Column manual

Metrosep A Supp 10 (6.1020.XX0)

Manual

8.0107.8009EN / 2021-10-08

Metrohm

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Technical Communication
Metrohm AG
CH-9100 Herisau

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

Disclaimer

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

Table of contents

1 General information

The Metrosep A Supp 10 is an anion separation column with high capacity. This column is particularly suitable for the determination of samples with high ionic strength and big fluctuations in concentration. The Metrosep A Supp 10 is used for analyzing inorganic low-molecular anions.

1.1 Ordering information

Table 1 4-mm columns

Order number	Designation
6.1020.050	Metrosep A Supp 10 - 50/4.0
6.1020.070	Metrosep A Supp 10 - 75/4.0
6.1020.010	Metrosep A Supp 10 - 100/4.0
6.1020.030	Metrosep A Supp 10 - 250/4.0

Table 2 2-mm columns

Order number	Designation
6.1020.250	Metrosep A Supp 10 - 50/2.0
6.1020.270	Metrosep A Supp 10 - 75/2.0
6.1020.210	Metrosep A Supp 10 - 100/2.0
6.1020.220	Metrosep A Supp 10 - 150/2.0
6.1020.230	Metrosep A Supp 10 - 250/2.0

Table 3 Guard columns

Order number	Designation
6.1020.500	Metrosep A Supp 10 Guard/4.0
6.1020.510	Metrosep A Supp 10 S-Guard/4.0
6.1020.520	Metrosep A Supp 10 Guard HC/4.0
6.1020.600	Metrosep A Supp 10 Guard/2.0
6.1020.600	Metrosep A Supp 10 S-Guard/4.0

1.2 Technical specifications

Column material

Polystyrene/divinylbenzene copolymer with quaternary ammonium groups

Particle size

4.6 µm

Measurements

Order number	Measurements
6.1020.050	50 x 4.0 mm
6.1020.070	75 x 4.0 mm
6.1020.010	100 x 4.0 mm
6.1020.030	250 x 4.0 mm
6.1020.250	50 x 2.0 mm
6.1020.270	75 x 2.0 mm
6.1020.210	100 x 2.0 mm
6.1020.220	150 x 2.0 mm
6.1020.230	250 x 2.0 mm

pH range eluent

0 … 14

ph range sample

0 … 14

Temperature range

10–70 °C

Recommended standard temperature

45 °C

Maximum pressure

all dimensions	25 MPa (250 bar)

Flow rate

Order number	Recommended flow rate	Maximum flow rate
6.1020.050	1.0 mL/min	2.0 mL/min
6.1020.070	1.0 mL/min	2.0 mL/min
6.1020.010	1.0 mL/min	2.0 mL/min
6.1020.030	1.0 mL/min	2.0 mL/min
6.1020.250	0.25 mL/min	1.3 mL/min
6.1020.270	0.25 mL/min	1.1 mL/min
6.1020.210	0.25 mL/min	0.9 mL/min
6.1020.220	0.25 mL/min	0.7 mL/min

Order number	Recommended flow rate	Maximum flow rate
6.1020.230	0.25 mL/min	0.7 mL/min

Standard eluent

5.0 mmol/L sodium carbonate (Na_2CO_3) and 5.0 mmol/L sodium hydrogen carbonate ($NaHCO_3$)

Permitted organic additives

In the eluent

0–100% acetonitrile, acetone and methanol

In the sample matrix

0–100% acetonitrile, acetone and methanol

Capacity

Order number	Capacity
6.1020.050	17 µmol (Cl^-)
6.1020.070	22 µmol (Cl^-)
6.1020.010	37 µmol (Cl^-)
6.1020.030	100 µmol (Cl^-)
6.1020.250	4.6 µmol (Cl^-)
6.1020.270	6.9 µmol (Cl^-)
6.1020.210	8.7 µmol (Cl^-)
6.1020.220	15 µmol (Cl^-)
6.1020.230	24 µmol (Cl^-)

Preparation

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

Storage

Store the column in the standard eluent and at ambient temperature.

Typical pressure

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

Order number	Typical pressure
6.1020.050	5.0 ± 1.0 MPa
6.1020.070	7.0 ± 1.0 MPa
6.1020.010	8.5 ± 1.0 MPa
6.1020.030	15.0 ± 1.0 MPa
6.1020.250	3.0 ± 1.0 MPa
6.1020.270	4.5 ± 1.0 MPa
6.1020.210	5.5 ± 1.0 MPa
6.1020.220	8.0 ± 1.0 MPa

Order number	Typical pressure
6.1020.230	11.0 ± 1.0 MPa

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

Application Determination of inorganic anions and low-molecular anions with chemical and sequential suppression

2 Key aspects of working with separation columns

Storage

Once the backpressure in 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 ambient temperature.

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% solvent-resistant.

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.

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).

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.

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 packing (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. 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 polymethacrylate columns and 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. If not specified otherwise on the column leaflet, store the column at ambient temperature.

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 resistivity 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 level of purity for chemicals for both standard production and eluent production.

3.1 Chemicals

Recommended chemicals

- Sodium carbonate
  Merck order number: 1.06393.1000
- Sodium hydrogen carbonate
  Merck order number: 1.06329.1000
- Ultrapure water of type I (see ASTM D1193)
  Resistance > 18.2 MΩ·cm (25 °C)
  TOC < 10 µg/L

3.2 Production of standard eluent

Proceed as follows to produce 2 L of standard eluent with 5.0 mmol/L of sodium carbonate and 5.0 mmol/L of sodium hydrogen 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 1059.9 mg of sodium carbonate.
- Weigh 840.1 mg of sodium hydrogen carbonate.

- Add the weighed amounts of sodium carbonate and sodium hydrogen carbonate to the ultrapure water.

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

This eluent (5.0 mmol/L of sodium carbonate and 5.0 mmol/L of sodium hydrogen carbonate) and chemical suppression can be used to achieve background conductivity of <24 µS/cm. The noise is typically less than 0.2 nS/cm.

4 Start-up

4.1 Connecting and rinsing the guard column

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

NOTICE

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

NOTICE

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

CAUTION

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

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

NOTICE

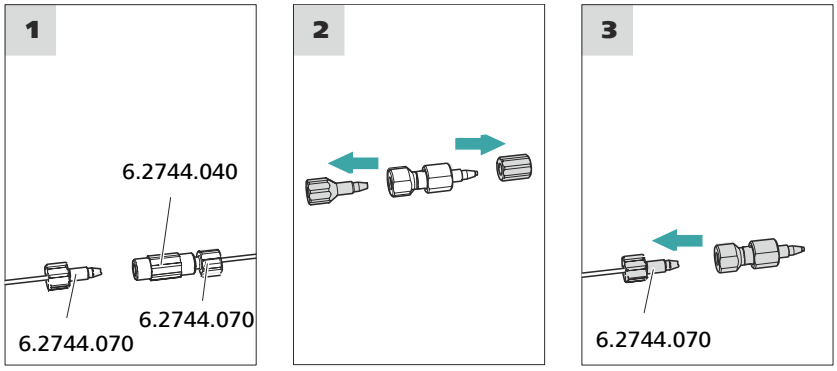
Only connect the guard column after the initial start-up of the instrument . Until then, replace the guard column and the separation column with couplings (6.2744.040).

Accessories

For this step, you need the following accessories:

- Guard column (suitable for separation column)

Connecting the guard column



1 Removing the coupling

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

2 Preparing the guard column

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

3 Connecting the guard column

CAUTION

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

- Fasten the inlet of the guard column to the column inlet capillary using a short pressure screw (6.2744.070).
- If the guard column is connected to the separation column using a connection capillary, fasten this connection capillary to the guard column outlet with a pressure screw.

Rinsing the guard column

1 Rinsing the guard column

- Place a beaker under the guard column's outlet.

- Start manual control in MagIC Net and select the high-pressure pump: **Manual ► Manual control ► Pump**
  - **Flow**: **in accordance with column leaflet**
  - **On**
- Rinse the guard column with eluent for approx. 5 minutes.
- Stop the high-pressure pump in the manual control in MagIC Net again: **Off**.

4.2 Connecting the separation column

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

NOTICE

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

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

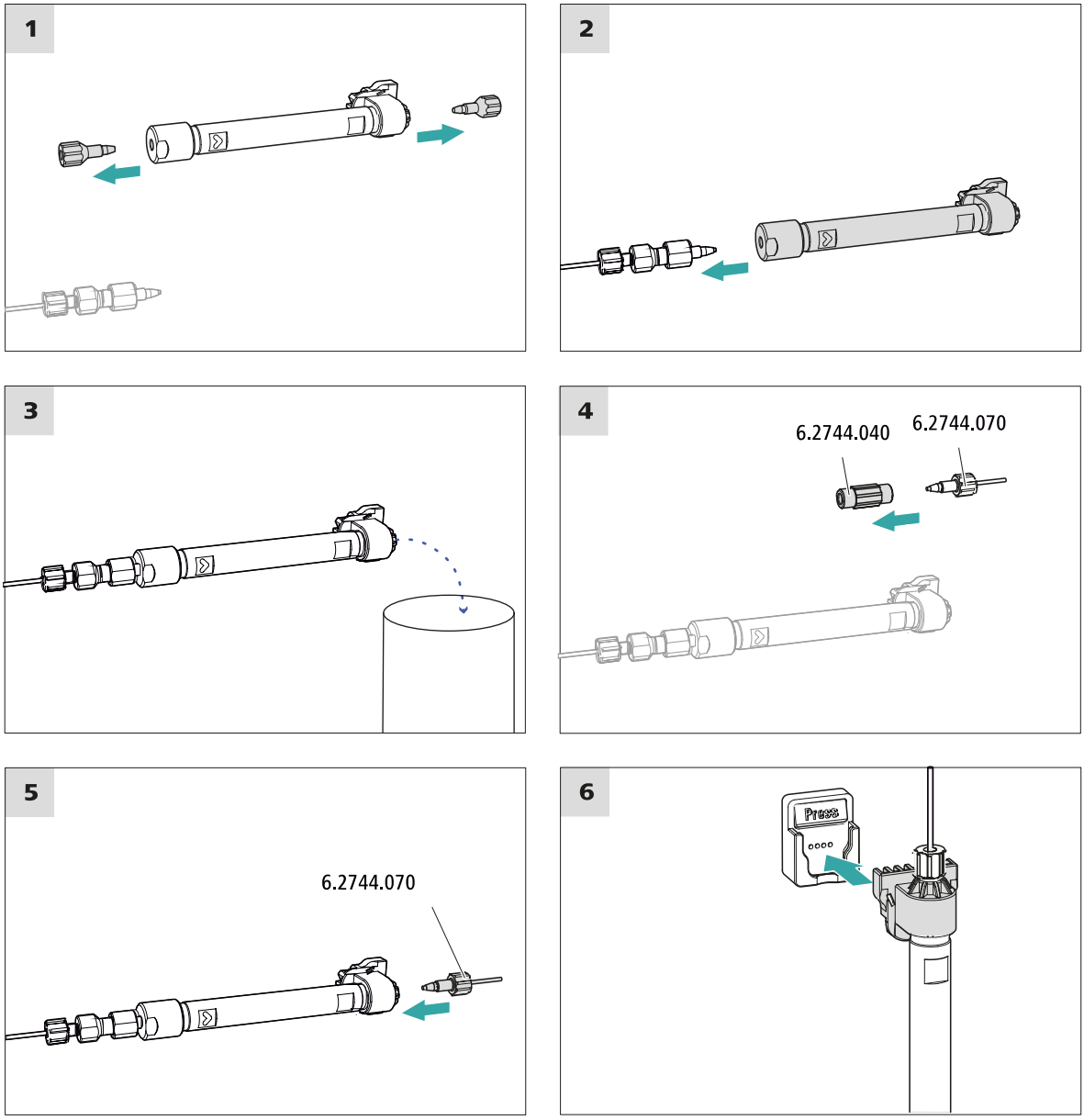
A test chromatogram accompanies every column. The column leaflet can be found online at *http://www.metrohm.com* with the corresponding article. Detailed information on special IC applications can be found in the corresponding **Application Bulletins** or **Application Notes**. You can find these online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

CAUTION

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

NOTICE

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



Connecting the separation column

1 Removing the stoppers

- Remove the stoppers from the separation column.

2 Installing the inlet of the separation column

CAUTION

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

There are 3 possibilities:

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

3 Rinsing the separation column

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

4 Removing the coupling

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

5 Installing the outlet of the separation column

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

6 Inserting the separation column

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

The separation column is now detected by MagIC Net.

4.3 Conditioning

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

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

NOTICE

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

Conditioning the system

1 Preparing the software

CAUTION

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

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

2 Preparing the instrument

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

3 Starting equilibration

- Start the equilibration in MagIC Net: **Workplace ► Run ► Equilibration ► Start HW**.
- Visually inspect whether all capillaries and their connections from the high-pressure pump to the detector are leak-tight. If eluent is leaking out anywhere, tighten the corresponding pressure screw further, or loosen the pressure screw, check the end of the capillary and shorten it using the capillary cutter if necessary and retighten the pressure screw.

4 Conditioning the system

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

The instrument is now ready for measuring samples.

