Column manual

Metrosep C 6 (6.1051.XX0 / 6.01051.XX0)

Manual

8.107.8050EN / 2022-05-12

Metrohm

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Technical Communication
Metrohm AG
CH-9100 Herisau

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Disclaimer

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1 General information

The Metrosep C 6 is a high-capacity separation column. With it, not only standard cations but also a large amount of amines as well as transition metals can be determined. The resolution of sodium to ammonium is excellent on this column.

1.1 Ordering information

Table 1 4-mm columns

Order number	Designation
6.1051.410	Metrosep C 6 - 100/4.0
6.1051.420	Metrosep C 6 - 150/4.0
6.1051.430	Metrosep C 6 - 250/4.0

Table 2 2-mm columns

Order number	Designation
6.01051.210	Metrosep C 6 - 100/2.0
6.01051.220	Metrosep C 6 - 150/2.0
6.01051.230	Metrosep C 6 - 250/2.0

Table 3 Guard columns

Order number	Designation
6.1051.500	Metrosep C 6 Guard/4.0
6.1051.510	Metrosep C 6 S-Guard/4.0
6.01051.600	Metrosep C 6 Guard/2.0
6.01051.610	Metrosep C 6 S-Guard/2.0

1.2 Technical specifications

Column material: Silica gel with carboxyl groups

Particle size: 5 µm

Dimensions

Order number	Dimensions
6.1051.410	100 x 4.0 mm
6.1051.420	150 x 4.0 mm
6.1051.430	250 x 4.0 mm
6.01051.210	100 x 2.0 mm
6.01051.220	150 x 2.0 mm
6.01051.230	250 x 2.0 mm

pH range: Eluent: 2 to 7
Sample: 2 to 10

Temperature range: 20 to 60 °C

Recommended standard temperature: 30 °C

Maximum pressure

4 mm	20 MPa (200 bar)
2 mm	20 MPa (200 bar)

Flow rate

Order number	Recommended flow rate	Maximum flow rate
6.1051.410	0.9 mL/min	3.5 mL/min
6.1051.420	0.9 mL/min	2.5 mL/min
6.1051.430	0.9 mL/min	1.5 mL/min
6.01051.210	0.25 mL/min	1.0 mL/min
6.01051.220	0.25 mL/min	0.7 mL/min
6.01051.230	0.25 mL/min	0.4 mL/min

Standard eluent: 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid

Permitted organic additives

In the eluent: 0 to 100% acetonitrile, acetone and no alcohol

In the sample matrix: 0 to 100% acetonitrile, acetone and alcohol

Capacity

Order number	Capacity
6.1051.410	20 (K^+)
6.1051.420	30 (K^+)
6.1051.430	50 (K^+)
6.01051.210	5 (K^+)
6.01051.220	8 (K^+)
6.01051.230	13 (K^+)

Preparation

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

Storage

Store the column in standard eluent at ambient temperature.

Typical pressure

For columns without a guard column under standard conditions

Order number	Typical pressure
6.1051.410	4.8 ± 2 MPa
6.1051.420	6.9 ± 2 MPa
6.1051.430	10.7 ± 2 MPa
6.01051.210	3.4 ± 2 MPa
6.01051.220	6.3 ± 2 MPa
6.01051.230	9.9 ± 2 MPa

Column housing

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

Application

Determination of standard cations, alkaline metals and alkaline-earth metals as well as amines and transition metals in aqueous media.

2 Key aspects of working with separation columns

Storage

Rinse the column with ultrapure water. Once the backpressure in your ion chromatograph has dissipated, remove the column at ambient temperature. Seal the column at both ends using the original stoppers (6.2744.060). Keep them refrigerated at 4 to 8 °C, if possible.

Bacterial growth

Bacterial growth significantly affects chromatography and ruins separation columns. A vast array of problems in chromatography are caused by the growth of algae, bacteria and fungi.

In order to prevent bacterial growth, always use fresh eluents, rinsing solutions and regeneration solutions. Do not use any solutions that have not been used for a prolonged period. We recommend cleaning all vessels as follows before filling them:

1. Thoroughly rinse with ultrapure, UV-treated water (> 18.2 MΩ).
2. Swirl an acetone-water mixture around in the vessel.
3. Rinse again with ultrapure water.

If you notice the growth of bacteria or algae despite these precautionary measures, add 5% acetonitrile or acetone to the eluent. Only do this if you are *not using a membrane suppressor*. Membrane suppressors can be destroyed by organic solvents. 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.

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).

Degassing the eluent

In order to prevent bubbles from forming, we recommend degassing the produced eluent before using it in your IC system. To degas the eluent, create a vacuum for approximately 10 minutes using a water-jet pump or

an oil pump. Alternatively, use an ultrasonic bath or work with an eluent degasser.

Filter

Problems that occur in IC systems are usually related to particles. These particles can be introduced from the following sources:

- Bacterial growth
- Unfiltered eluents
- The sample
- The rinsing solution and/or regeneration solution

Minimize this risk by using an aspiration filter (6.2821.090), 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. We also recommend changing the filters regularly.

Filtering the eluent

All eluents have to be microfiltered (0.45 µm) immediately before use.

Mechanical stress

Mechanical loads on the column should be avoided. For example, the column impacting a hard surface can cause a break or gap in the column packing (separation phase material). This affects the chromatography results. The column would be irreparably damaged as a result.

Particles

All solutions, samples, regeneration solutions, water and eluents must be free of particles. Particles clog separation columns over time. The column pressure increases. Be especially conscious of ensuring that there are no particles present when producing eluents. The eluent continuously flows through the column at a rate of 500 to 1,000 mL per workday compared to about 0.5 mL of the sample solution. Filter or dialyze your sample automatically with one of the Metrohm Inline Sample Preparation techniques (MISP).

Sample preparation cartridges

Sample preparation cartridges are used to prepare critical samples that cannot be injected directly into the separation column. They perform tasks such as removing organic contaminants or neutralizing heavily alkaline or acidic samples. Sample preparation cartridges are consumables that generally cannot be regenerated. Sample preparation cartridges do not replace the guard columns, which should always be used with each separation column. Metrohm Inline Sample Preparation techniques (MISP) can be used as an alternative to sample preparation cartridges.

Pulsation absorber

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

Regenerating separation columns

If separation columns are operated with clean eluents and filled with samples free of particles, you can expect the column to have a long service

life. This means regenerating the column is not required and, after a multitude of injections, no longer possible.

If the pressure in the column increases unexpectedly despite this or if the separating efficiency decreases, the regeneration steps specified for every column can be carried out. Generally, it is important to keep in mind that the regeneration takes place outside the analytical line. Connect the separation column to the pump directly. Route the regeneration solution through the column directly into a waste container. Before reinstalling the separation column, it must be properly rinsed with fresh eluent.

Shutting down the ion chromatograph

If you will not be working with the ion chromatograph for a prolonged period (> 1 week), we recommend removing the separation column and sealing it with the stoppers provided. Rinse the ion chromatograph, including all three suppressor chambers, with methanol/water (1:4). Store the separation column in the medium indicated on the column leaflet and, ideally, at a temperature between 4 and 8 °C if not specified otherwise.

When you return the instrument to operation, rinse the ion chromatograph with fresh eluent. Bring the separation column back to ambient temperature before you install it. Then increase the temperature if necessary.

Fun

Ion chromatography should be fun and should not stress you out. Metrohm puts all its work into ensuring you can work reliably with your IC systems with minimal maintenance, servicing and costs. Metrosep separation columns embody the attributes of quality, long service life and excellent results.

Environmental protection

A significant advantage of ion chromatography is that most of the work involves aqueous media. As a result, the chemicals used in ion chromatography are largely non-toxic and do not impact the environment. However, if you are working with acids, bases, organic solvents or heavy metal standards, dispose of them properly after use.

Guard columns

Guard columns are used to protect separation columns. We strongly recommend their use. They normally contain the same stationary phase also used in the separation columns. However, the quantity is significantly reduced to avoid impacting the chromatography. Guard columns remove critical contaminants that could react with column material; they also effectively remove particles and bacterial contaminants. Replace the guard column in the following cases:

- If the backpressure in the system increases.
- If the chromatography results deteriorate.

Guard columns are available for all Metrosep separation columns.

We recommend replacing the guard column three to four times during the service life of the analytical column.

Water quality

Aqueous media are mostly used in work involving ion chromatography. This means that water quality is a critical factor for good chromatography. If the water quality is inadequate, the results will be as well. In addition, there is a risk of damaging instruments and separation columns when using water with inadequate quality. The ultrapure water being used should have a resistivity greater than 18.2 MΩ·cm and should be free of particles. Therefore, we recommend filtering the water using a 0.45 µm filter and treating it with UV light. Modern ultrapure water systems for laboratory use ensure this level of water quality (Type I).

3 Eluent production

We recommend selecting a high level of purity for chemicals for both standard production and eluent production.

3.1 Chemicals

Recommended chemicals

- Nitric acid, HNO_3, 2 mol/L
  Sigma Aldrich order number: 35278
- Dipicolinic acid, $C_7H_5NO_4$, 99%
  Sigma Aldrich order number: 63808
- 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 1.7 mmol/L of nitric acid and 1.7 mmol/L of dipicolinic acid:

Producing 2 L of standard eluent

1
- Pre-rinse the eluent bottle with ultrapure water several times.
- Set out 1.7 L of ultrapure water.

2 If the eluent is not degassed using an eluent degasser:

- Degas the ultrapure water for the eluent using a vacuum pump. This prevents problems with air bubbles in the high-pressure pump.

3 Measure the following quantity of chemicals:

- Nitric acid: 1.7 mL
- Dipicolinic acid: 568.1 mg

4
- Pipette 1.7 mL nitric acid into the eluent bottle.
- Put approx. 200 mL ultrapure water in a beaker.
- Add 568.1 mg dipicolinic acid into the beaker.
- Heat the beaker for approx. 1 minute in the microwave at full potential.
  If no microwave is available, heat the suspension to 80 °C.

- Afterwards, swirl the beaker until the dipicolinic acid has dissolved.
- Pour the solution into the eluent bottle.
- Fill the eluent bottle with ultrapure water to 2 L.

This eluent (1.7 mmol/L of nitric acid, 1.7 mmol/L of dipicolinic acid) can be used to achieve background conductivity of approx. 900 µS/cm. The noise is typically less than 2 nS/cm.

4 Start-up

4.1 Connecting and rinsing the guard column

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

NOTICE

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

NOTICE

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

CAUTION

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

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

NOTICE

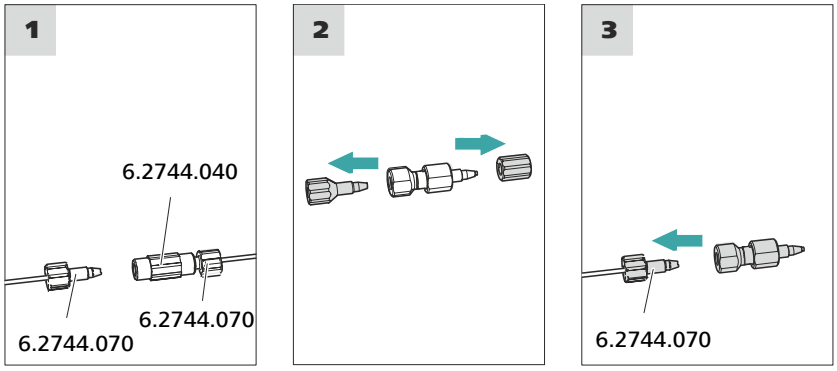
Only connect the guard column after the initial start-up of the instrument . Until then, replace the guard column and the separation column with couplings (6.2744.040).

