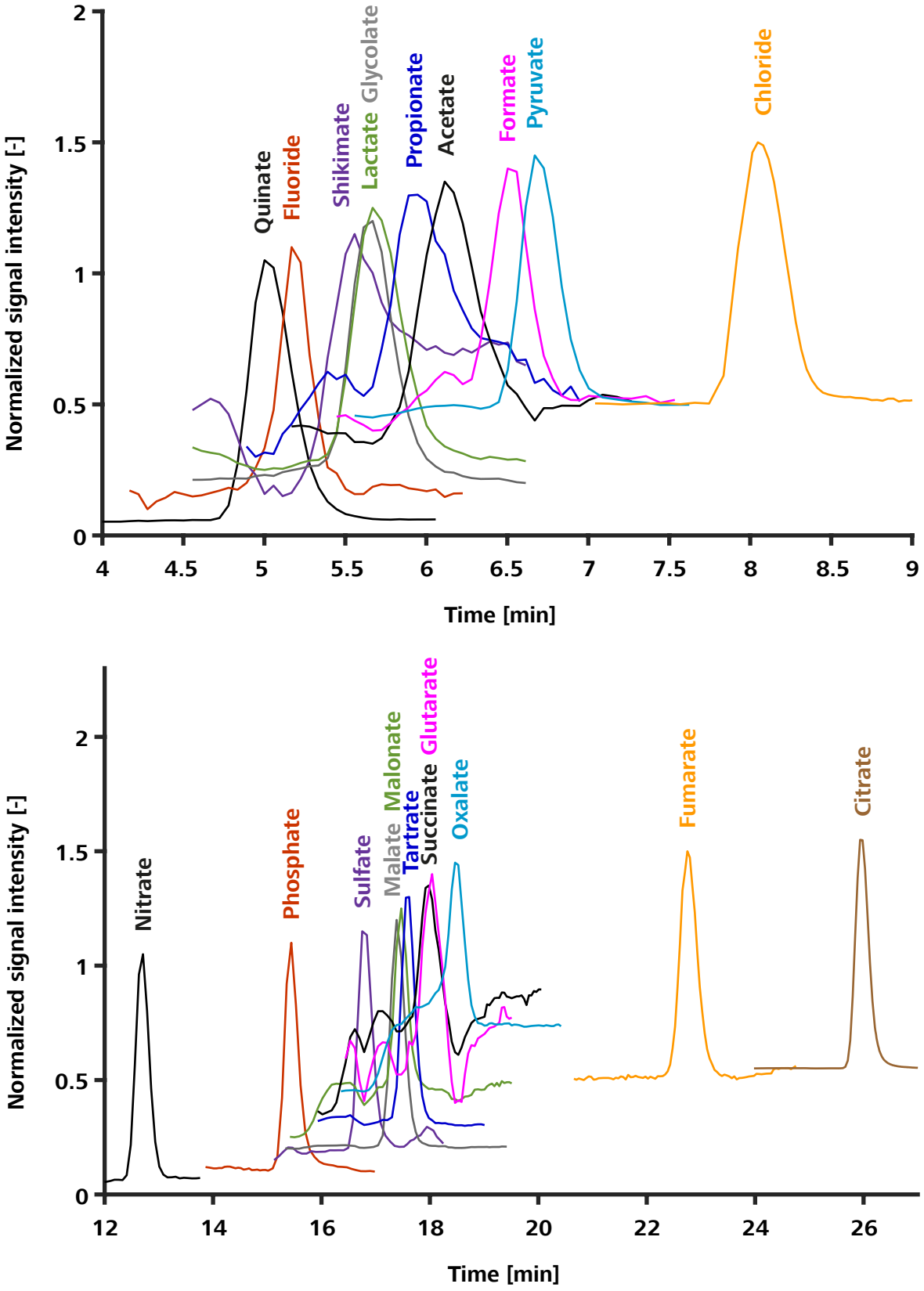
8–15 min: 99–80% A

15–20.9 min: 80–75% A

20.9–21 min: 75–20% A

21–28 min: 20% A

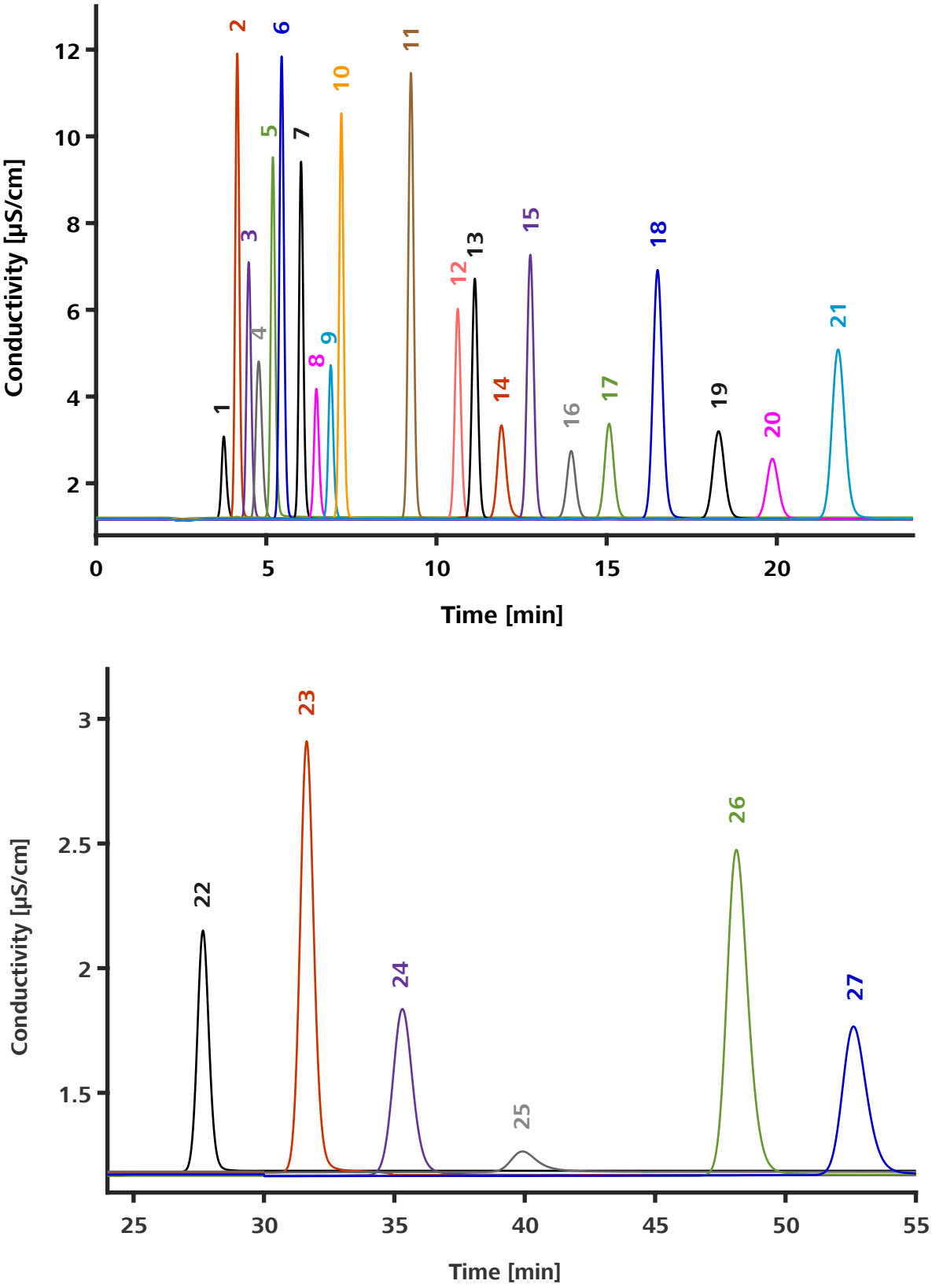
28–29.9 min: 99% A


Normalized signal intensity [-]
Time [min]
Quinate
Fluoride
Shikimate
Lactate
Glycolate
Propionate
Acetate
Formate
Pyruvate
Chloride
Nitrate
Phosphate
Sulfate
Malonate
Malate
Tartrate
Succinate
Glutarate
Oxalate
Fumarate
Citrate

Mass spectrometry can be used to determine analytes that are not completely separated on the analytical column. The analysis time of 30 minutes can thus be significantly reduced. In the application shown here, 200 µg/L of a wide variety of organic acids were added to a tea. In the front part of the chromatogram, 9 analytes can be determined: Quinate, fluoride, shikimate, lactate, glycolate, propionate, acetate, formate and pyruvate. For multivalent organic acids, malate, malonate, tartrate, succinate, glutarate, oxalate, fumarate and citrate are determined.

5.13 27 anions with one column

Column:	Metrosep A Supp 19 - 150/4.0
Sample preparation:	-
Detection:	Conductivity
Suppression:	Sequential suppression with MSM and MCS
Temperature:	30 °C
Loop:	20 µL
Flow rate:	0.7 mL/min
Eluent:	0.25 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3


Conductivity [µS/cm]
Time [min]
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27