5 Applications

5.1 Standard chromatogram

2-mm columns

Columns:
- Metrosep A Supp 10 - 50/2.0
- Metrosep A Supp 10 - 75/2.0
- Metrosep A Supp 10 - 100/2.0
- Metrosep A Supp 10 - 150/2.0
- Metrosep A Supp 10 - 250/2.0

Sample preparation: –

Detection: Conductivity

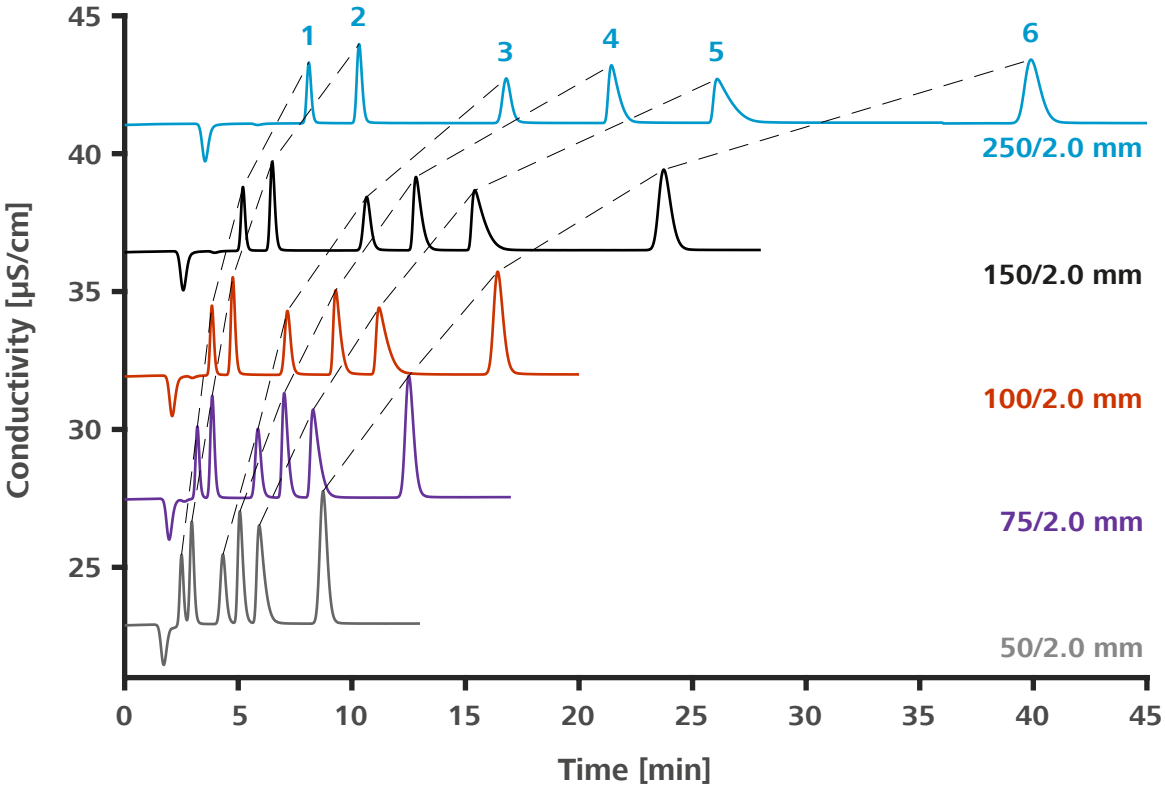
Suppression: Chemical suppression with MSM

Temperature: 45 °C

Loop: 10 µL

Flow rate: 0.25 mL/min

Eluent: 5.0 mmol/L $NaHCO_3$ and 5.0 mmol/L Na_2CO_3



	Metrosep A Supp 10 - XXX/2.0	mg/L
1	Chloride	5
2	Nitrite	5
3	Phosphate	10
4	Bromide	10
5	Nitrate	10
6	Sulfate	10

4-mm columns

Columns:
- Metrosep A Supp 10 - 50/4.0
- Metrosep A Supp 10 - 75/4.0
- Metrosep A Supp 10 - 100/4.0
- Metrosep A Supp 10 - 250/4.0

Sample preparation: –

Detection: Conductivity

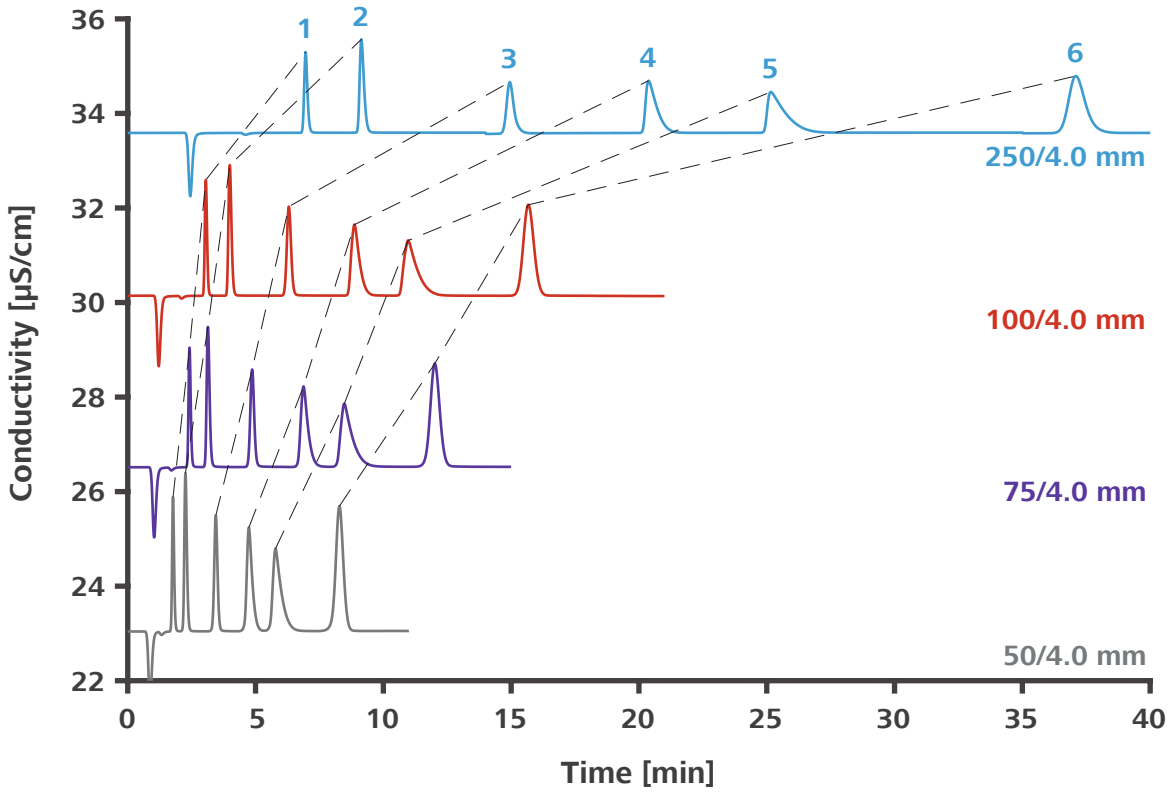
Suppression: Chemical suppression with MSM

Temperature: 45 °C

Loop: 20 µL

Flow rate: 1.0 mL/min

Eluent: 5.0 mmol/L $NaHCO_3$ and 5.0 mmol/L Na_2CO_3



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

5.2 Effects of temperature

Column: Metrosep A Supp 10 - 100/4.0

Sample preparation: –

Detection: Conductivity

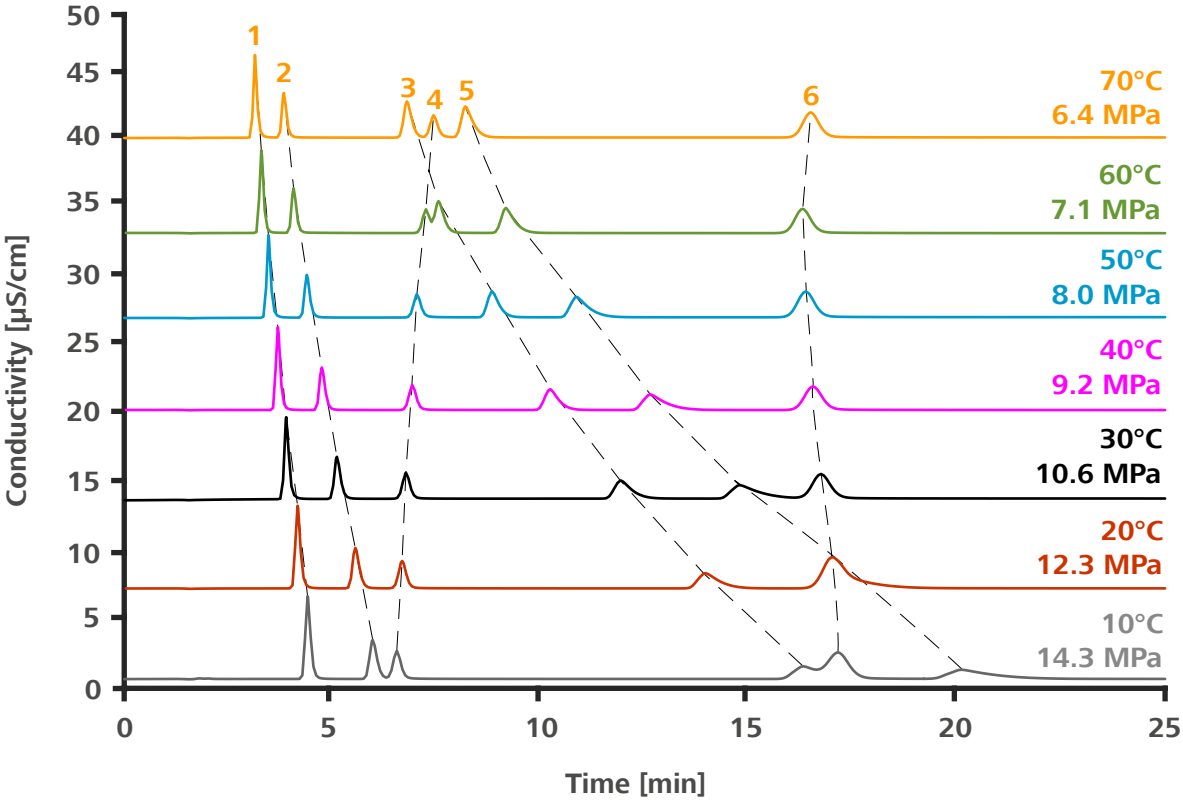
Suppression: Sequential suppression with MSM and MCS

Temperature: 10–70 °C

Loop: 20 µL

Flow rate: 1.0 mL/min

Eluent: 5.0 mmol/L $NaHCO_3$ and 5.0 mmol/L Na_2CO_3



	Metrosep A Supp 10 - 100/4.0	mg/L
1	Chloride	10
2	Nitrite	10

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

The Metrosep A Supp 10 can be used at temperatures from 10 to 70 °C. When the temperature increases, the retention times of the monovalent anions decrease. An increase in temperature particularly accelerates the polarizable ions bromide and nitrate. Higher temperatures significantly enhance the peak shapes of bromide and nitrate . The retention time of phosphate increases slightly when the temperature increases. The retention time of sulfate remains almost the same at different temperatures. At 20 °C, nitrate and sulfate coelute.

Increasing the temperature causes the column backpressure to decrease considerably. At 70 °C, the column backpressure is approx. 6.4 MPa. At 10 °C, the column backpressure is approx. 14.3 MPa. Due to the high backpressure, standard flow measurements at 10 °C are not possible on the long Metrosep A Supp 10 - 250/4.0 column.

With organic acids, only few shifts can be caused by the temperature (chromatograms not shown). Acetate, glycolate, formate, lactate, propionate and pyruvate elute either in the injection peak or are scarcely retained on the column. This behavior does not depend on the temperature. The retention times of the organic acids succinate, malate, adipate and malonate increase when the temperature increases. The retention times of tartrate and oxalate become slightly shorter with rising temperature.

5.3 Variation of the eluent flow rate

Column: Metrosep A Supp 10 - 100/4.0

Sample preparation: –

Detection: Conductivity

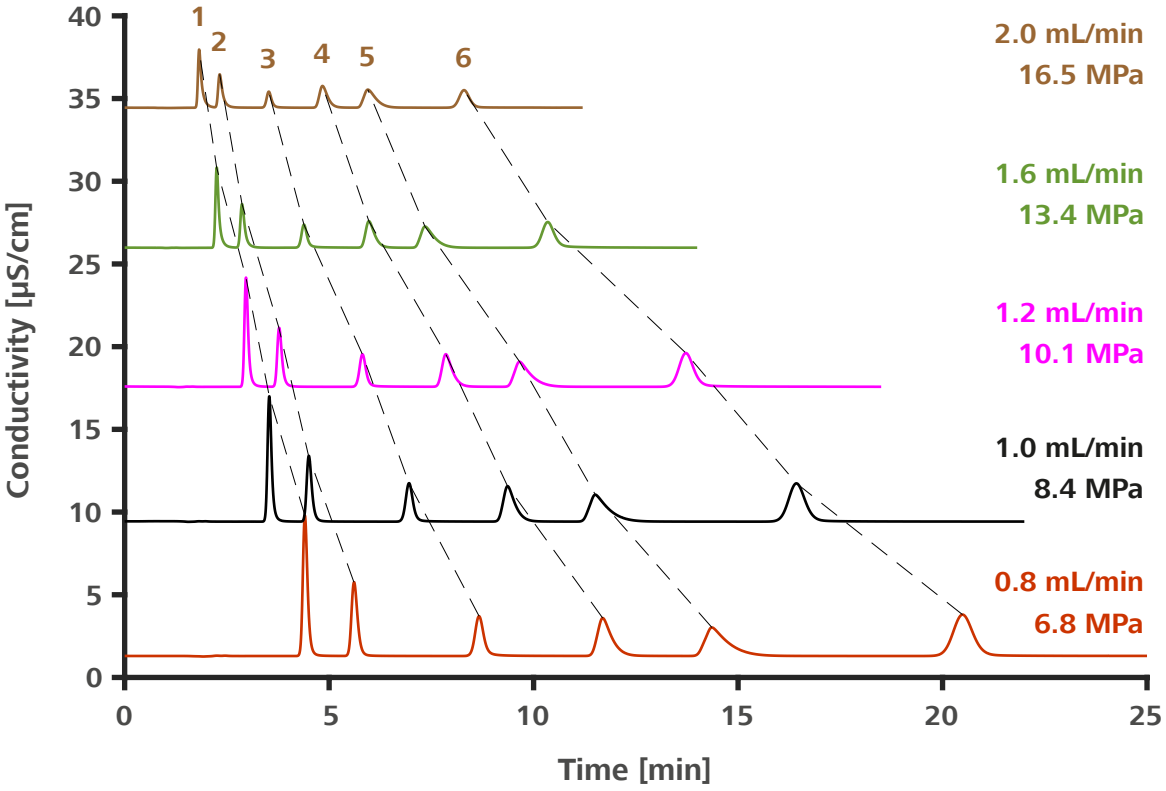
Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 20 µL