Accessories

For this step, you need the following accessories:

- Guard column (suitable for separation column)

Connecting the guard column



1 Removing the coupling

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

2 Preparing the guard column

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

3 Connecting the guard column

CAUTION

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

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

Rinsing the guard column

1 Rinsing the guard column

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

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

4.2 Connecting the separation column

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

NOTICE

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

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

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

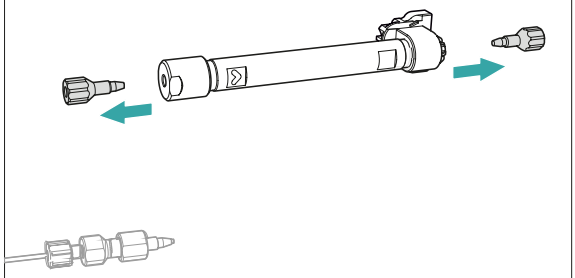
CAUTION

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

NOTICE

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

1



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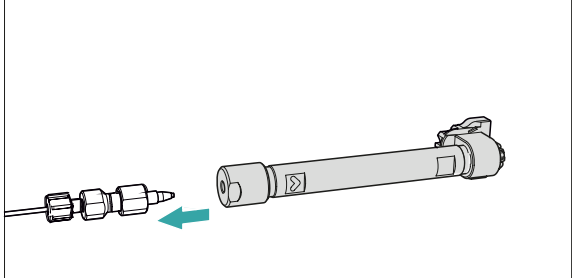

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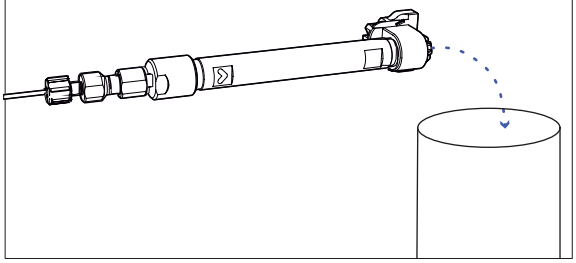

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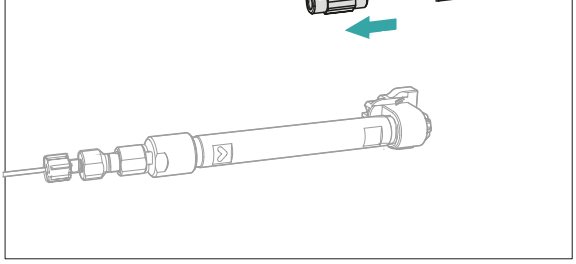

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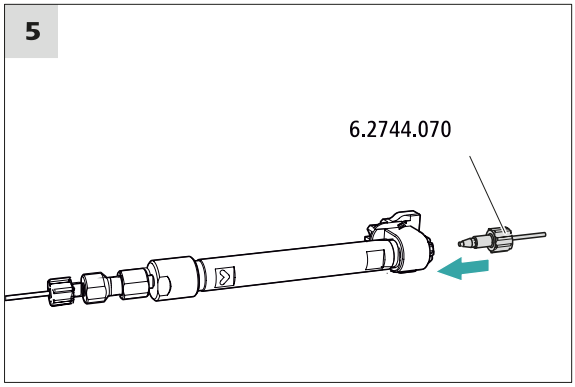

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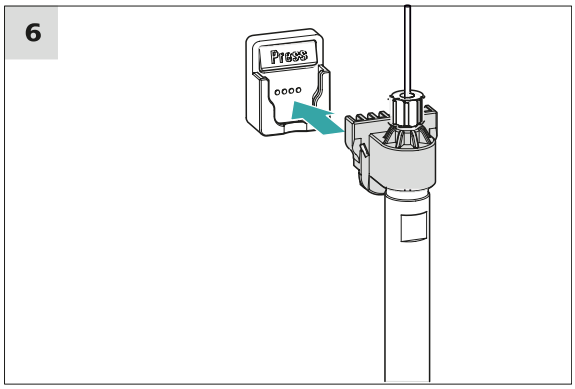


Connecting the separation column

1 Removing the stoppers

- Remove the stoppers from the separation column.

2 Installing the inlet of the separation column

CAUTION

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

There are 3 possibilities:

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

3 Rinsing the separation column

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

4 Removing the coupling

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

5 Installing the outlet of the separation column

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

6 Inserting the separation column

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

The separation column is now detected by MagIC Net.

4.3 Conditioning

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

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

NOTICE

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

Conditioning the system

1 Preparing the software

CAUTION

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

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

2 Preparing the instrument

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

3 Starting equilibration

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

4 Conditioning the system

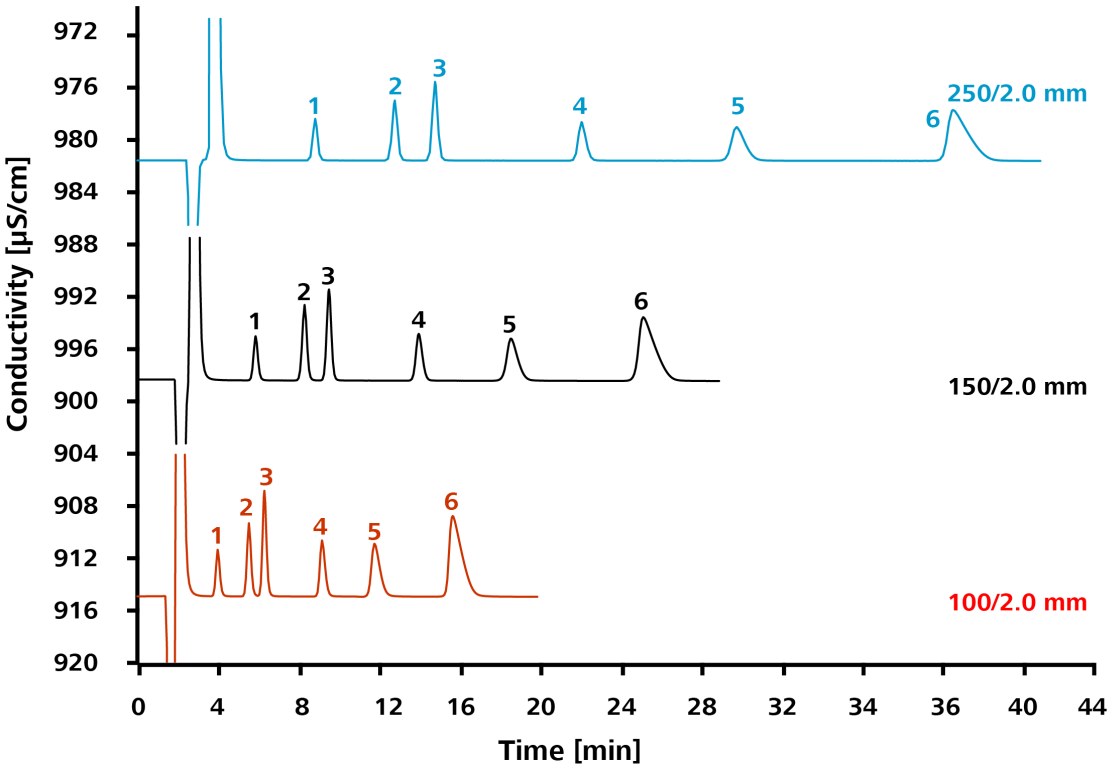
Continue rinsing the system with eluent until the desired stability level for the baseline has been attained .

The instrument is now ready for measuring samples.

5 Applications

5.1 Standard chromatogram

	2-mm columns
Sample preparation:	–
Detection:	Conductivity
Suppression:	–
Temperature:	30 °C
Loop:	20 µL
Flow rate:	0.25 mL/min
Eluent:	1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid



	Metrosep C 6 - XX0/2.0	mg/L
1	Lithium	1

	Metrosep C 6 - XX0/2.0	mg/L
2	Sodium	5
3	Ammonium	5
4	Potassium	10
5	Calcium	10
6	Magnesium	10

4-mm columns

Sample preparation: –

Detection: Conductivity

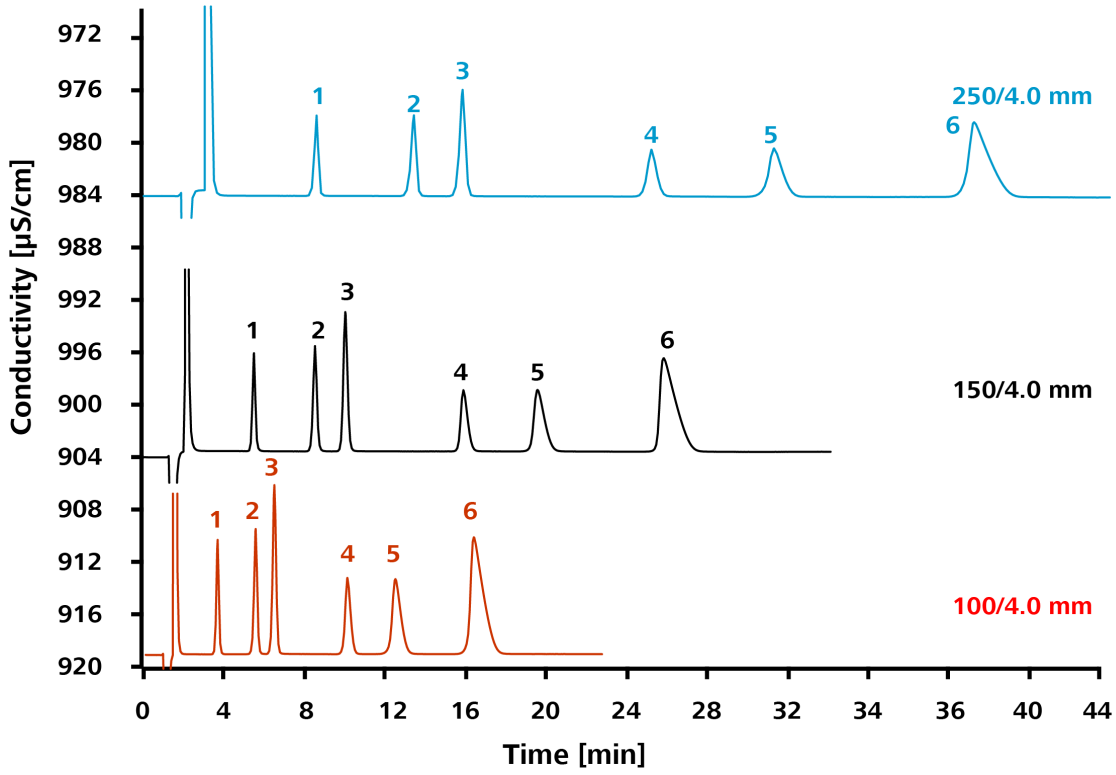
Suppression: –

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.9 mL/min

Eluent: 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid



	Metrosep C 6 - XX0/4.0	mg/L
1	Lithium	1
2	Sodium	5
3	Ammonium	5
4	Potassium	10
5	Calcium	10
6	Magnesium	10