	Metrosep A Supp 19 - 150/4.0	mg/L
1	Gluconate	10
2	Fluoride	10
3	Glycolate	10
4	Acetate	10
5	Monofluoracetate	10
6	Formate	10
7	Chlorite	10
8	Monochloroacetate	10
9	Difluoracetate	10
10	Chloride	10
11	Nitrite	10
12	Chlorate	10
13	Bromide	10
14	Dalapon	10
15	Nitrate	10
16	Phosphite	10
17	phosphate	10
18	Sulfate	10
19	Malate	10
20	Maleate	10
21	Oxalate	10
22	Iodide	10
23	Thiosulfate	10
24	Molybdate	10
25	Chloride bromoacetate	10
26	Thiocyanate	10
27	Perchlorate	10

A large number of different anions can be separated isocratically with the Metrosep A Supp 19 - 150/4.0.

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

NOTICE

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 59)*

- Contamination with inorganic components *(see table 4, page 59)*.

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	0.4	Direction against the flow
2	Acetonitrile-water mixture (50:50)	2	0.4	Direction against the flow
3	Ultrapure water	1	0.4	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 Na_2CO_3, 2.5 mmol/L $NaHCO_3$	2	0.4	Regular
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:

- Application Note D-003: Quality control of dialysis concentrates
- White Paper WP-086: Measuring organic acids and inorganic anions with ion chromatography mass spectrometry
- Column catalog, 8.000.5347

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