Flow rate: 0.8–2.0 mL/min

Eluent: 5.0 mmol/L $NaHCO_3$ and 5.0 mmol/L Na_2CO_3



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

The Metrosep A Supp 10 - 100/4.0 can be operated with a flow of up to 2.0 mL/min. Increasing the flow accelerates all ions equally. Sulfate elutes in less than 10 minutes at 2.0 mL/min. The pressure increases almost proportionally to the flow. The higher flow rate decreases the dwell time of the analytes in the detector. This leads to smaller peak areas. The long Metrosep A Supp 10 - 250/4.0 column can also be operated at a flow of up to 2.0 mL/min. At a high flow rate on the long column, the temperature must be increased. If not, there is 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 10 - 250/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 20 µL

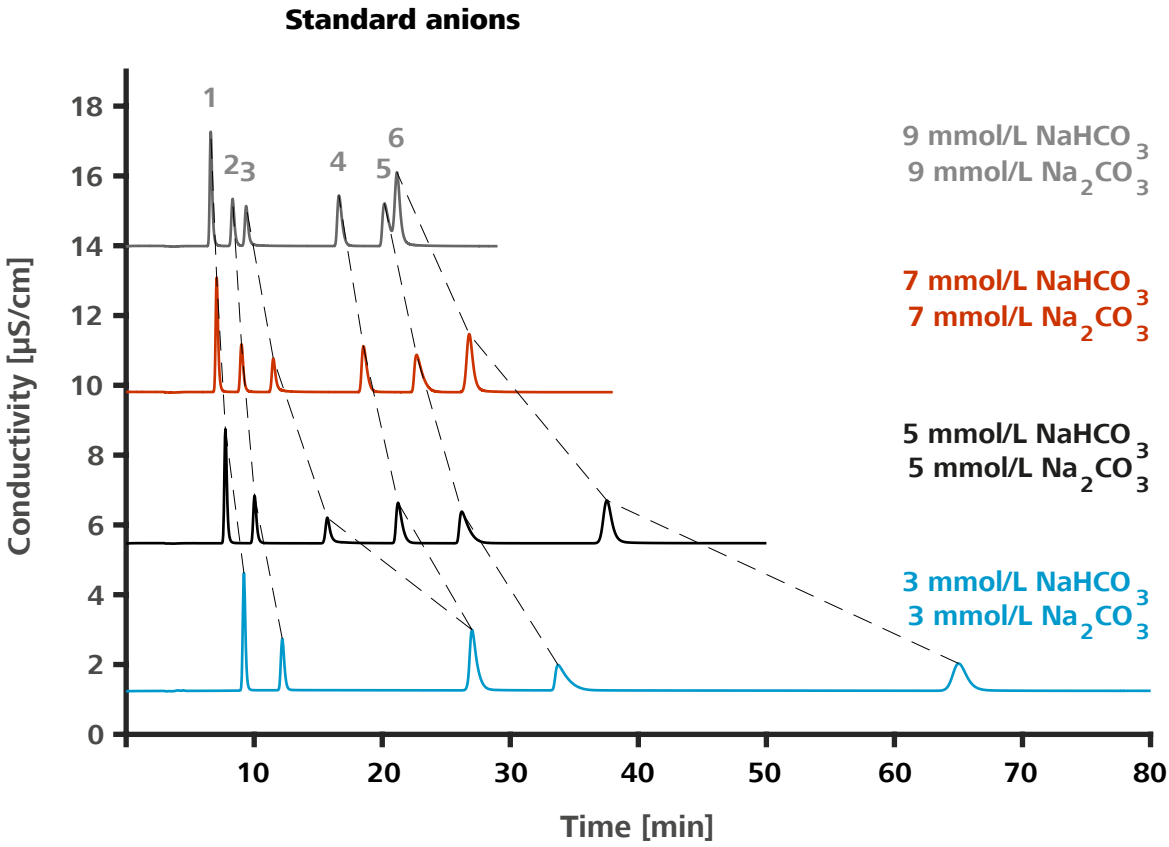
Flow rate: 1.0 mL/min

Eluent: A) 3.0 mmol/L $NaHCO_3$, 3.0 mmol/L Na_2CO_3

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

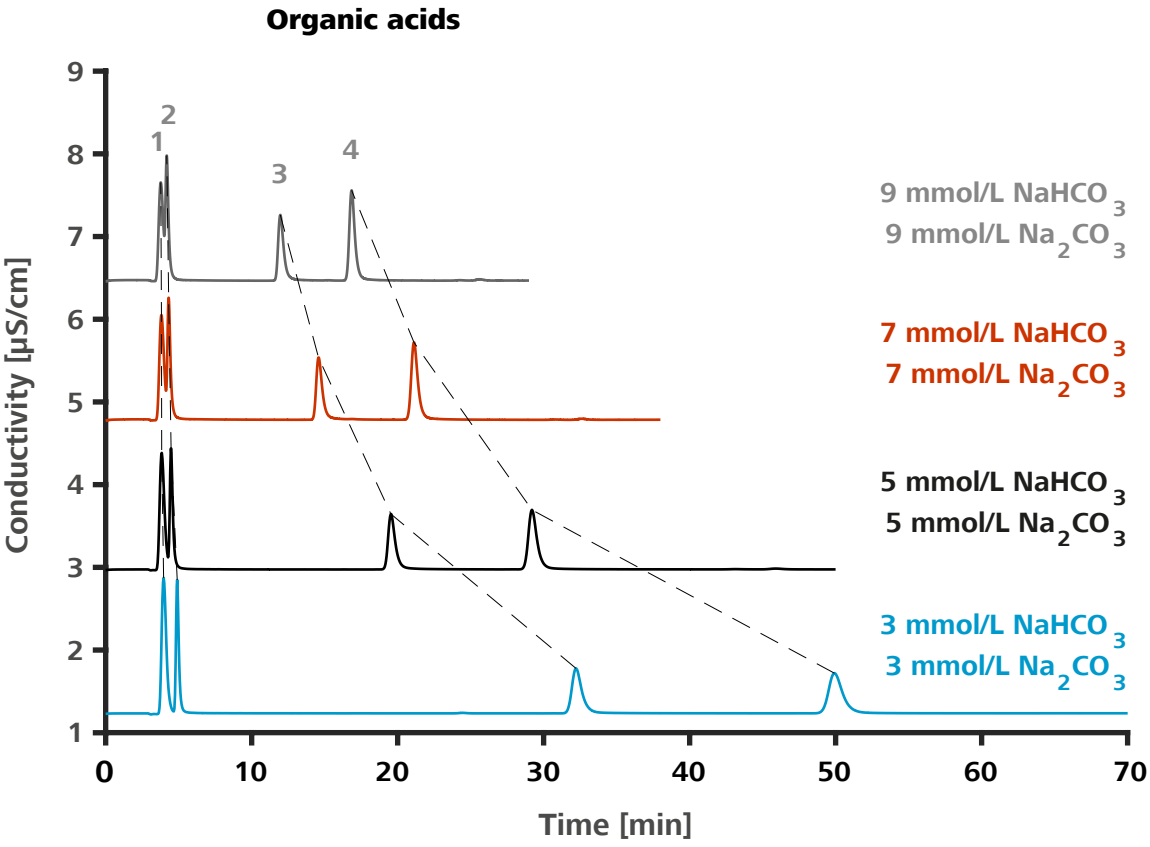
C) 7.0 mmol/L $NaHCO_3$, 7.0 mmol/L Na_2CO_3

D) 9.0 mmol/L $NaHCO_3$, 9.0 mmol/L Na_2CO_3

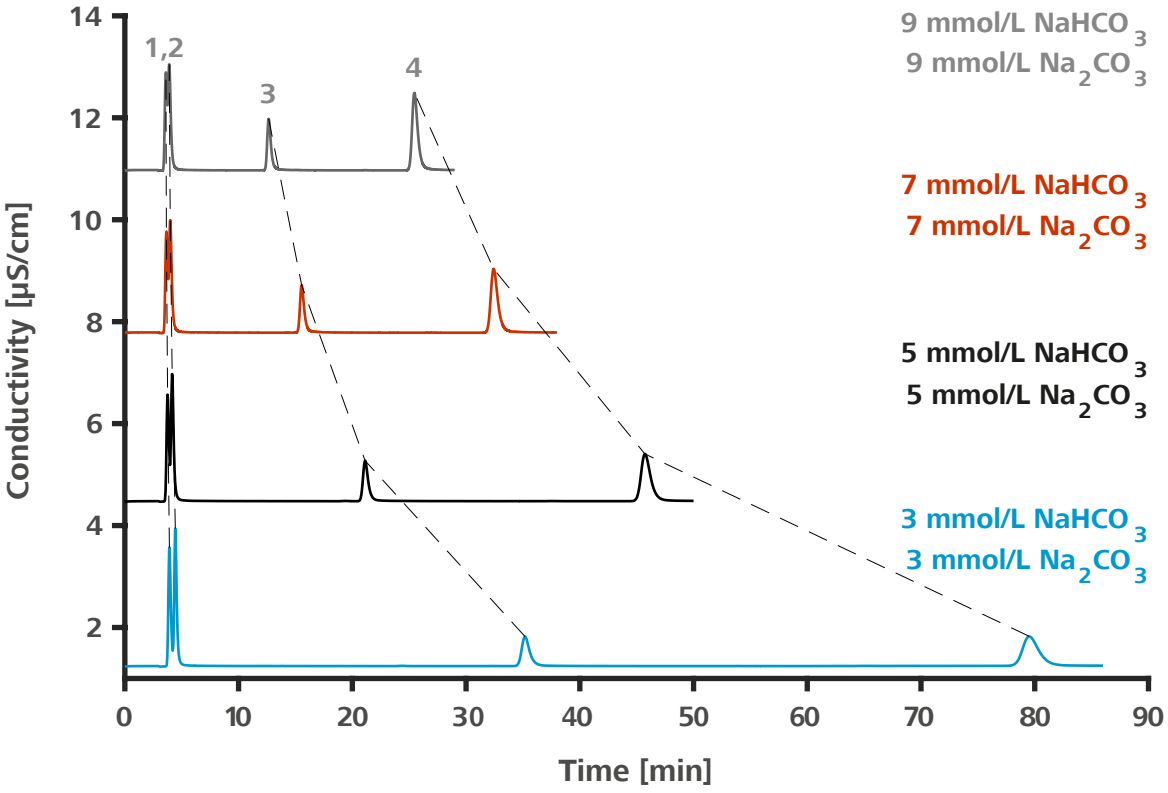


	Metrosep A Supp 10 - 250/4.0	mg/L
1	Chloride	10
2	Nitrite	10
3	Phosphate	10
4	Bromide	10
5	Nitrate	10
6	Sulfate	10

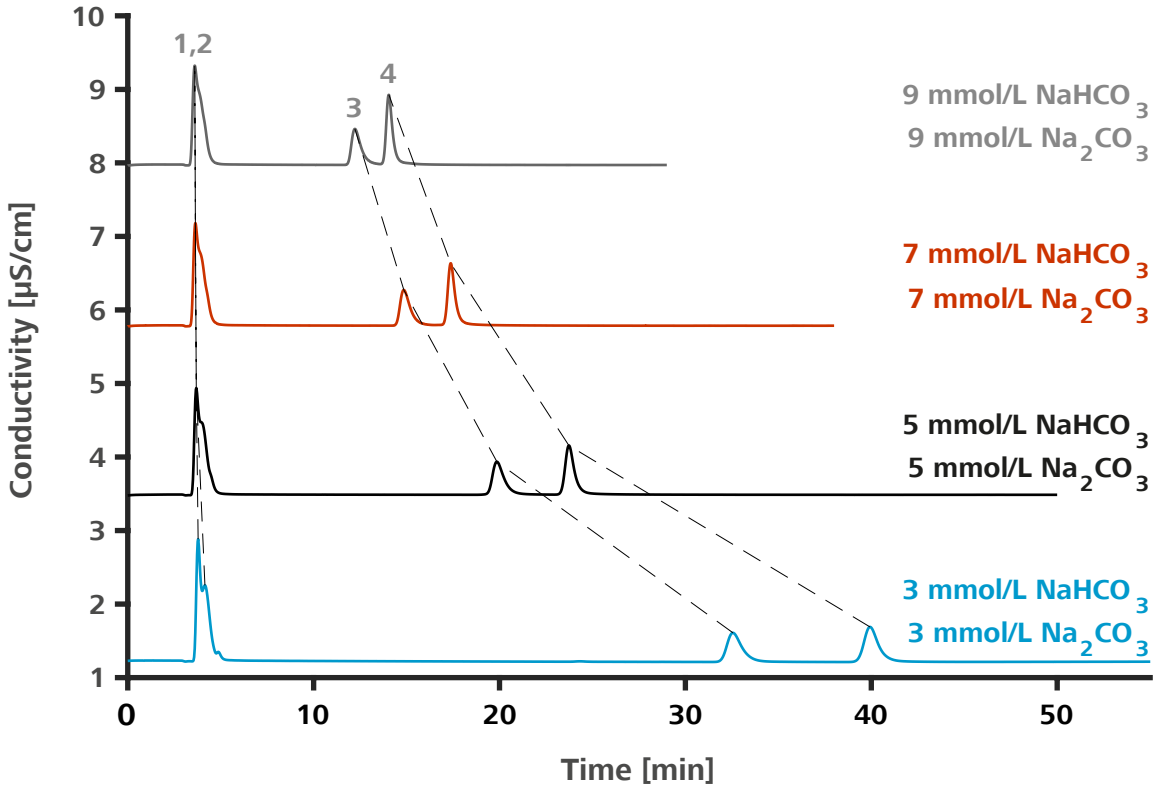
An increase in the eluent concentration accelerates all anions. The polyvalent anions phosphate and sulfate are accelerated faster than the monovalent anions. The stronger the eluent is, the sharper and higher the peaks are. In a weak eluent (3.0 mmol/L Na_2CO_3, 3.0 mmol/L $NaHCO_3$), phosphate and bromide coelute. In a strong eluent (9.0 mmol/L Na_2CO_3, 9.0 mmol/L $NaHCO_3$), nitrate and sulfate are not optimally separated.