5.2 Effects of temperature

Column: Metrosep C 6 - 150/4.0

Sample preparation: –

Detection: Conductivity

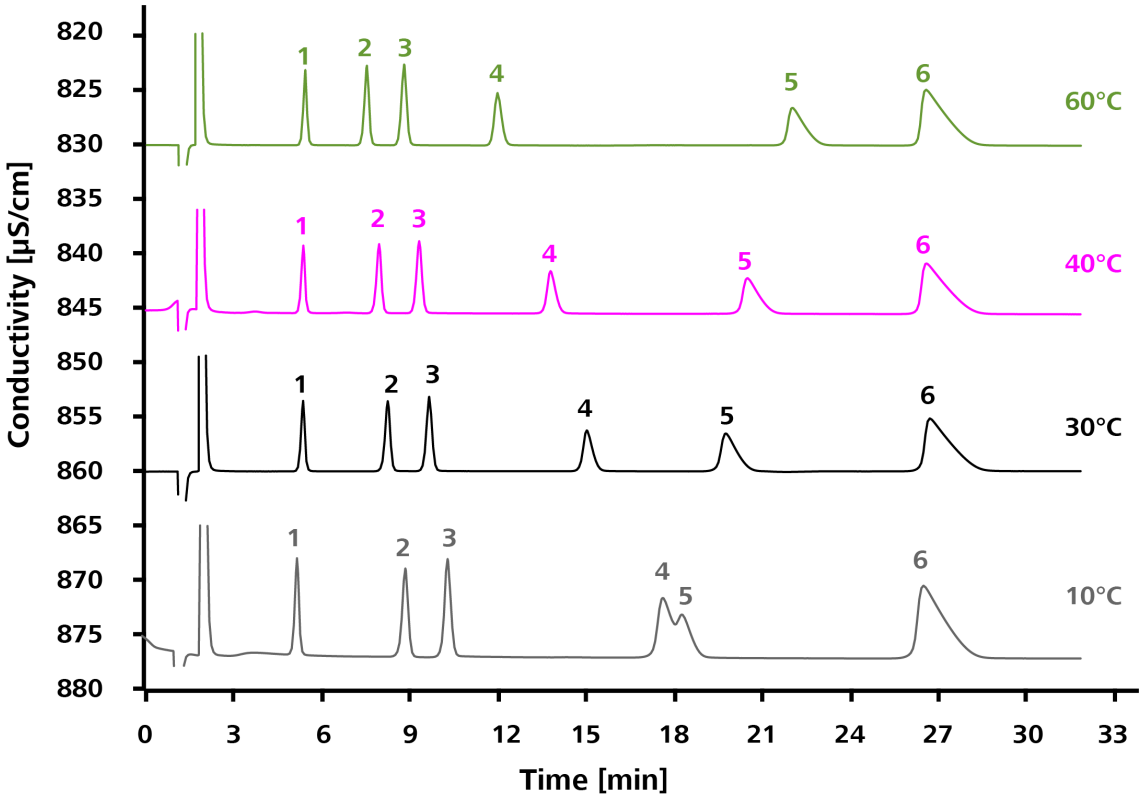
Suppression: –

Temperature: 20 to 60 °C

Loop: 20 µL

Flow rate: 0.9 mL/min

Eluent: 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid



	Metrosep C 6 - 150/4.0	mg/L
1	Lithium	1
2	Sodium	5
3	Ammonium	5
4	Potassium	10
5	Calcium	10
6	Magnesium	10

In the case of calcium, the complexation with the dipicolinic acid is weakened with higher temperature. This results in it eluting later. An increase in temperature results in slightly longer retention times for lithium and magnesium. The other cations such as sodium, ammonium and potassium elute earlier.

5.3 Eluent flow rate variation

Column: Metrosep C 6 - 150/4.0

Sample preparation: –

Detection: Conductivity

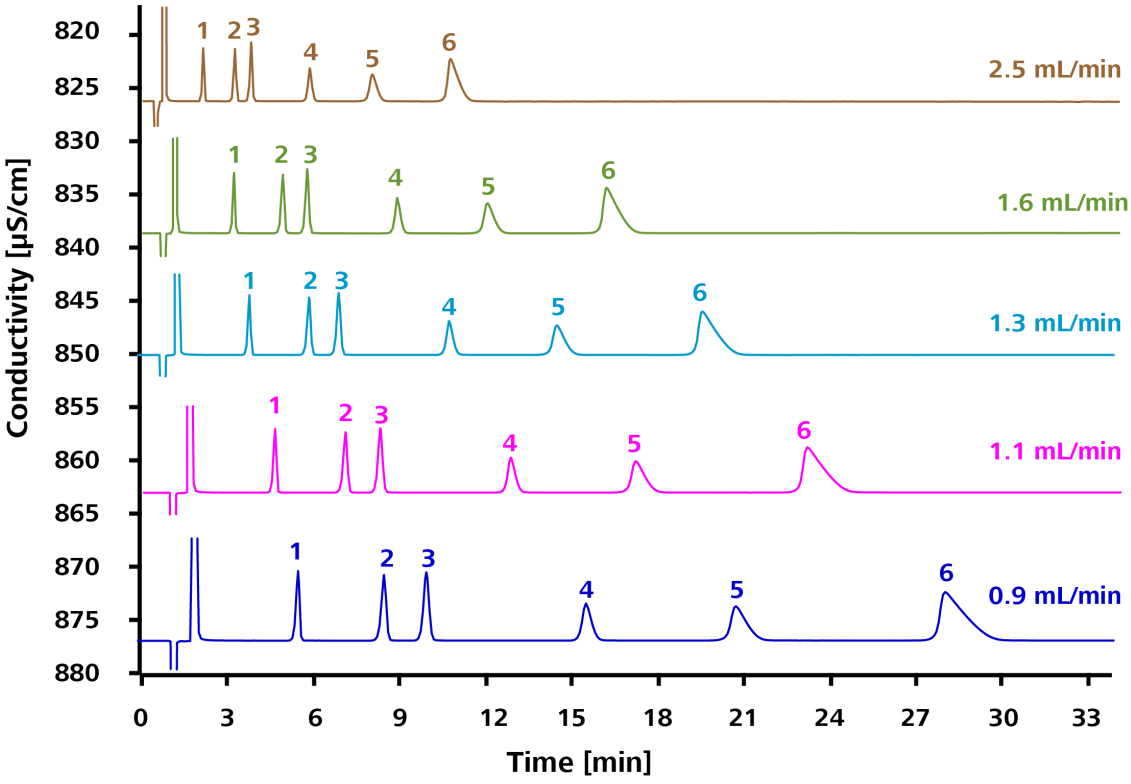
Suppression: –

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.9 up to 2.5 mL/min

Eluent: 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid



	Metrosep C 6 - 150/4.0	mg/L
1	Lithium	1
2	Sodium	5
3	Ammonium	5

	Metrosep C 6 - 150/4.0	mg/L
4	Potassium	10
5	Calcium	10
6	Magnesium	10

All cations elute faster when the flow rate is higher. At 2.5 mL/min, the backpressure reaches 17 MPa.

5.4 Variation of the eluent

5.4.1 Variation of the nitric acid concentration

Influence on standard cations

Column: Metrosep C 6 - 150/4.0

Sample preparation: –

Detection: Conductivity

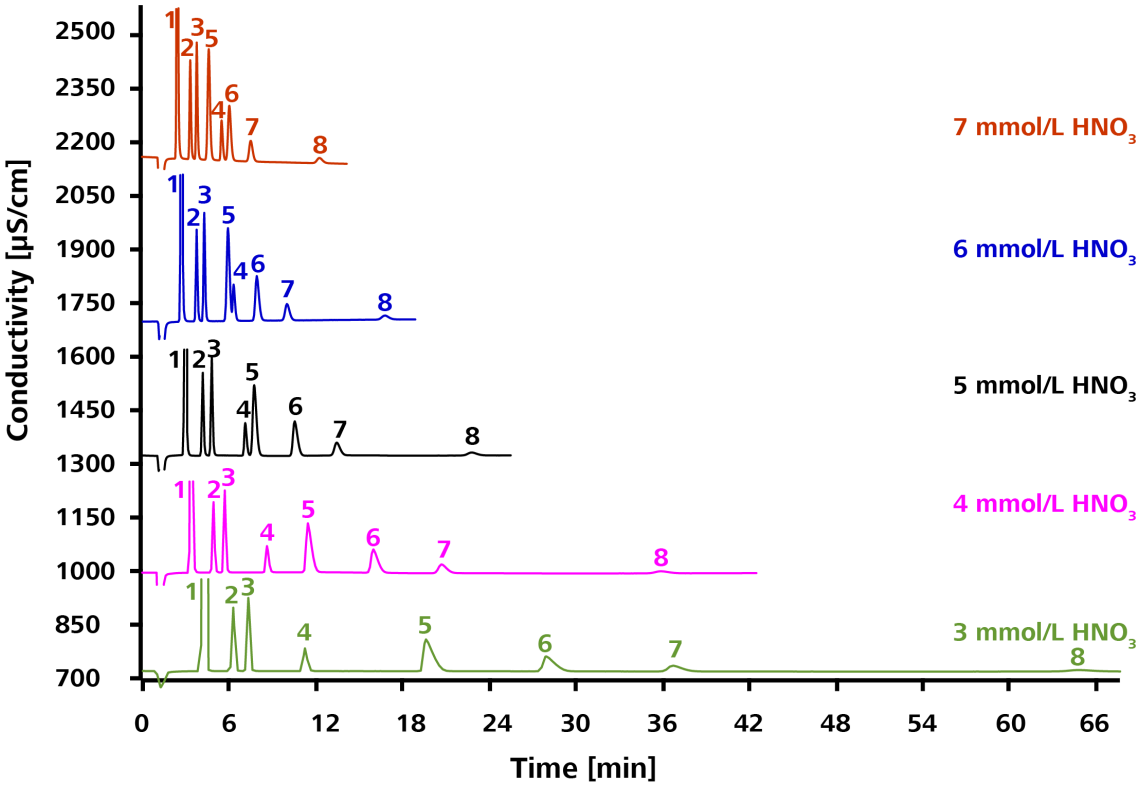
Suppression: –

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.9 mL/min

Eluent: A) 3 mmol/L nitric acid
B) 4 mmol/L nitric acid
C) 5 mmol/L nitric acid
D) 6 mmol/L nitric acid
E) 7 mmol/L nitric acid



	Metrosep C 6 - 150/4.0	mg/L
1	Lithium	1
2	Sodium	5
3	Ammonium	5
4	Potassium	10
5	Magnesium	10
6	Calcium	10
7	Strontium	10
8	Barium	10

Increasing the nitric acid concentration accelerates all standard cations. The divalent cations are accelerated disproportionately. At 5 mmol/L, calcium co-elutes with potassium. From 6 mmol/L, magnesium elutes before potassium.