	Metrosep A Supp 10 - 250/4.0	mg/L
1	Acetate	10
2	Pyruvate	10
3	Succinate	10
4	Tartrate	10



	Metrosep A Supp 10 - 250/4.0	mg/L
1	Glycolate	10
2	Formate	10
3	Malate	10
4	Oxalate	10



	Metrosep A Supp 10 - 250/4.0	mg/L
1	Lactate	10
2	Propionate	10
3	Adipate	10
4	Malonate	10

An increase of the eluent concentration accelerates organic acids. The peaks become narrower and higher.

5.4.2 $NaHCO_3$ variation at constant Na_2CO_3

Column: Metrosep A Supp 10 - 250/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 20 µL

Flow rate: 1.0 mL/min

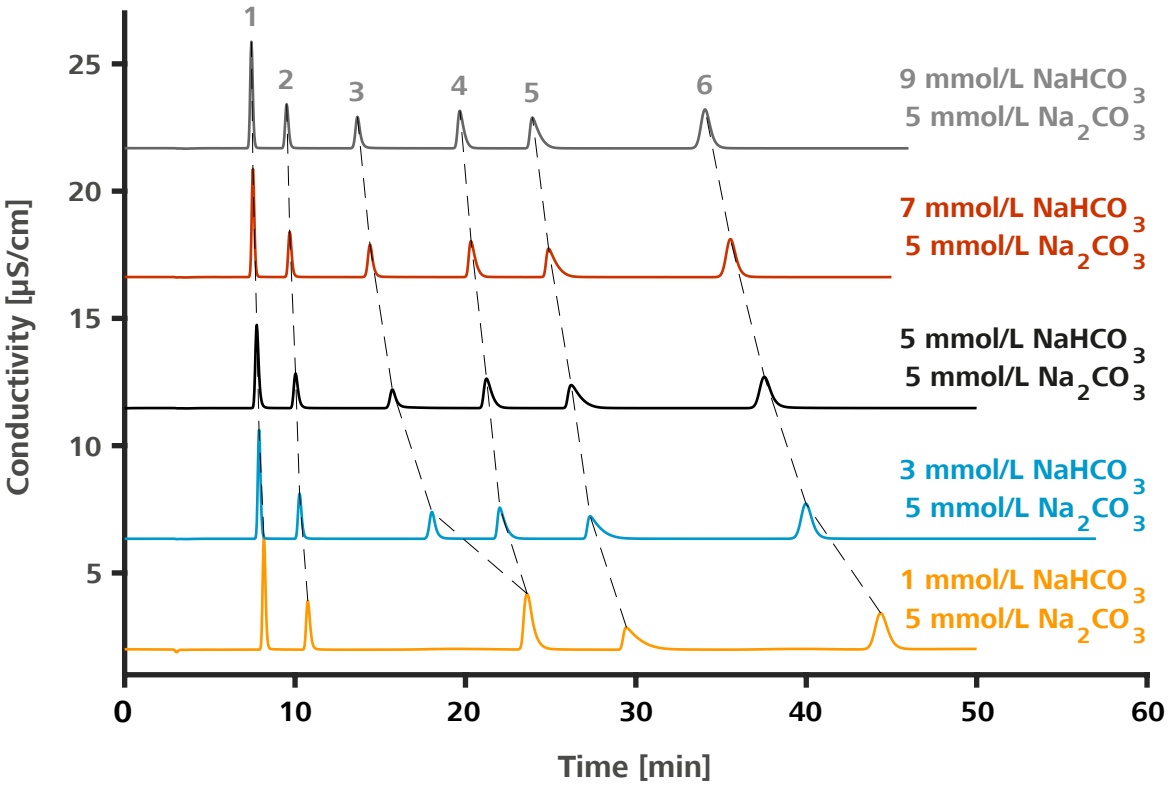
Eluent: A) 1.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3

B) 3.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3

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

D) 7.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3

E) 9.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3



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

Sodium hydrogen carbonate does not influence the eluent strength as much as sodium carbonate. Therefore, an increase in the content of sodium hydrogen carbonate only shortens the retention times of the anions slightly. Only the retention time of phosphate is shortened signifi-

cantly. This is due to the fact that the pH value of the eluent and thus the real charge of the phosphate ion changes considerably. In the tested range of sodium hydrogen carbonate, no significant change in peak height can be observed. In an eluent composition of 1.0 mmol/L $NaHCO_3$ and 5.0 mmol/L Na_2CO_3, bromide and phosphate coelute.

The organic acids acetate, glycolate, formate, lactate, propionate and pyruvate elute almost in the injection peak. They hardly change if the amount of sodium hydrogen carbonate in the eluent changes (chromatograms not shown). The organic acids succinate, malate, adipate, malonate, tartrate and oxalate are retained on the column. An increase in the content of sodium hydrogen carbonate in the eluent shortens their retention time.

5.4.3 Na_2CO_3 variation at constant $NaHCO_3$

Column: Metrosep A Supp 10 - 250/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 20 µL

Flow rate: 1.0 mL/min

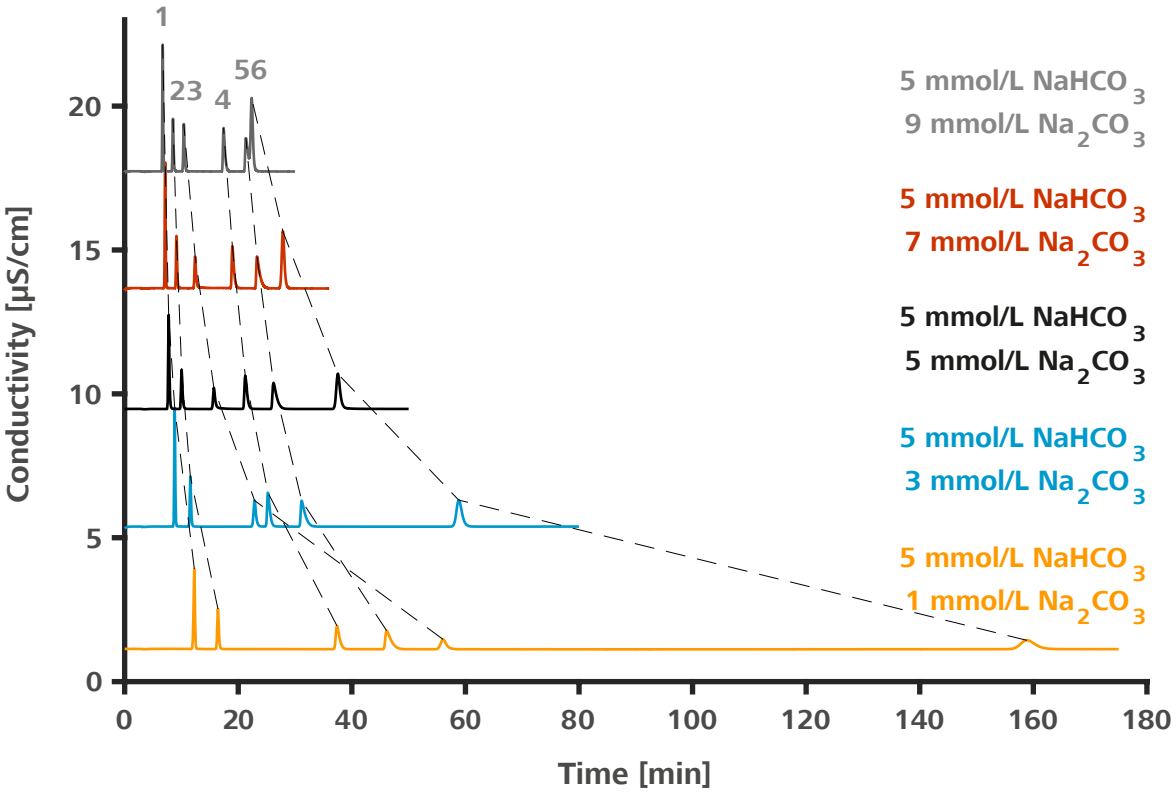
Eluent: A) 5.0 mmol/L $NaHCO_3$, 1.0 mmol/L Na_2CO_3

B) 5.0 mmol/L $NaHCO_3$, 3.0 mmol/L Na_2CO_3

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

D) 5.0 mmol/L $NaHCO_3$, 7.0 mmol/L Na_2CO_3

E) 5.0 mmol/L $NaHCO_3$, 9.0 mmol/L Na_2CO_3



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

Sodium carbonate has a big elution strength. Thus, the influence of the sodium carbonate content in the eluent is stronger than the influence of the sodium hydrogen carbonate content. An increase in the content of sodium carbonate shortens the retention times of all anions considerably. The polyvalent anions phosphate and sulfate are accelerated the most. In a weak eluent (5 mmol/L $NaHCO_3$ and 1 mmol/L Na_2CO_3), phosphate elutes after nitrate. However, in a strong eluent (5 mmol/L $NaHCO_3$ and 9 mmol/L Na_2CO_3), phosphate elutes before bromide. This is due to the fact that the pH value of the eluent and thus the real charge of the phosphate ion changes. In a strong eluent (5 mmol/L $NaHCO_3$ and 9 mmol/L Na_2CO_3), the baselines of nitrate and sulfate cannot be separated anymore. The acceleration of the anions due to the use of a stronger eluent causes higher peaks.

The organic acids acetate, glycolate, formate, lactate, propionate and pyruvate elute almost in the injection peak. They hardly change if the amount of sodium carbonate in the eluent changes (chromatograms not shown). The organic acids succinate, malate, adipate, malonate, tartrate and oxalate are retained on the column. An increase in the content of sodium carbonate in the eluent shortens their retention time. The influence of the amount of sodium carbonate is considerably stronger than the influence of the amount of sodium hydrogen carbonate.

5.5 Variation with organic modifier

5.5.1 Variation of the acetone concentration

Column: Metrosep A Supp 10 - 100/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 20 µL

Flow rate: 1.0 mL/min

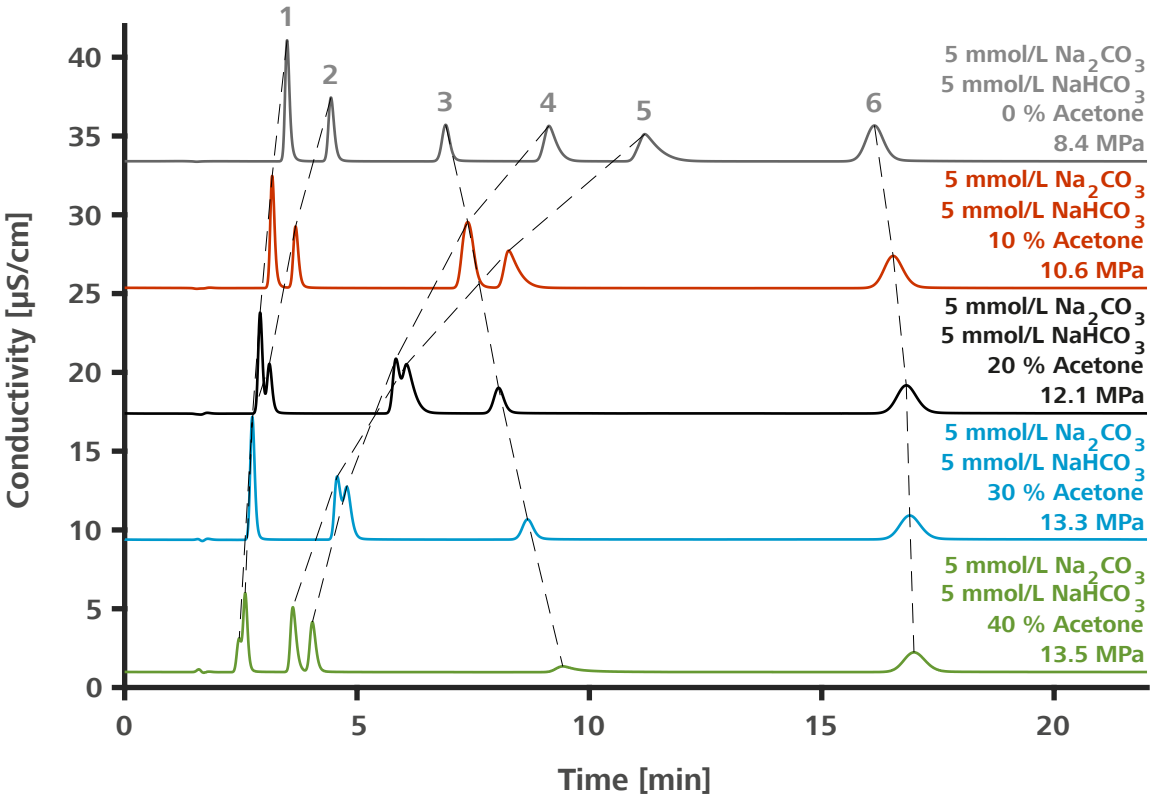
Eluent: A) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 0% acetone

B) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 10% acetone

C) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 20% acetone

D) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 30% acetone

E) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 40% acetone



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

In samples with an organic matrix, an organic modifier can be added to the eluent. Adding acetone to the eluent changes the order of elution and the selectivity of the standard anions. An increase of the acetone content in the eluent accelerates the elution of chloride, nitrite, bromide and nitrate and retards phosphate. At 10% acetone, bromide and phosphate coelute. The influence of acetone on sulfate is negligible. For nitrite and nitrate the acceleration is pronounced. Therefore, bromide and nitrate switch their elution order at approx. 25% acetone in the eluent. At approx. 35% acetone in the eluent, chloride and nitrite switch elution order. When adding acetone to the eluent, the peak shapes and thus the peak heights deteriorate.

The column backpressure increases with a higher acetone content in the eluent. This is due to the increased viscosity of the eluent mixture. At

approx. 40% acetone in the eluent, viscosity and backpressure are the highest. At a higher acetone content, the backpressure decreases again.

Adding acetone to the eluent accelerates all organic acids (chromatograms not shown). This results from the better solubility of the organic acids in the mobile phase. This effect is especially pronounced for adipate.

5.5.2 Variation of the methanol concentration

Column: Metrosep A Supp 10 - 100/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 20 µL

Flow rate: 1.0 mL/min

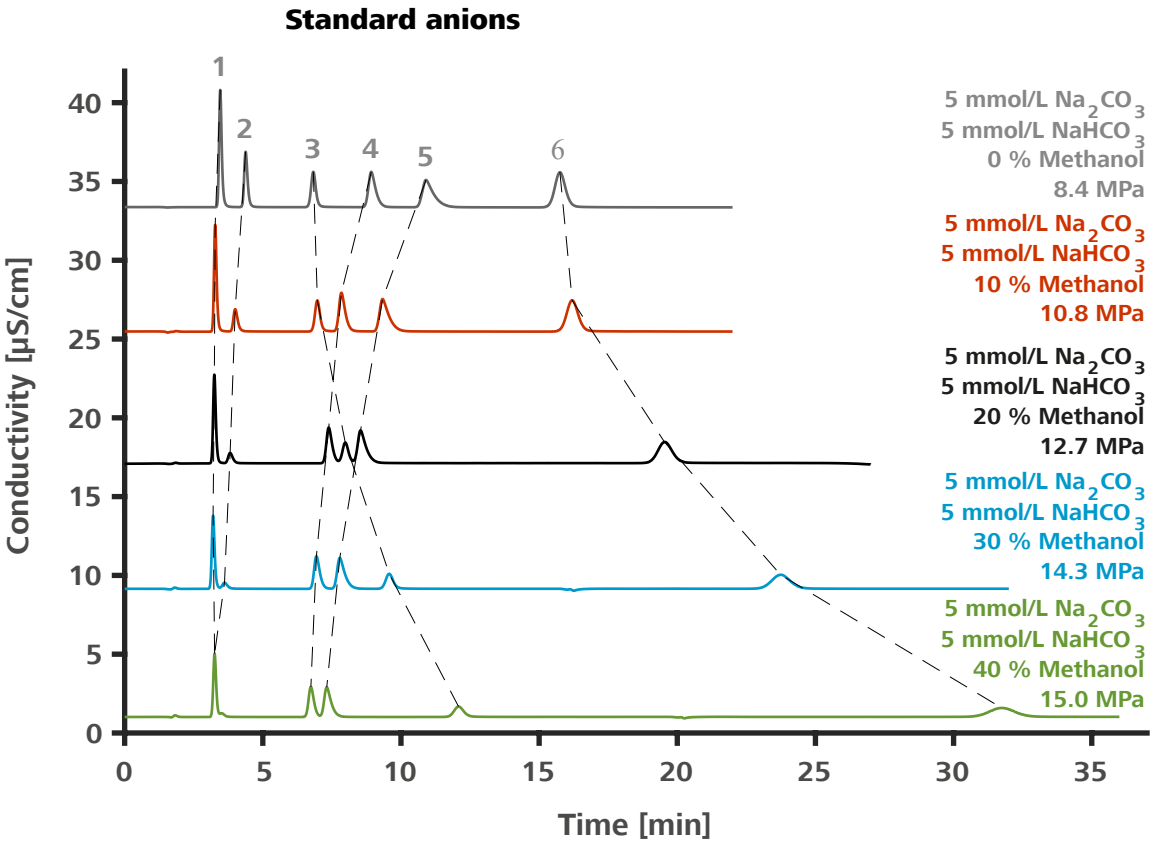
Eluent: A) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 0% methanol

B) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 10% methanol

C) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 20% methanol

D) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 30% methanol

E) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 40% methanol



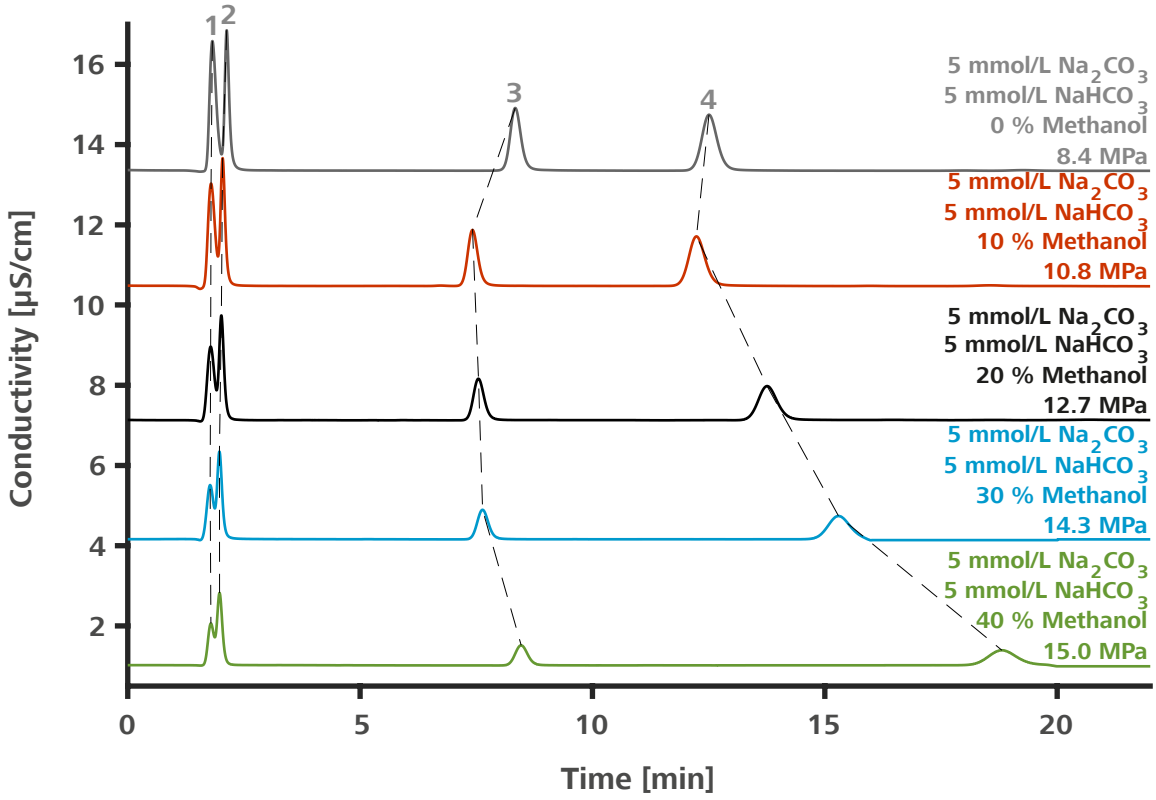
	Metrosep A Supp 10 - 100/4.0	mg/L
1	Chloride	10
2	Nitrite	10
3	Phosphate	10
4	Bromide	10
5	Nitrate	10
6	Sulfate	10

Instead of acetone, methanol can also be used as organic modifier. Similarly to the addition of acetone, the retention times of chloride, nitrite, bromide and nitrate are shorter when adding methanol. With increasing methanol content, the retention times of phosphate and sulfate are prolonged. As a result, the order of elution changes. At 20% methanol in the eluent, phosphate elutes between bromide and nitrate. At 30% methanol, phosphate elutes after nitrate. At 40% methanol in the eluent, nitrite coelutes with chloride. As methanol content increases, the peak areas and peak heights of nitrite and phosphate are decreased.

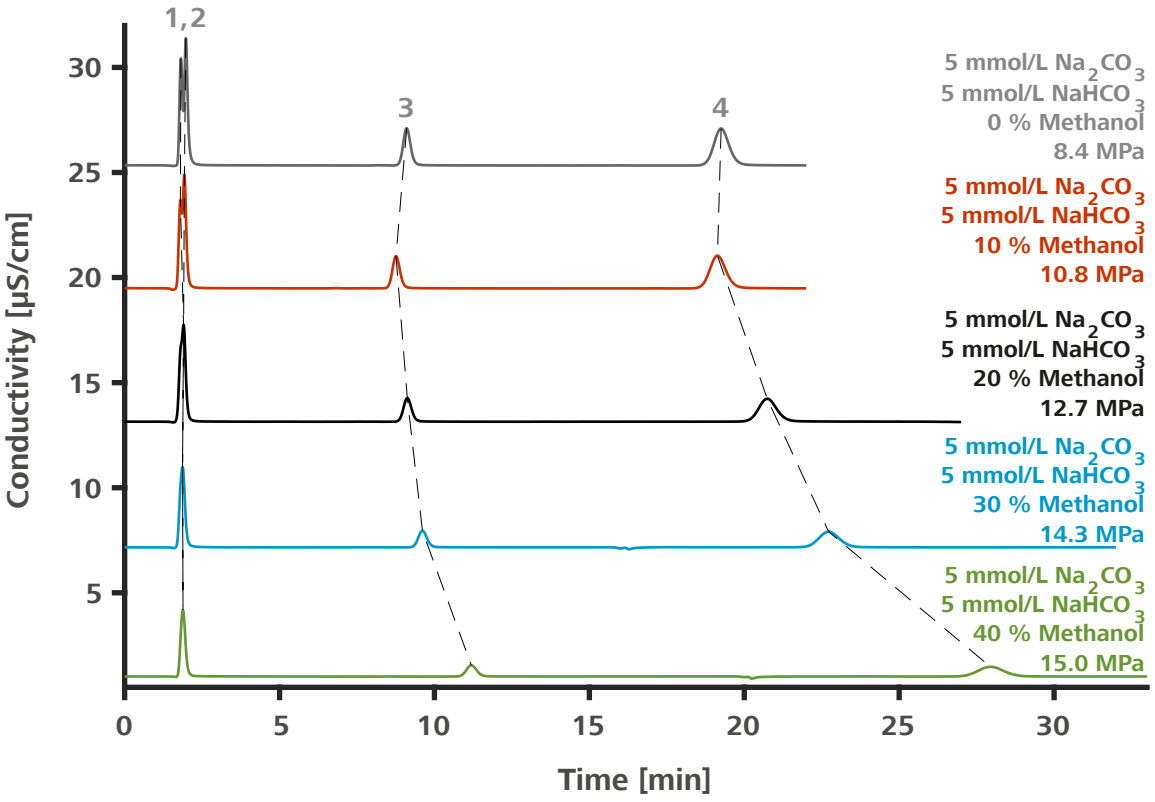
Methanol has an even bigger influence on the eluent viscosity than acetone. The column backpressure increases significantly with the addition of

methanol. At a methanol content of over 50%, the column backpressure decreases again.

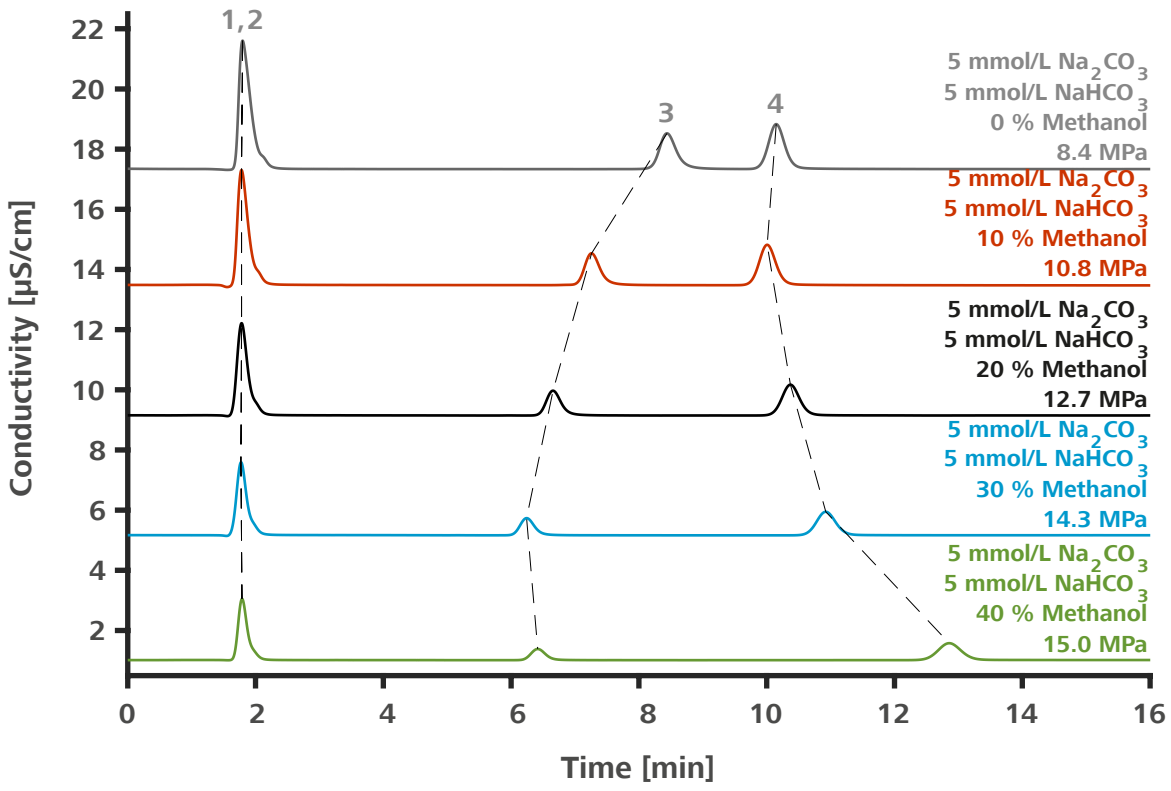
Organic acids



	Metrosep A Supp 10 - 100/4.0	mg/L
1	Acetate	10
2	Pyruvate	10
3	Succinate	10
4	Tartrate	10



	Metrosep A Supp 10 - 100/4.0	mg/L
1	Glycolate	10
2	Formate	10
3	Malate	10
4	Oxalate	10



	Metrosep A Supp 10 - 100/4.0	mg/L
1	Lactate	10
2	Propionate	10
3	Adipate	10
4	Malonate	10

The influence of methanol on the retention times of the organic acids differs depending on the acid. By adding 10% methanol to the eluent, the retention time of all organic acids is shortened. If the methanol content is increased further, this trend continues for adipate. The retention times of the other organic acids increase again with higher methanol content.