Influence on amines

Column: Metrosep C 6 - 150/4.0

Sample preparation: –

Detection: Conductivity

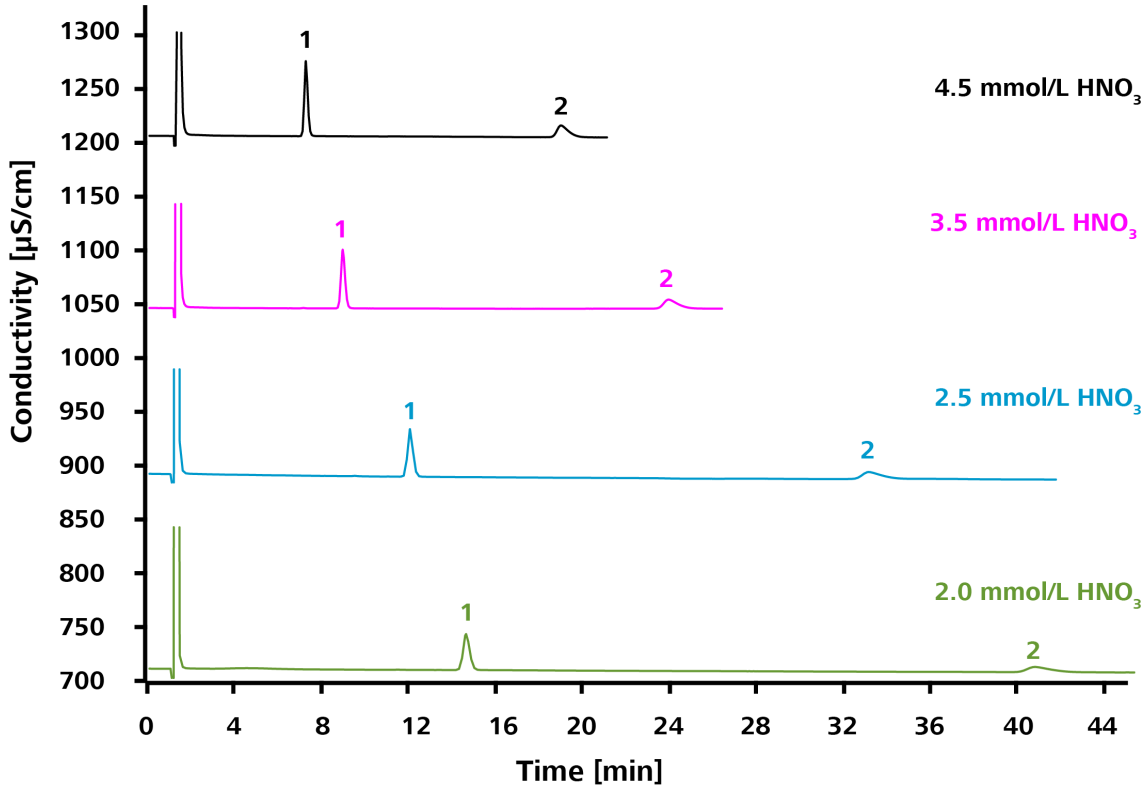
Suppression: –

Temperature: 30 °C

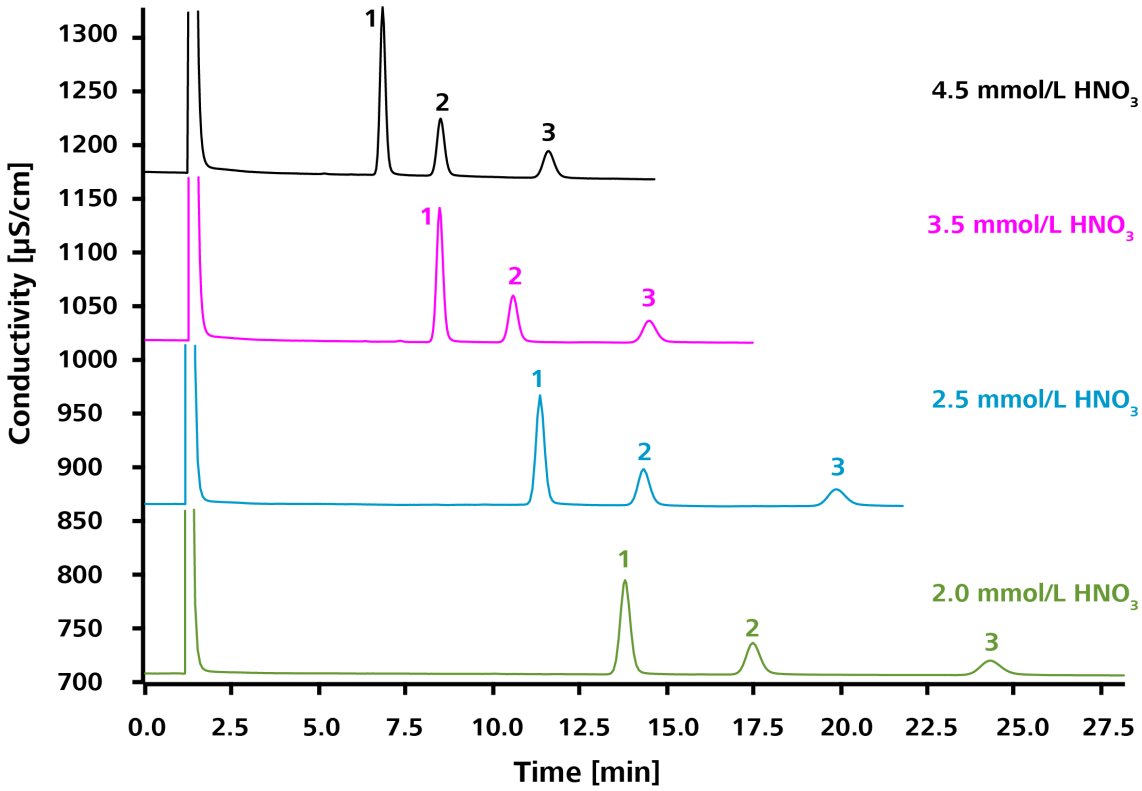
Loop: 20 µL

Flow rate: 0.9 mL/min

Eluent: A) 2 mmol/L nitric acid
B) 2.5 mmol/L nitric acid
C) 3.5 mmol/L nitric acid
D) 4.5 mmol/L nitric acid



	Metrosep C 6 - 150/4.0	mg/L
1	Methylamine	10
2	Trimethylamine	10



	Metrosep C 6 - 150/4.0	mg/L
1	Monoethanolamine	10
2	Diethanolamine	10
3	Triethanolamine	10

Increasing the nitric acid concentration shortens the retention time of the amines.

5.4.2 Variation of the dipicolinic acid concentration

Column: Metrosep C 6 - 150/4.0

Sample preparation: –

Detection: Conductivity

Suppression: –

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.9 mL/min

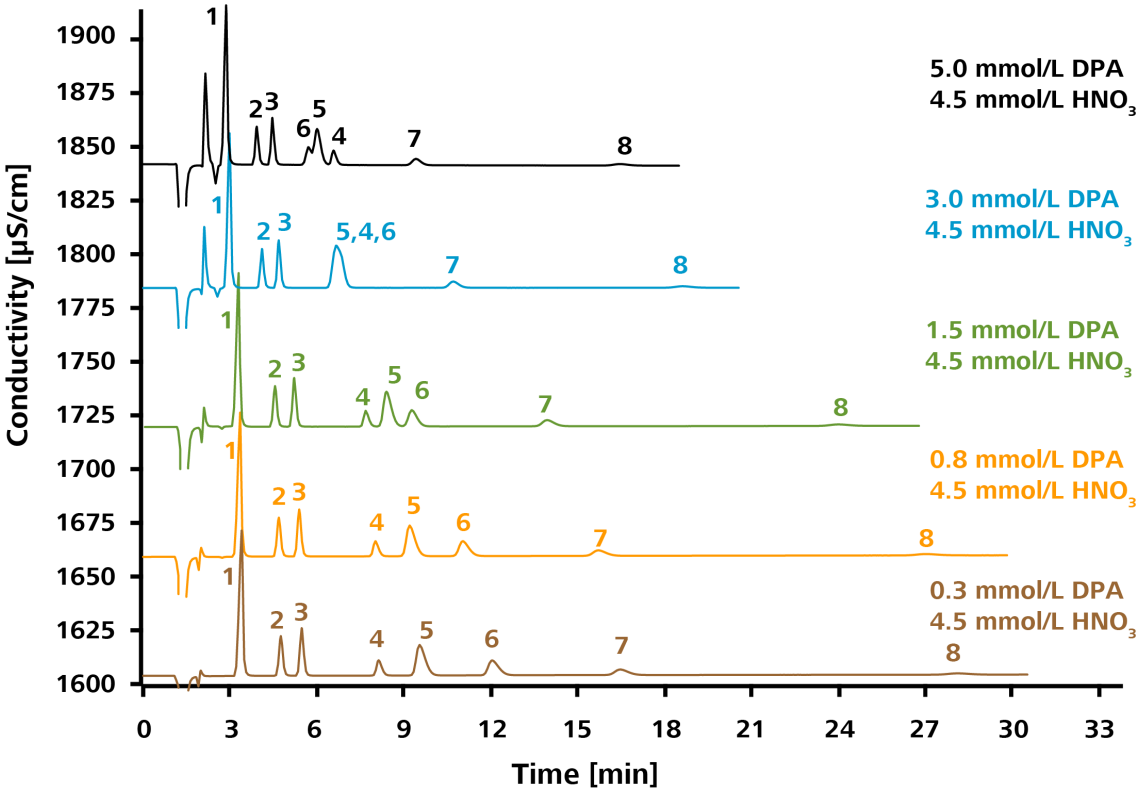
Eluent: A) 4.5 mmol/L nitric acid, 0.3 mmol/L dipicolinic acid

B) 4.5 mmol/L nitric acid, 0.8 mmol/L dipicolinic acid

C) 4.5 mmol/L nitric acid, 1.5 mmol/L dipicolinic acid

D) 4.5 mmol/L nitric acid, 3.0 mmol/L dipicolinic acid

E) 4.5 mmol/L nitric acid, 5.0 mmol/L dipicolinic acid



	Metrosep C 6 - 150/4.0	mg/L
1	Lithium	10
2	Sodium	10
3	Ammonium	10
4	Potassium	10
5	Magnesium	10
6	Calcium	10
7	Strontium	10
8	Barium	10

The addition of dipicolinic acid to the nitric acid influences the retention of calcium, magnesium, barium and strontium through complexation. Increasing the dipicolinic acid concentration accelerates these cations. The best separation is reached with 1.5 mmol/L dipicolinic acid. From 3.0 mmol/L dipicolinic acid, calcium, magnesium and potassium co-elute. At 5 mmol/L, calcium and magnesium elute before potassium.