5.5.3 Variation of the acetonitrile concentration

Column: Metrosep A Supp 10 - 100/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 45 °C

Loop: 20 µL

Flow rate: 1.0 mL/min

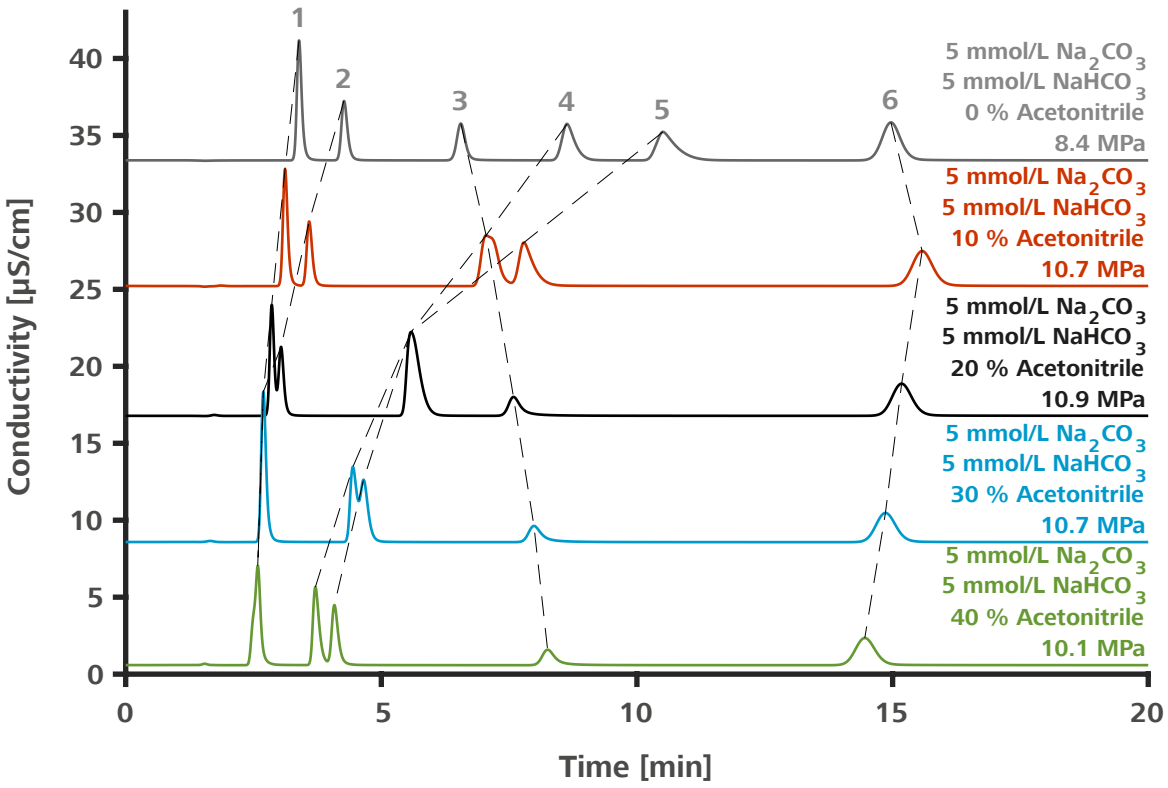
Eluent: A) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 0% acetonitrile

B) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 10% acetonitrile

C) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 20% acetonitrile

D) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 30% acetonitrile

E) 5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 40% acetonitrile



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

The influence of the addition of acetonitrile to the eluent is comparable to the influence of acetone. At 10% acetonitrile, phosphate and bromide

coelute. At 20% acetonitrile, nitrate and bromide switch their elution order, as nitrate is accelerated more than bromide. At an addition of 30% acetonitrile or higher to the eluent, chloride and nitrite coelute.

Acetontrile as organic modifier generates a smaller rise in pressure than acetone and methanol. At approx. 20% acetonitrile, the maximum pressure is reached.

Adding acetonitrile to the eluent accelerates the organic acids (chromatograms not shown). As with acetone, the acceleration effect is most pronounced in adipate.

5.6 Determination of standard anions and organic acids in wine samples

Column: Metrosep A Supp 5 - 250/4.0 with Metrosep A Supp 10 - 50/4.0 as guard column

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM and MCS

Temperature: 50 °C

Loop: 20 µL

Flow rate: 0.7 mL/min

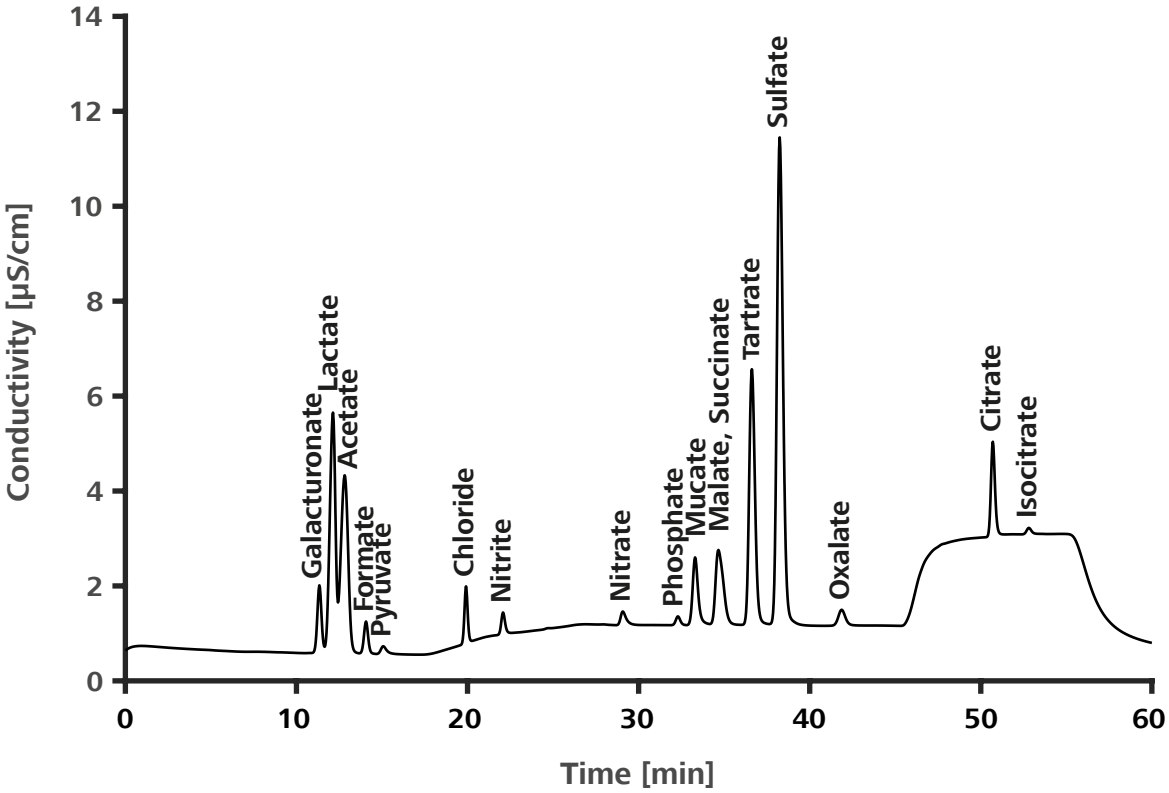
Eluent: Dose-in gradient

A) 0.2 mmol/L NaOH, 0.64 mmol/L Na_2CO_3

B) 20 mmol/L NaOH, 64 mmol/L Na_2CO_3

Gradient profile:

- 0–10 min: 100% A, 0% B
- 10–20 min: 100–90% A, 0–10% B
- 20–40 min: 90% A, 10% B
- 40–50 min: 10% A, 90% B
- 50–60 min: 100% A, 0% B



	Metrosep A Supp 5 - 250/4.0 and Metrosep A Supp 10 - 50/4.0	mg/L
1	Galacturonate	10
2	Lactate	20
3	Acetate	10
4	Formate	1
5	Pyruvate	1
6	Chloride	1
7	Nitrite	1
8	Nitrate	1
9	Phosphate	1
10	Mucate	10
11	Malate + Succinate	5
12	Tartrate	20
13	Sulfate	20
14	Oxalate	1
15	Citrate	10
16	Isocitrate	1

5.7 Determination of phosphate in Coca-Cola

Column: Metrosep A Supp 10 - 75/4.0

Sample preparation: –

Detection: Conductivity

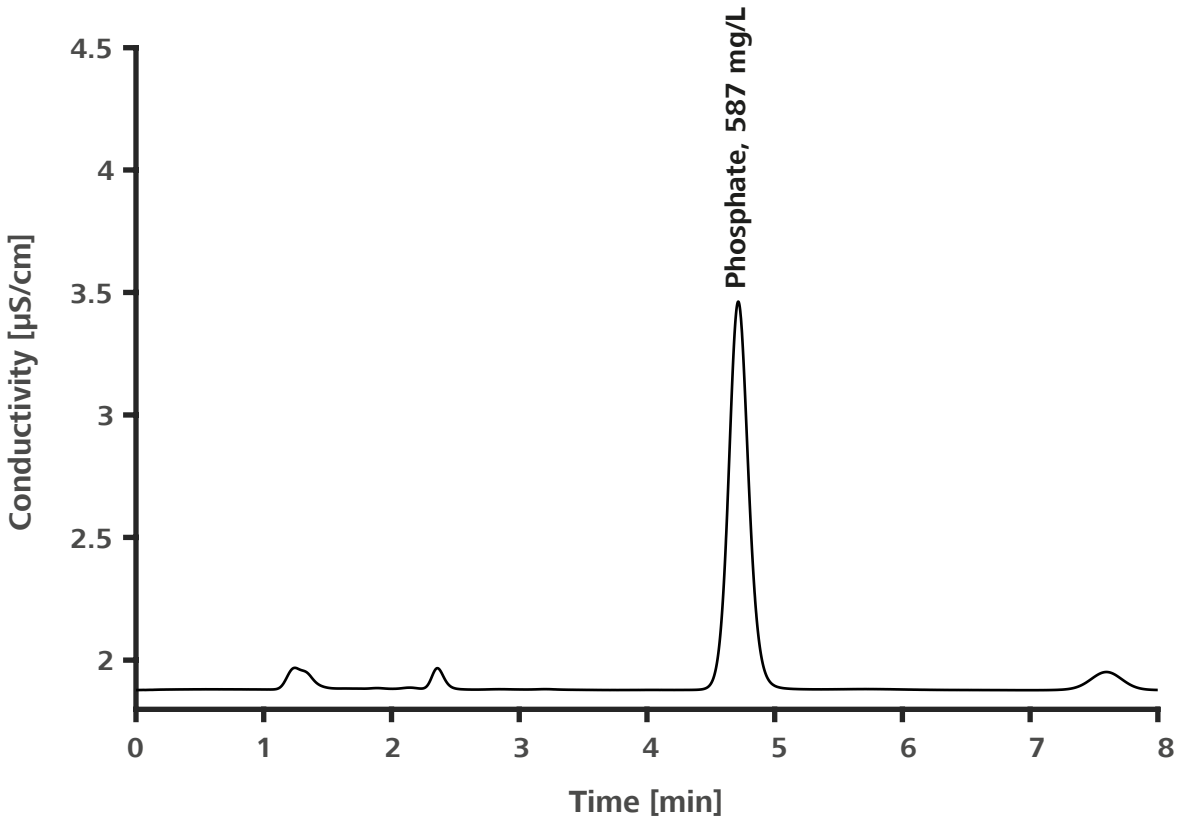
Suppression: Sequential suppression with MSM and MCS

Temperature: 30 °C

Loop: 0.25 µL

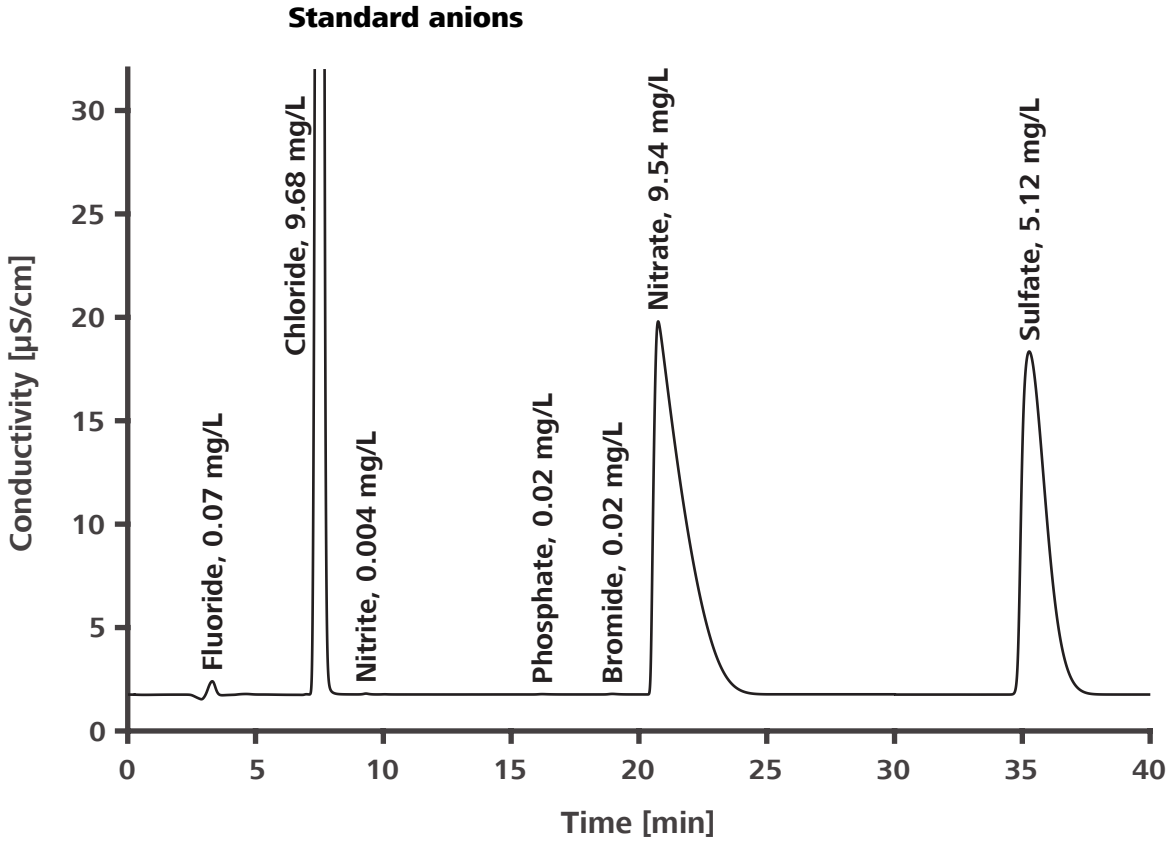
Flow rate: 1.2 mL/min

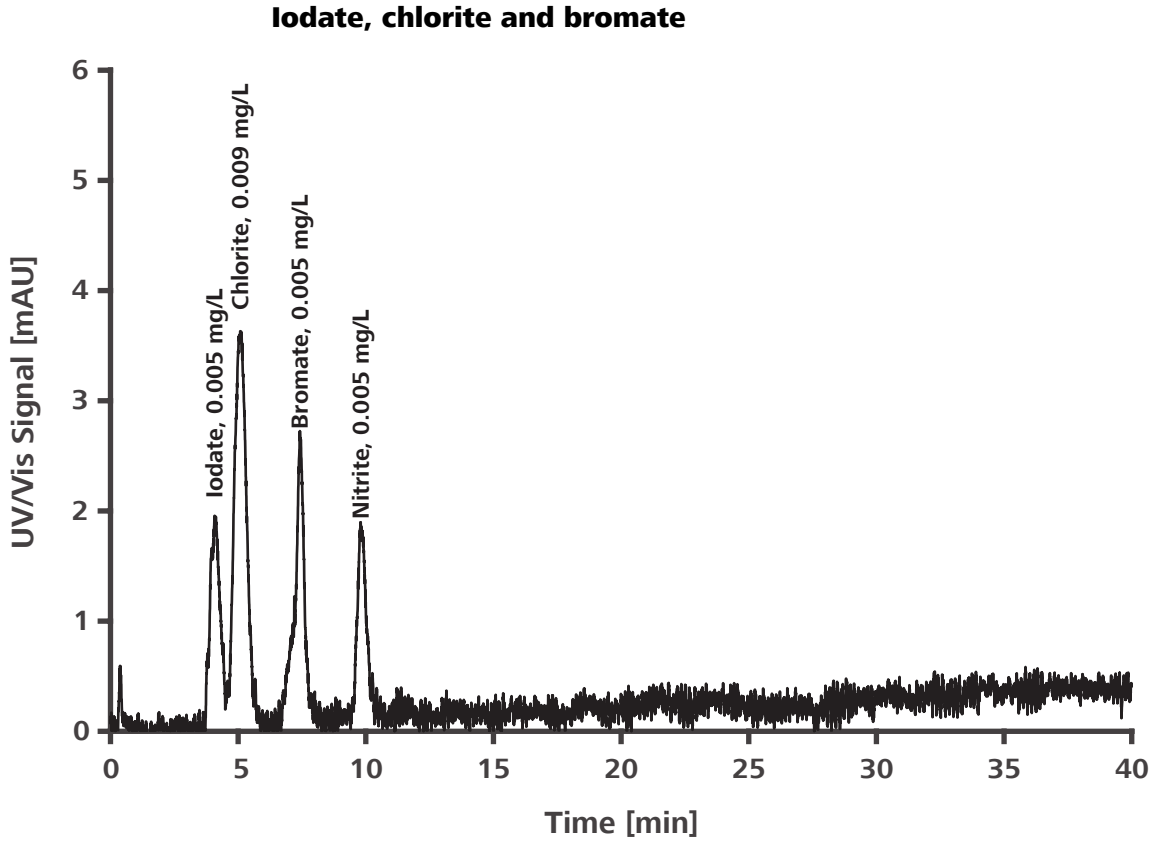
Eluent: 0.4 mmol/L $NaHCO_3$, 8.0 mmol/L Na_2CO_3



5.8 Determination of standard anions and iodate, chlorite and bromate in tap water

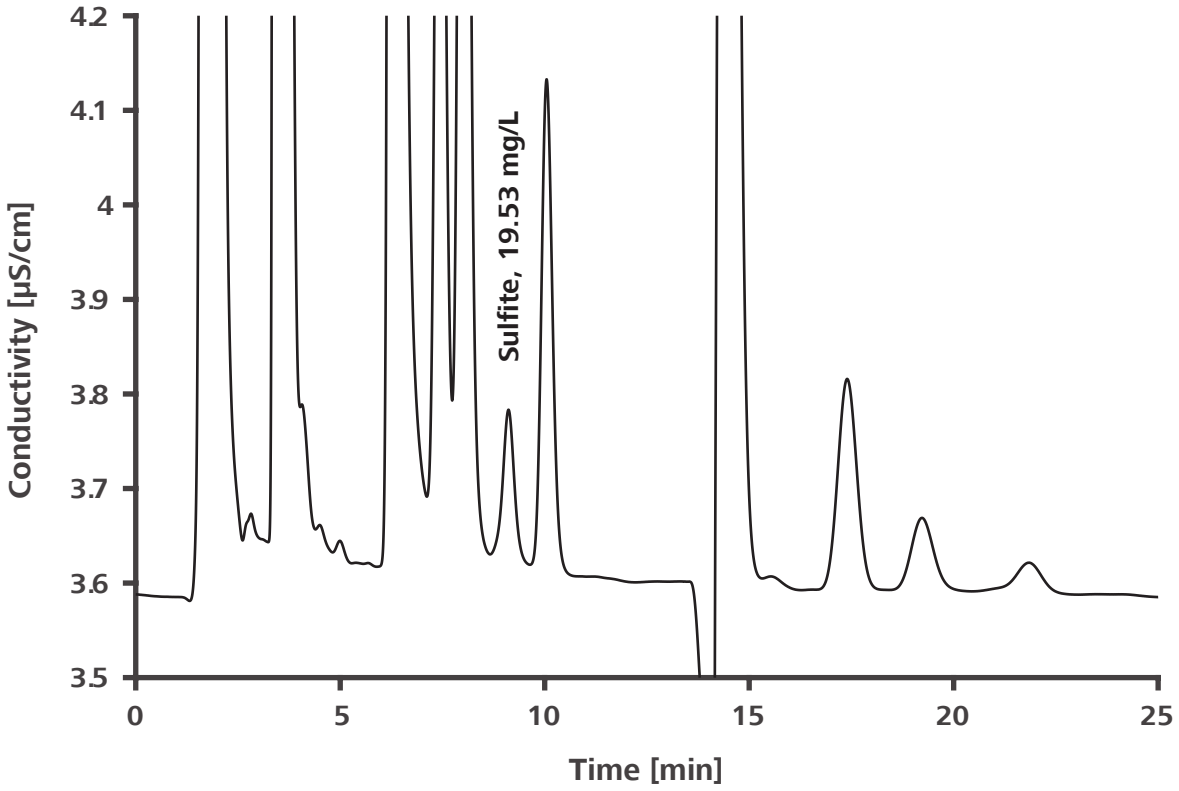
Column:	Metrosep A Supp 10 - 250/4.0
Sample preparation:	–
Detection:	Conductivity and UV/VIS after PCR (352 nm)
Suppression:	Sequential suppression with MSM and MCS
Temperature:	45 °C
Loop:	550 µL
Flow rate:	1.0 mL/min
Eluent:	5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3
Post-column derivatization reagent:	1) 0.2 mmol/L ammonium molybdate in 1 mol/L H_2SO_4 2) 40 g/L KI
Post-column derivatization temperature:	30 °C


Standard anions
Conductivity [µS/cm]
Time [min]
Fluoride, 0.07 mg/L
Chloride, 9.68 mg/L
Nitrite, 0.004 mg/L
Phosphate, 0.02 mg/L
Bromide, 0.02 mg/L
Nitrate, 9.54 mg/L
Sulfate, 5.12 mg/L
0
5
10
15
20
25
30
35
40



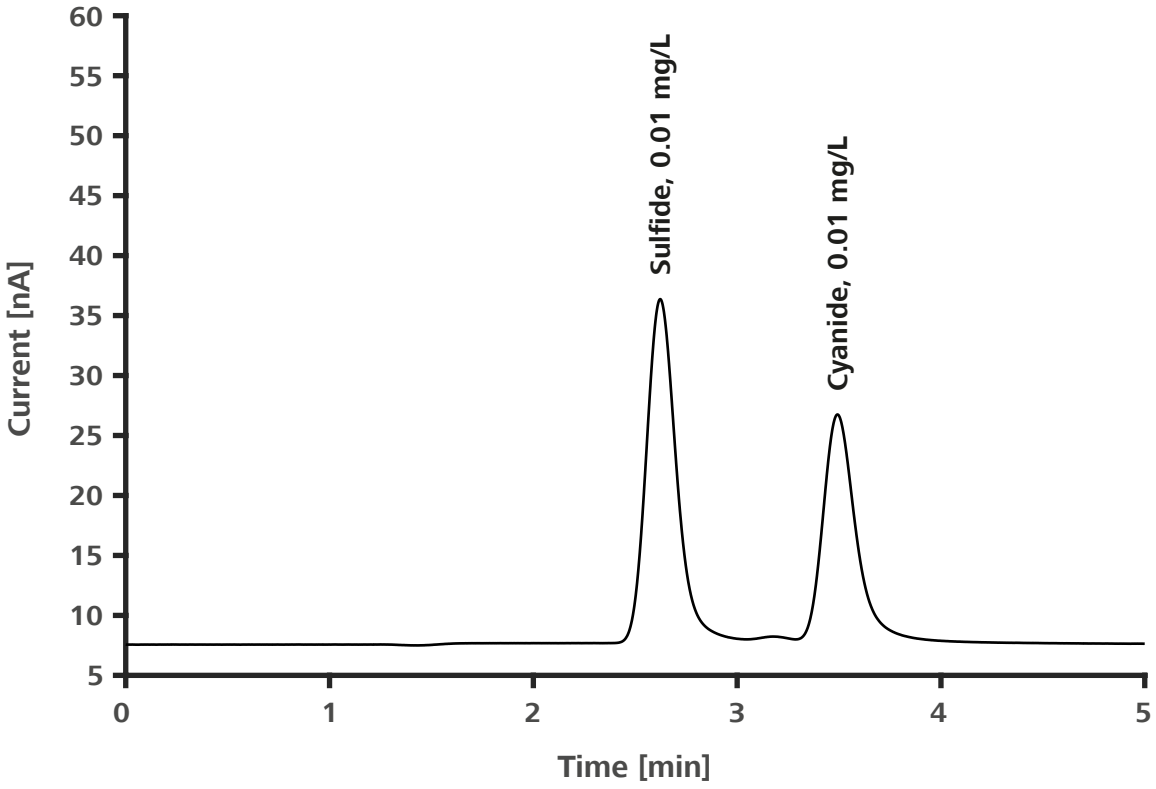
5.9 Determination of sulfite in beer

Column:	Metrosep A Supp 10 - 100/4.0
Sample preparation:	–
Detection:	Conductivity
Suppression:	Sequential suppression with MSM and MCS
Temperature:	35 °C
Loop:	20 µL
Flow rate:	1.0 mL/min
Eluent:	5.0 mmol/L $NaHCO_3$, 5.0 mmol/L Na_2CO_3, 35 µmol/L $HClO_4$