5.4.3 Variation of the oxalic acid concentration

Column: Metrosep C 6 - 150/4.0

Sample preparation: –

Detection: Conductivity

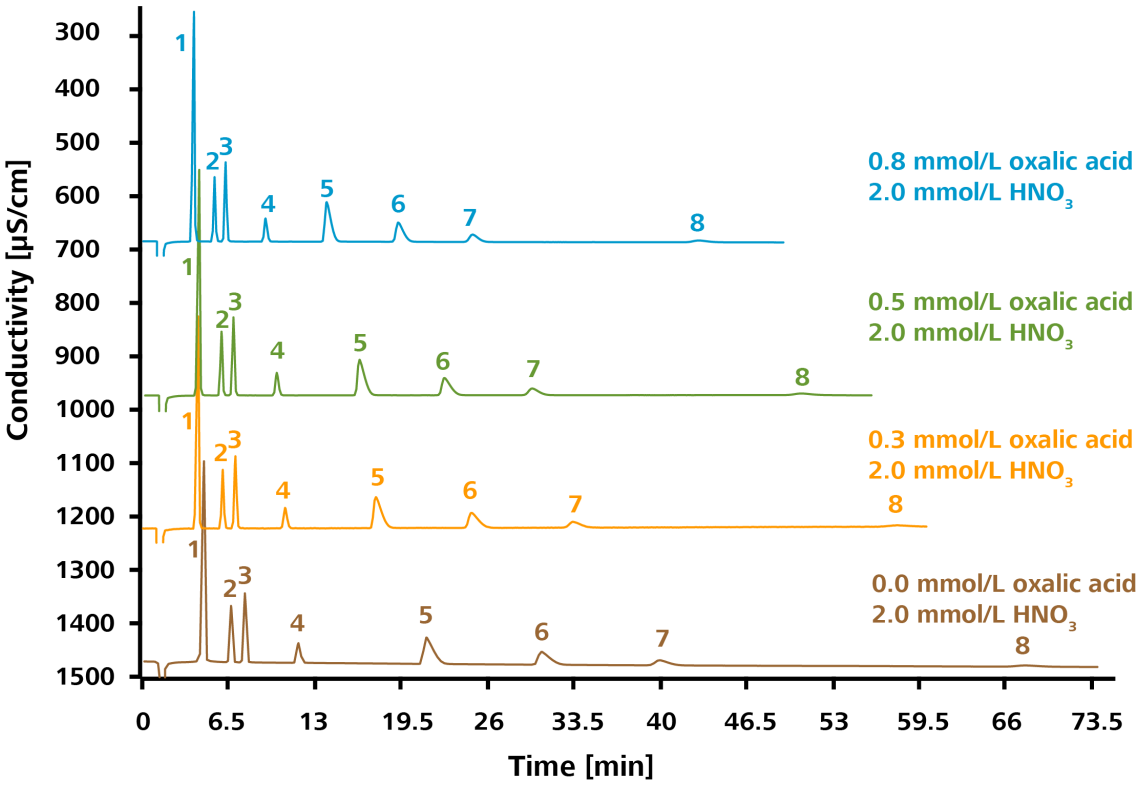
Suppression: –

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.9 mL/min

Eluent: A) 2 mmol/L nitric acid, 0 mmol/L oxalic acid
B) 2 mmol/L nitric acid, 0.3 mmol/L oxalic acid
C) 2 mmol/L nitric acid, 0.5 mmol/L oxalic acid
D) 2 mmol/L nitric acid, 0.8 mmol/L oxalic acid



	Metrosep C 6 - 150/4.0	mg/L
1	Lithium	10

	Metrosep C 6 - 150/4.0	mg/L
2	Sodium	10
3	Ammonium	10
4	Potassium	10
5	Magnesium	10
6	Calcium	10
7	Strontium	10
8	Barium	10

When the oxalic acid is increased, the cations elute earlier.

Column: Metrosep C 6 - 150/4.0

Sample preparation: –

Detection: Conductivity

Suppression: –

Temperature: 30 °C

Loop: 20 µL

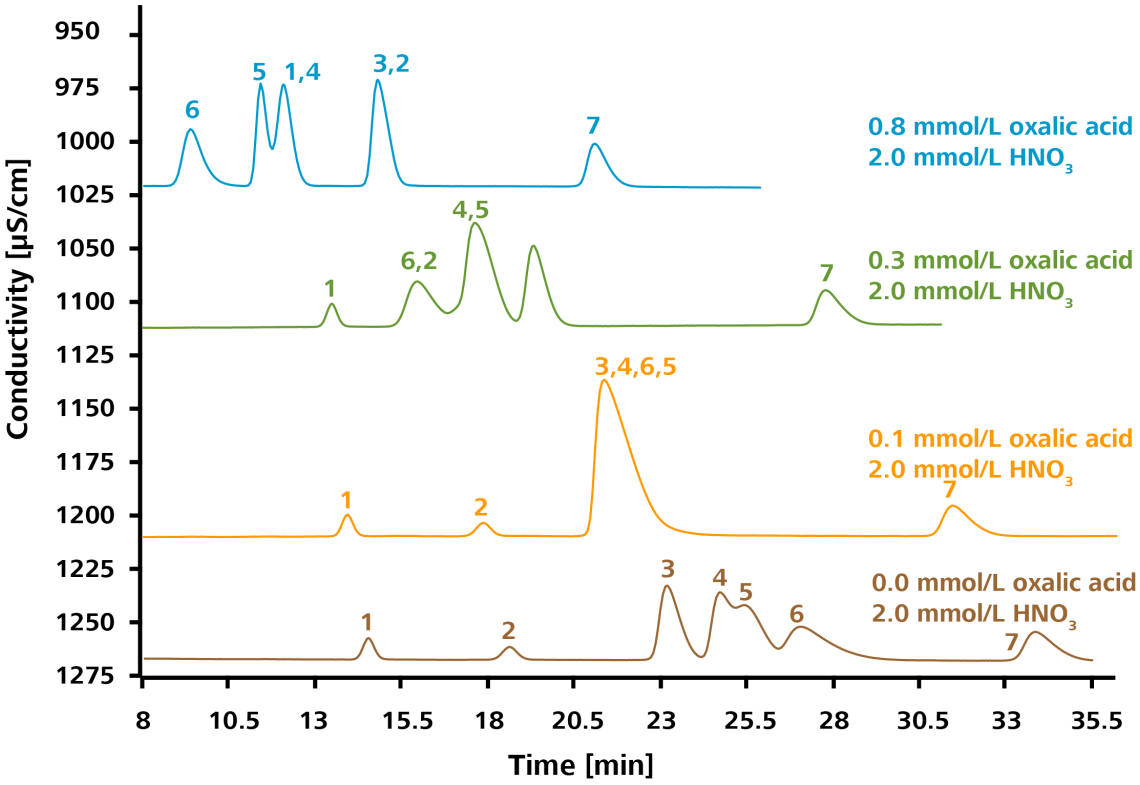
Flow rate: 0.9 mL/min

Eluent: A) 2 mmol/L nitric acid, 0 mmol/L oxalic acid

B) 2 mmol/L nitric acid, 0.1 mmol/L oxalic acid

C) 2 mmol/L nitric acid, 0.3 mmol/L oxalic acid

D) 2 mmol/L nitric acid, 0.8 mmol/L oxalic acid



	Metrosep C 6 - 150/4.0	mg/L
1	Rubidium	10
2	Cesium	10
3	Manganese	10
4	Cobalt	10
5	Zinc	10
6	Nickel	10
7	Cadmium	10

Increasing the oxalic acid influences the rubidium and cesium only slightly. Nickel and zinc, however, are accelerated considerably by adding oxalic acid.

5.5 Variation of organic modifiers

5.5.1 Variation of the acetone concentration

Column: Metrosep C 6 - 150/4.0

Sample preparation: –

Detection: Conductivity

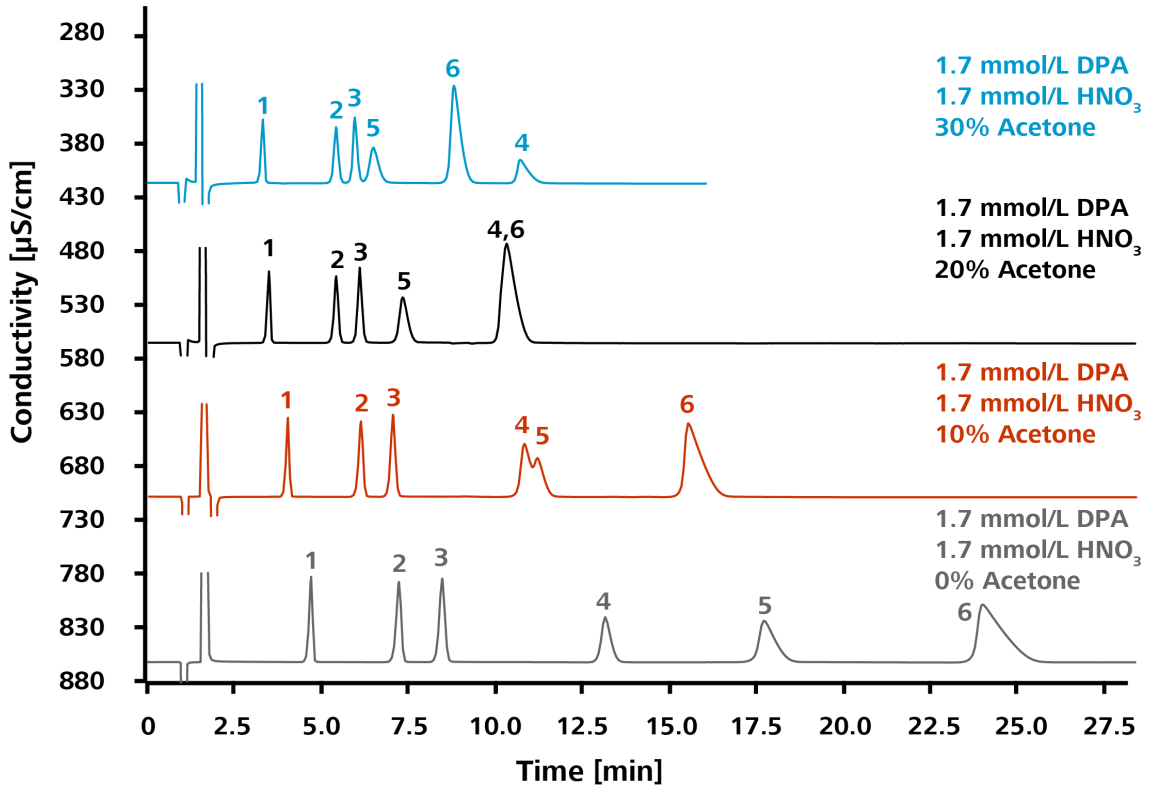
Suppression: –

Temperature: 30 °C

Loop: 10 µL

Flow rate: 0.9 mL/min

Eluent: A) 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid, 0% acetone
B) 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid, 10% acetone
C) 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid, 20% acetone
D) 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid, 30% acetone



	Metrosep C 6 - 150/4.0	mg/L
1	Lithium	1
2	Sodium	5
3	Ammonium	5
4	Potassium	10
5	Calcium	10
6	Magnesium	10

By adding acetone to the eluent, the retention time of all cations is shortened, whereat the effect is more visible in divalent cations. From 10% acetone in the eluent, potassium and calcium co-elute. From 20% acetone, magnesium and potassium co-elute. The pressure increases more by adding acetone than by adding acetonitrile.

5.5.2 Variation of the acetonitrile concentration

Column: Metrosep C 6 - 150/4.0

Sample preparation: –

Detection: Conductivity

Suppression: –

Temperature: 30 °C

Loop: 20 µL

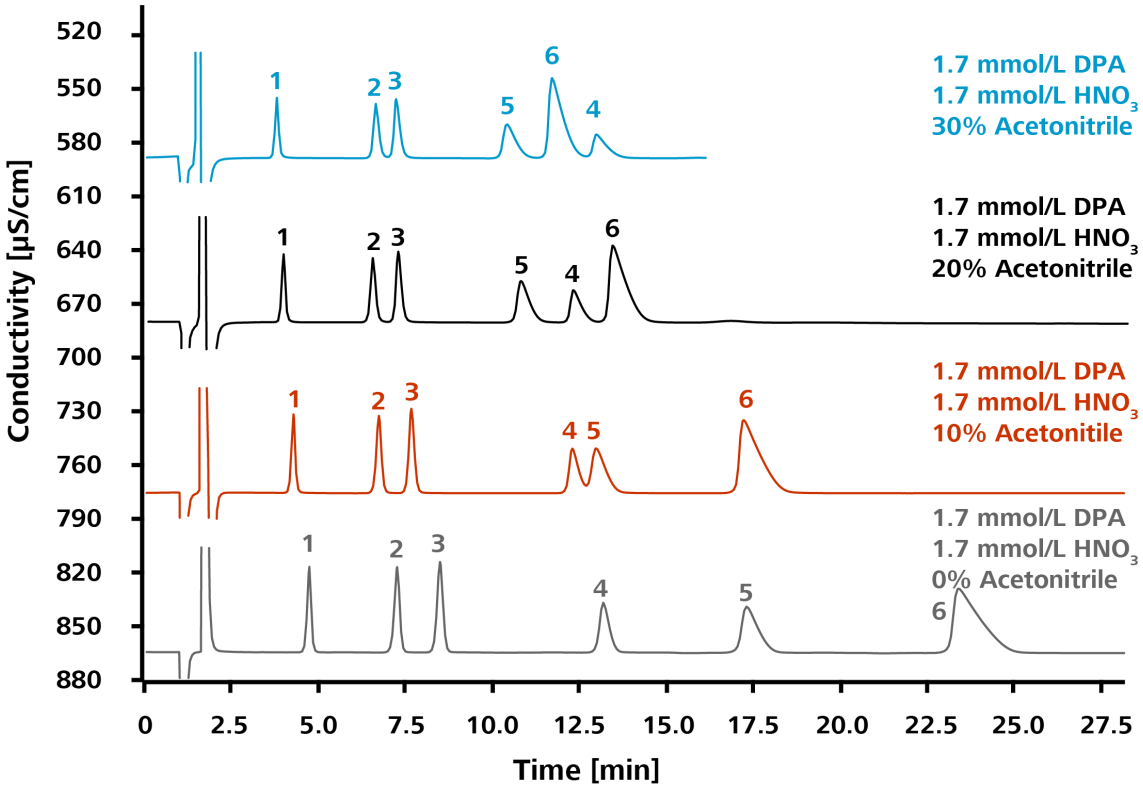
Flow rate: 0.9 mL/min

Eluent: A) 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid, 0% acetonitrile

B) 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid, 10% acetonitrile

C) 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid, 20% acetonitrile

D) 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid, 30% acetonitrile



	Metrosep C 6 - 150/4.0	mg/L
1	Lithium	1
2	Sodium	5
3	Ammonium	5
4	Potassium	10
5	Calcium	10
6	Magnesium	10

By adding acetonitrile to the eluent, the retention time of all cations is shortened, whereat the effect is more visible in divalent cations. When adding 10% acetonitrile, potassium and calcium co-elute. When adding 20% acetonitrile, calcium elutes before potassium.

5.6 Determination of ammonium in sodium-rich sample

Column: Metrosep C 6 - 250/4.0

Sample preparation: –

Detection: Conductivity

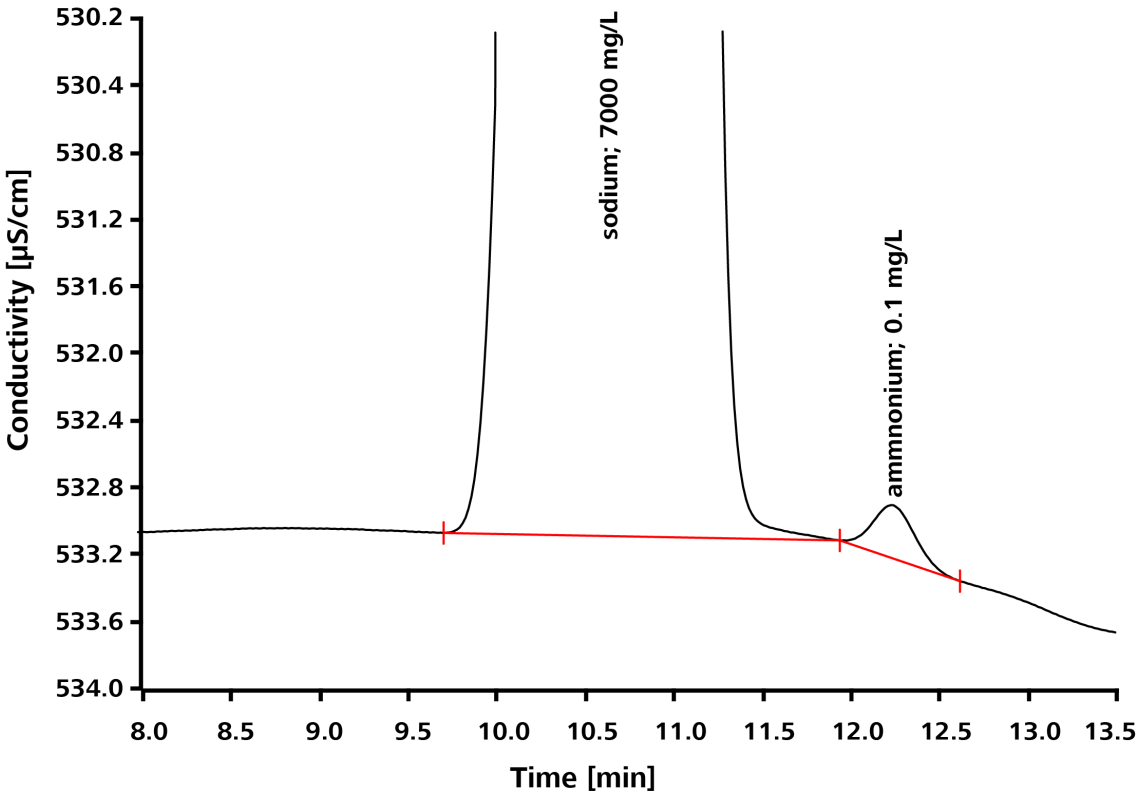
Suppression: –

Temperature: 30 °C

Loop: 5 µL

Flow rate: 0.9 mL/min

Eluent: 4.0 mmol/L nitric acid, 1.0 mmol/L dipicolinic acid



The very high sodium/ammonium ratio reached is 70,000:1 here.

5.7 Determination of trimethylamine and choline in acetone-free eluent

Column: Metrosep C 6 - 100/4.0

Sample preparation: Dilution (1:1,000) in 4 mmol/L nitric acid

Detection: Conductivity

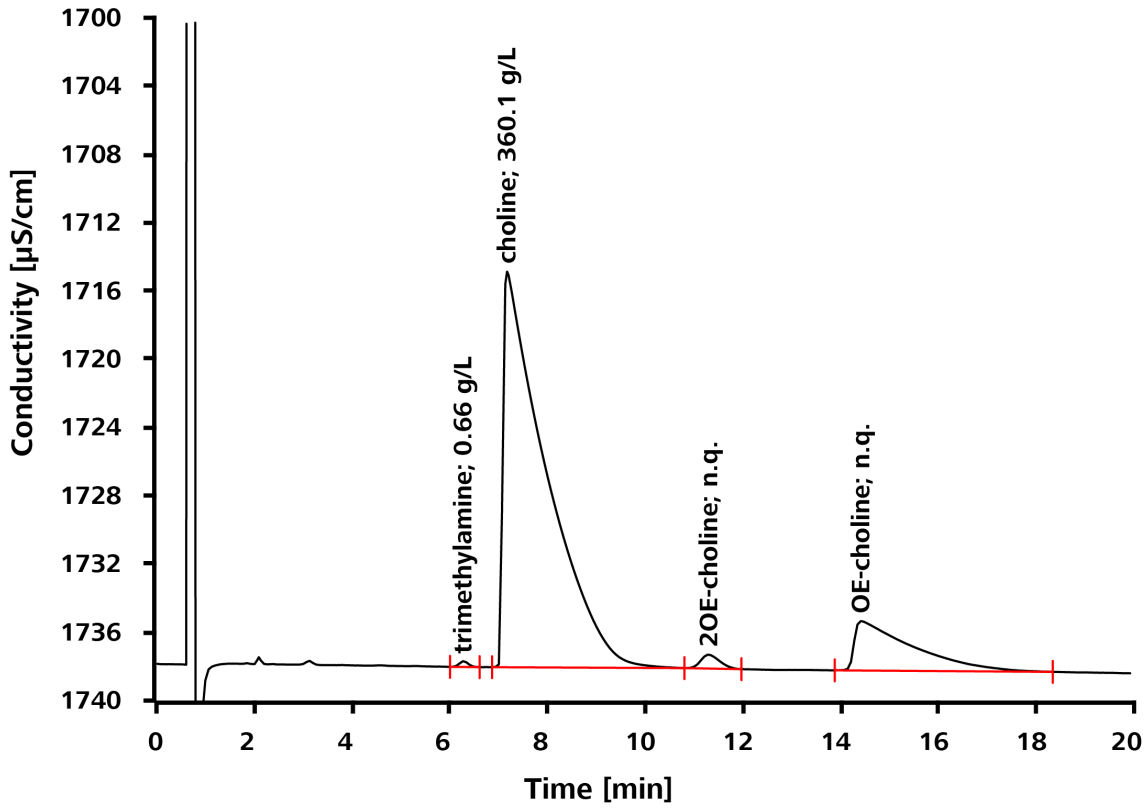
Suppression: –

Temperature: 30 °C

Loop: 50 µL

Flow rate: 1.3 mL/min

Eluent: 6.4 mmol/L nitric acid



2OE-choline and OE-choline are byproducts of the analyte, which have not been quantified.