5.10 Determination of cyanide and sulfide in beverages

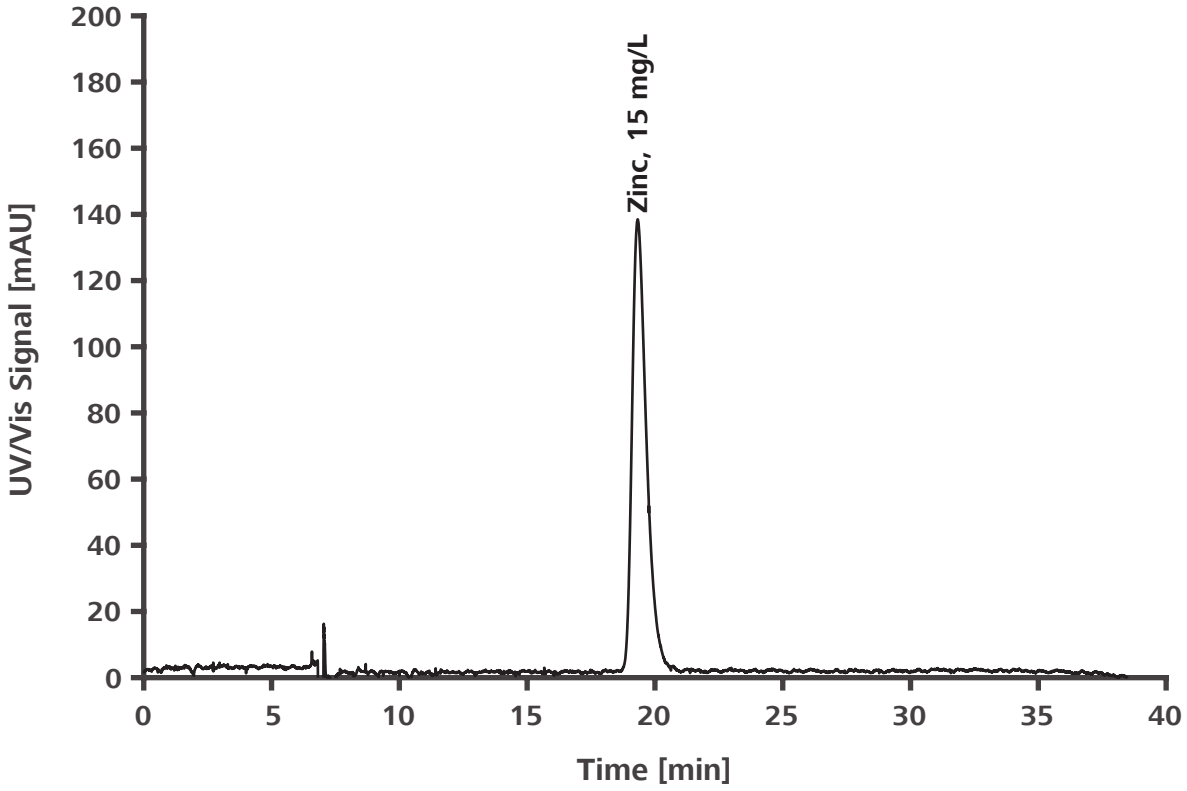
Column:	Metrosep A Supp 10 - 100/2.0
Sample preparation:	–
Detection:	Pulsed amperometric detection, DC mode
Suppression:	–
Temperature:	35 °C
Loop:	20 µL
Flow rate:	0.25 mL/min
Eluent:	100 mmol/L NaOH, 0.007 mmol/L EDTA



5.11 Determination of zinc in cosmetics in accordance with USP General Chapter <591>

Column:	Metrosep A Supp 10 - 250/4.0
Sample preparation:	–
Detection:	UV/VIS after PCR (530 nm)
Suppression:	–
Temperature:	30 °C
Loop:	10 µL
Flow rate:	1.2 mL/min
Eluent:	7.0 mmol/L dipicolinic acid , 66.0 mmol/L NaOH, 5.6 mmol/L Na_2SO_4, 74 mmol/L formic acid (pH 4.2)
Post-column derivatization reagent:	0.5 mmol/L 4-(2-pyridylazo)resorcinol monosodium salt hydrate, 1.0 mol/L methylaminoethanol, 0.5 mol/L ammonium hydroxide, 0.3 mol/L $NaHCO_3$

Post-column derivatization reagent flow rate: 0.6 mL/min



5.12 Determination of iron and transition metals

Column: Metrosep A Supp 10 - 250/2.0

Sample preparation: –

Detection: UV/VIS after PCR (510 nm)

Suppression: –

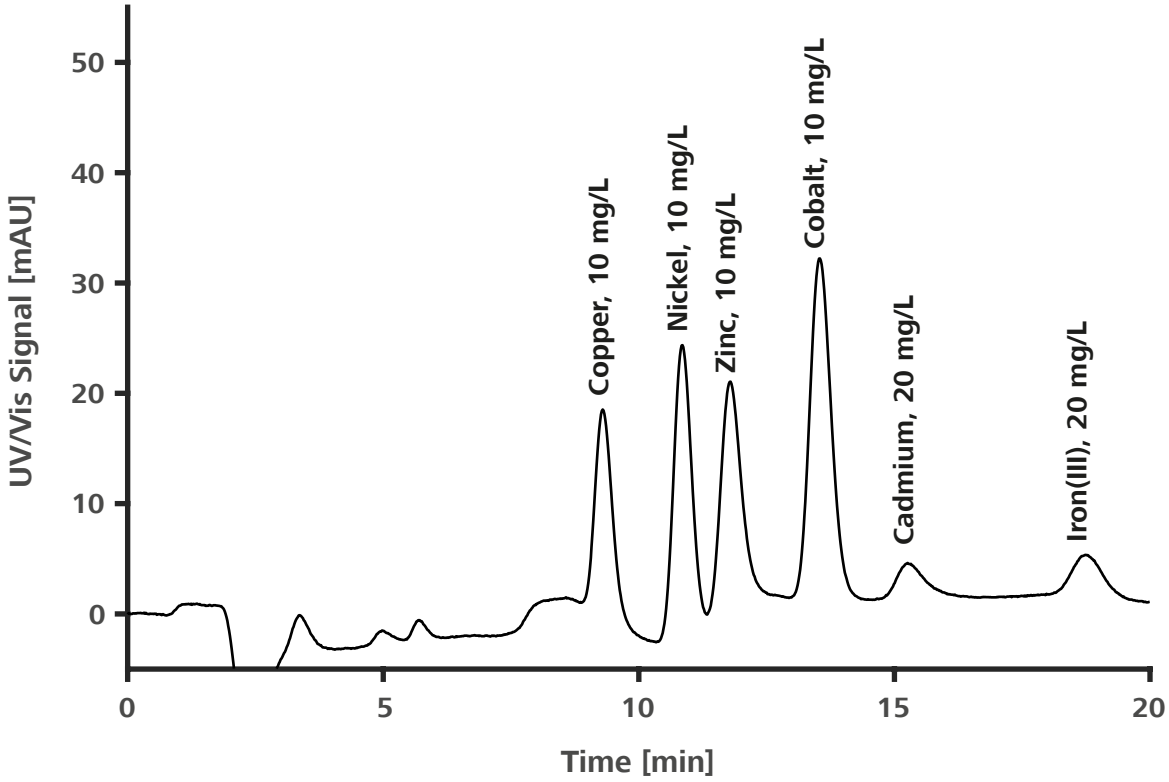
Temperature: 55 °C

Loop: 4000 µL (preconcentration)

Flow rate: 0.3 mL/min

Eluent: 3.0 mmol/L dipicolinic acid, 10.0 mmol/L Na_2SO_4, 66.0 mmol/L NaOH, 80 mmol/L formic acid (pH 4.3)

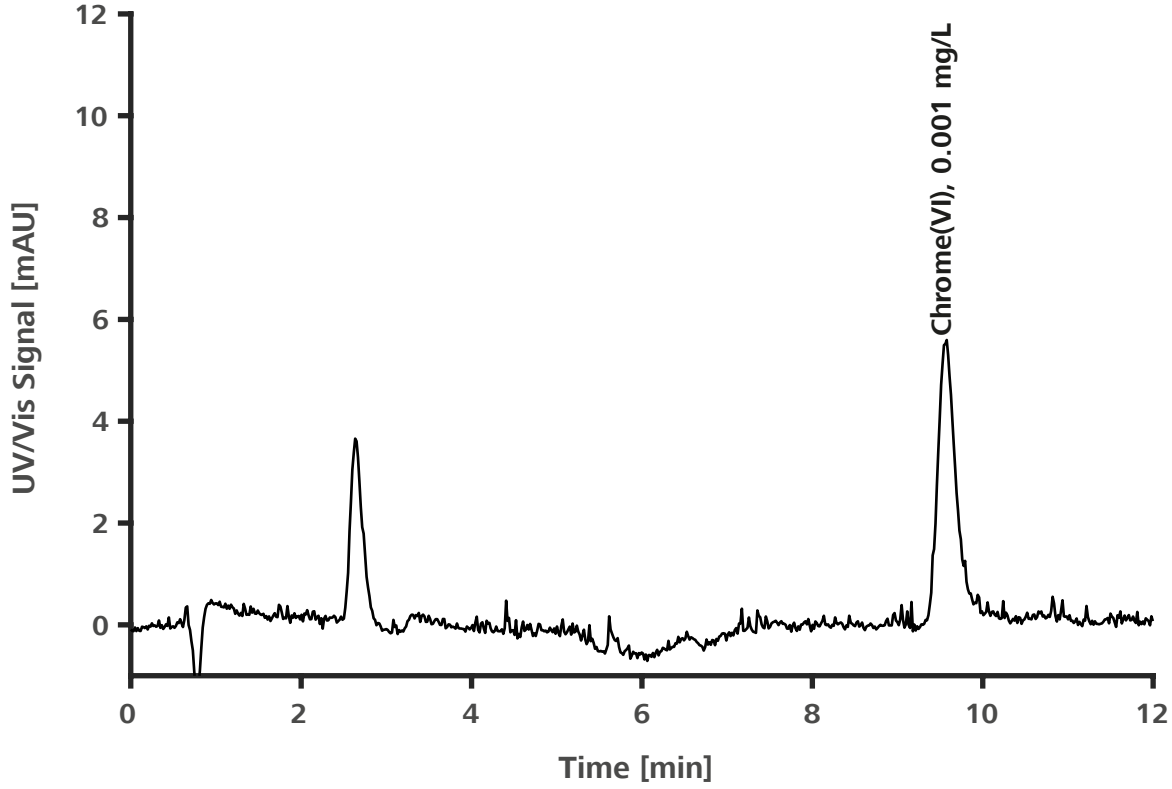
Post-column derivatization reagent:	0.15 mmol/L 4-(2-pyridylazo)resorcinol monosodium salt hydrate, 80 mmol/L HNO_3, 0.4 mol/L ammonium hydroxide
Post-column derivatization reagent flow rate:	0.2 mL/min
Post-column derivatization temperature:	55 °C



5.13 Determination of hexavalent chromium in tap water

Column:	Metrosep A Supp 10 - 250/2.0
Sample preparation:	–
Detection:	UV/VIS after PCR (530 nm)
Suppression:	–
Temperature:	50 °C
Loop:	1000 µL

Flow rate:	0.3 mL/min
Eluent:	10 g/L ammonium sulfate, 6.5 mL/L ammonium hydroxide
Post-column derivatization reagent:	2.0 mmol/L 1,5-Diphenylcarbazide
Post-column derivatization temperature:	50 °C



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 7*.

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 4, page 52)*.

- Contamination with inorganic components *(see table 5, page 52)*.

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

Table 4 Contamination with organic components

	Rinse with	Duration [h]	Flow rate 4 mm [mL/min]	Flow rate 2 mm [mL/min]
1	70% methanol possibly with 1% acetate added	12	1.0	0.25
2	Eluent	2	1.0	0.25

Table 5 Contamination with inorganic components

	Rinse with	Duration [min]	Flow rate 4 mm [mL/min]	Flow rate 2 mm [mL/min]
1	Ultrapure water	30	0.5	0.12
2	0.05 mol/L Na_4 EDTA	100	0.5	0.12
3	0.1 mol/L NaOH	60	0.5	0.12
4	Eluent	120	0.5	0.12

6.2 Decreasing resolution and asymmetrical peaks

Problem

The resolution of peaks deteriorates or peak shapes are asymmetrical.

Causes and prevention

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

Causes	Prevention/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/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/correction
Analytes eluting late	Some 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/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 M-012: Iron speciation analysis in soil using IC-ICP/MS in accordance with EPA SW846 Method 6800
- Application Note P-051: Cyanide and sulfide on Metrosep A Supp 10 - 100/2.0 using amperometric detection
- Application Note P-052: Trace analysis of cyanide and sulfide in aqueous samples – DC amperometric determination after ion chromatographic separation
- Application Note P-059: Sulfide in wastewater with Metrosep A Supp 10 - 100/4.0 and amperometric detection
- Application Note S-223: Chlorate and sulfate in brine
- Application Note S-225: Sulfite besides standard anions in beer on the Metrosep A Supp 10 - 100/4.0
- Application Note S-230: Phosphate and sulfate in polymer samples after inline dilution plus inline dialysis
- Application Note S-231: Nitrite, nitrate and phosphate in sea water from a shrimp farm
- Application Note S-281: Anions in wine
- Application Note S-295: Anions in boiler water including sulfur speciation (sulfite and sulfate)
- Application Note S-318: Fast IC: Separation of standard anions within three minutes
- Application Note S-319: Fast IC: Separation of organic acids anions besides sulfate within three minutes
- Application Note S-320: Fast IC: Drinking water analysis within three minutes
- Application Note S-321: Fast IC: Sulfite besides sulfate in beer within less than ten minutes
- Application Note U-009: Iodate, chlorite, bromate and nitrite by suppressed ion chromatography applying post column reaction (PCR) and UV/VIS detection
- Application Note U-010: Nitrite, nitrate and phosphate in sea water from a shrimp farm
- Application Note U-022: Traces of nitrite in mineral water with UV detection
- Application Note U-056: Nitrite, bromide and nitrate in artificial sea water applying direct UV/VIS detection
- Application Note U-069: Chromate using post-column reaction and UV/VIS detection in accordance with EPA 218.7
- Application Note U-076: Zinc Oxide Assay as per USP General Chapter <591>

- Column catalog, 8.000.5245

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