5.8 Determination of various amines from refineries

Column: Metrosep C 6 - 250/4.0

Sample preparation: –

Detection: Conductivity

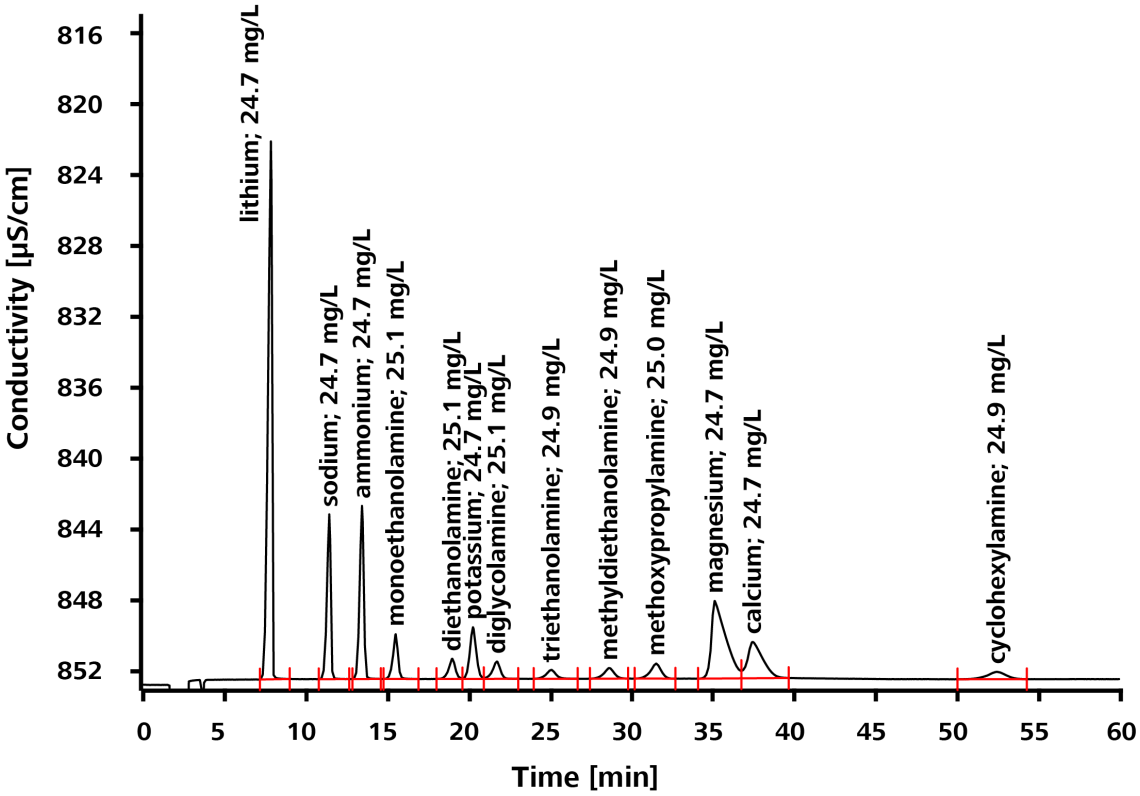
Suppression: –

Temperature: 40 °C

Loop: 10 µL

Flow rate: 0.9 mL/min

Eluent: 1.0 mmol/L nitric acid, 1.5 mmol/L oxalic acid, 0.75 mmol/L dipicolinic acid, 1% acetone



5.9 Determination of cations in beer

Column: Metrosep C 6 - 150/4.0

Sample preparation: MiDT 1:10 (Metrohm inline Dilution Technique), Inline Ultrafiltration

Detection: Conductivity

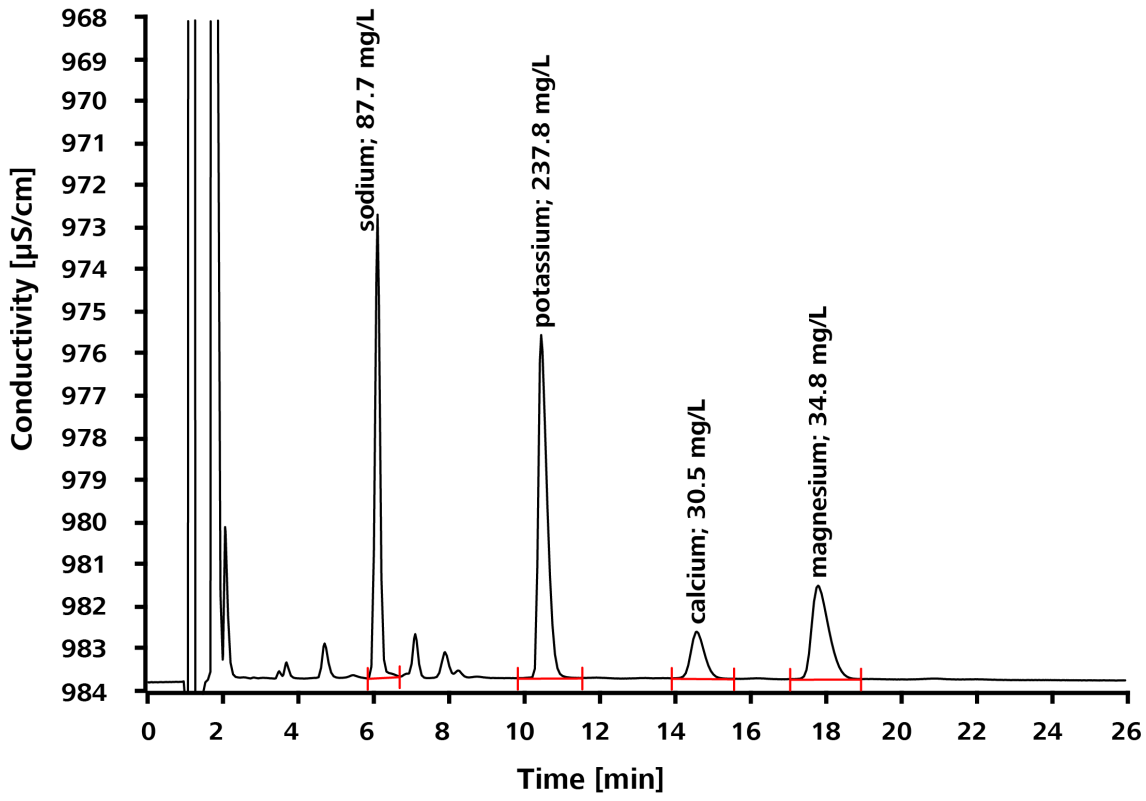
Suppression: –

Temperature: 35 °C

Loop: 20 µL

Flow rate: 0.9 mL/min

Eluent: 2.3 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid



5.10 Determination of cations in solar cell electrolytes

Column: Metrosep C 6 - 150/4.0

Sample preparation:
1. Add 0.5 g sample into a 10 mL volumetric flask.
2. Fill it to the mark with ultrapure water.
3. Treat in ultrasonic bath for 3 minutes.
4. Filter with a 0.2 µm particle filter.

Detection: Conductivity

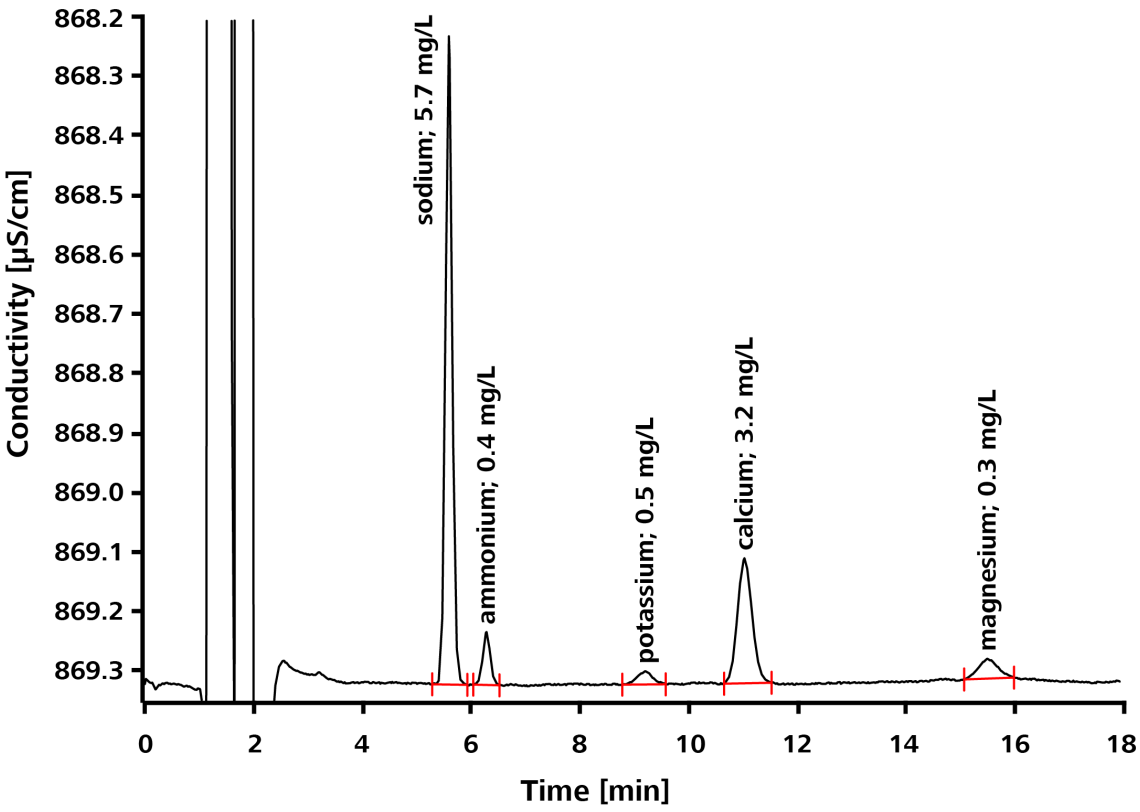
Suppression: –

Temperature: 25 °C

Loop: 50 µL

Flow rate: 0.9 mL/min

Eluent: 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid, 10% acetonitrile



5.11 Determination of sodium, calcium and magnesium in an infusion solution

Column: Metrosep C 6 - 250/4.0

Sample preparation: Dilution 1:10

Detection: Conductivity

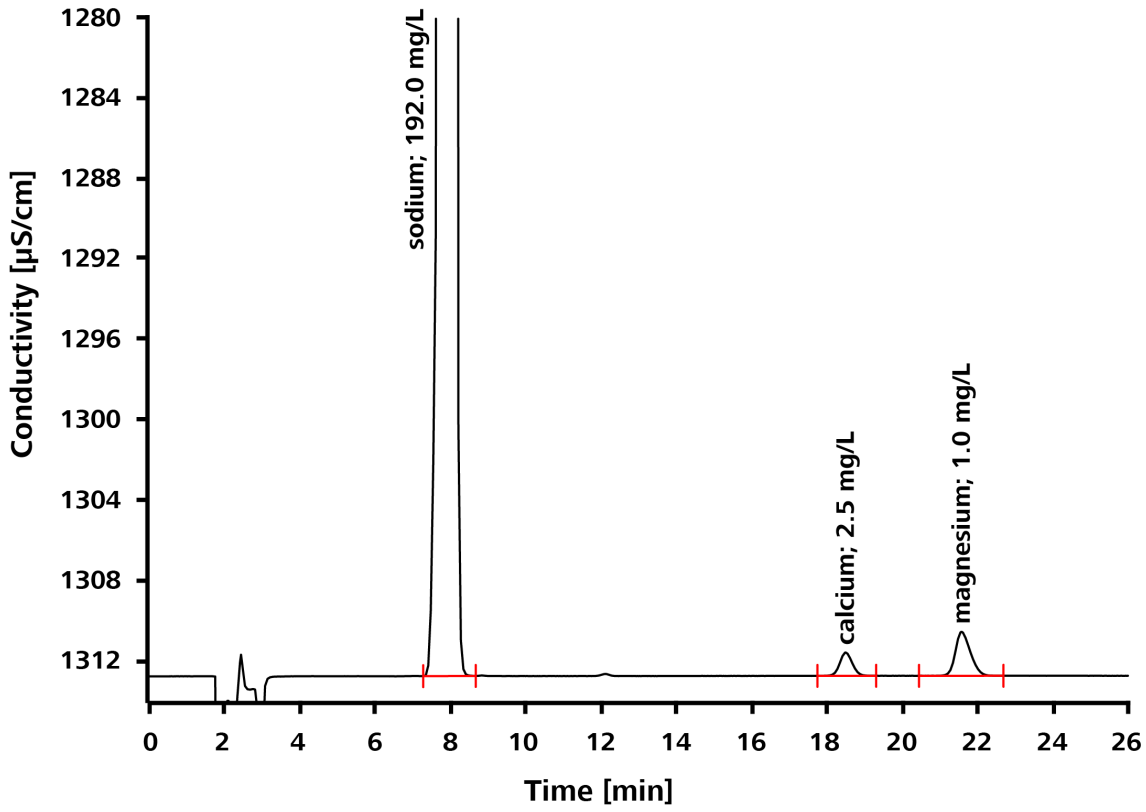
Suppression: –

Temperature: 40 °C

Loop: 10 µL

Flow rate: 0.9 mL/min

Eluent: 4.0 mmol/L nitric acid, 0.5 mmol/L dipicolinic acid



5.12 Determination of standard cations with a citric acid eluent

Column: Metrosep C 6 - 150/2.0

Sample preparation: –

Detection: Conductivity

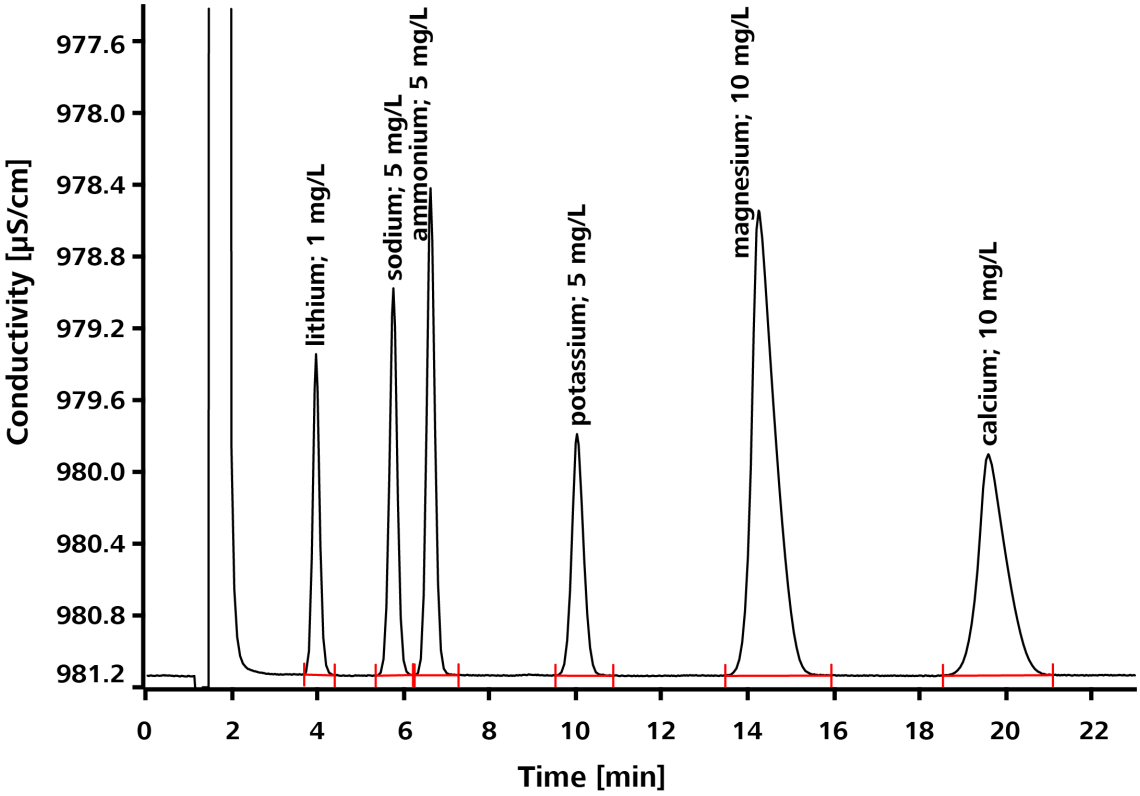
Suppression: –

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.25 mL/min

Eluent: 16 mmol/L citric acid



5.13 Determination of standard cations with an oxalic acid eluent

Column: Metrosep C 6 - 150/2.0

Sample preparation: –

Detection: Conductivity

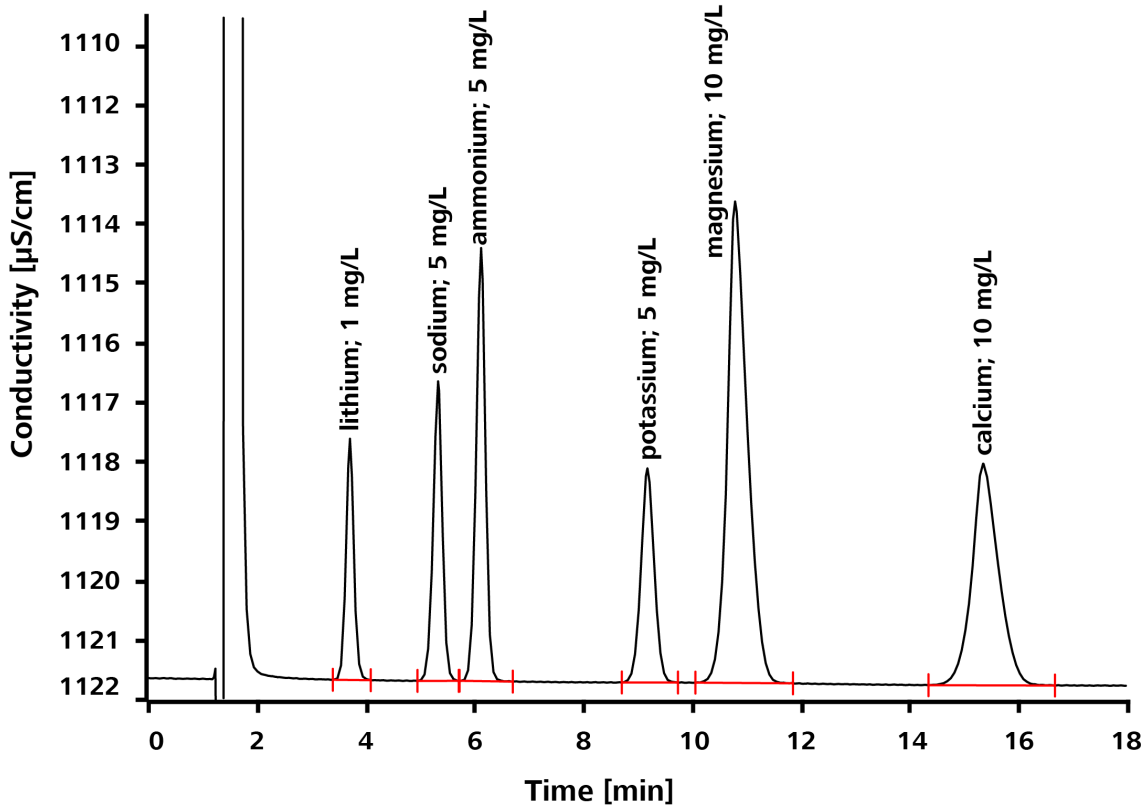
Suppression: –

Temperature: 30 °C

Loop: 20 µL

Flow rate: 0.25 mL/min

Eluent: 4 mmol/L oxalic acid



5.14 Fast determination of standard cations in raw water

Column: Metrosep C 6 - 250/2.0

Sample preparation: –

Detection: Conductivity

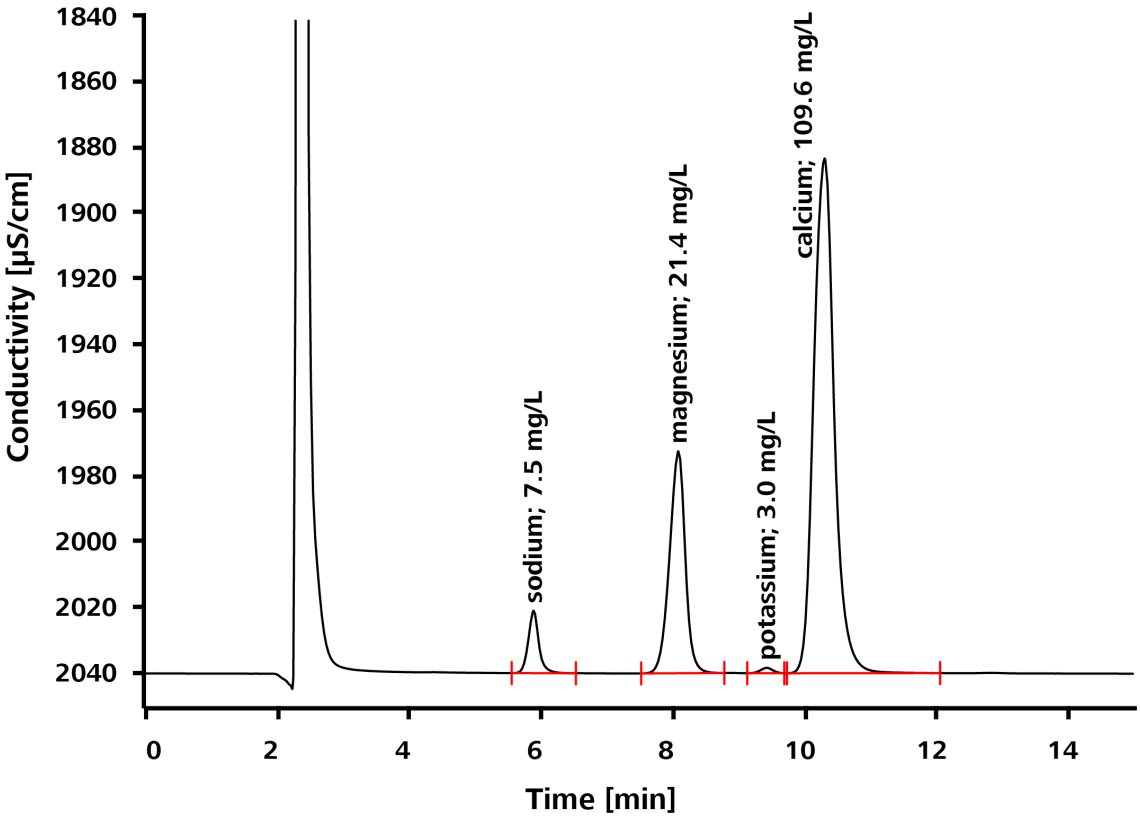
Suppression: –

Temperature: 30 °C

Loop: 10 µL

Flow rate: 0.25 mL/min

Eluent: 6.75 mmol/L nitric acid



5.15 Determination of standard cations in tap water

Column: Metrosep C 6 - 150/2.0

Sample preparation: –

Detection: Conductivity

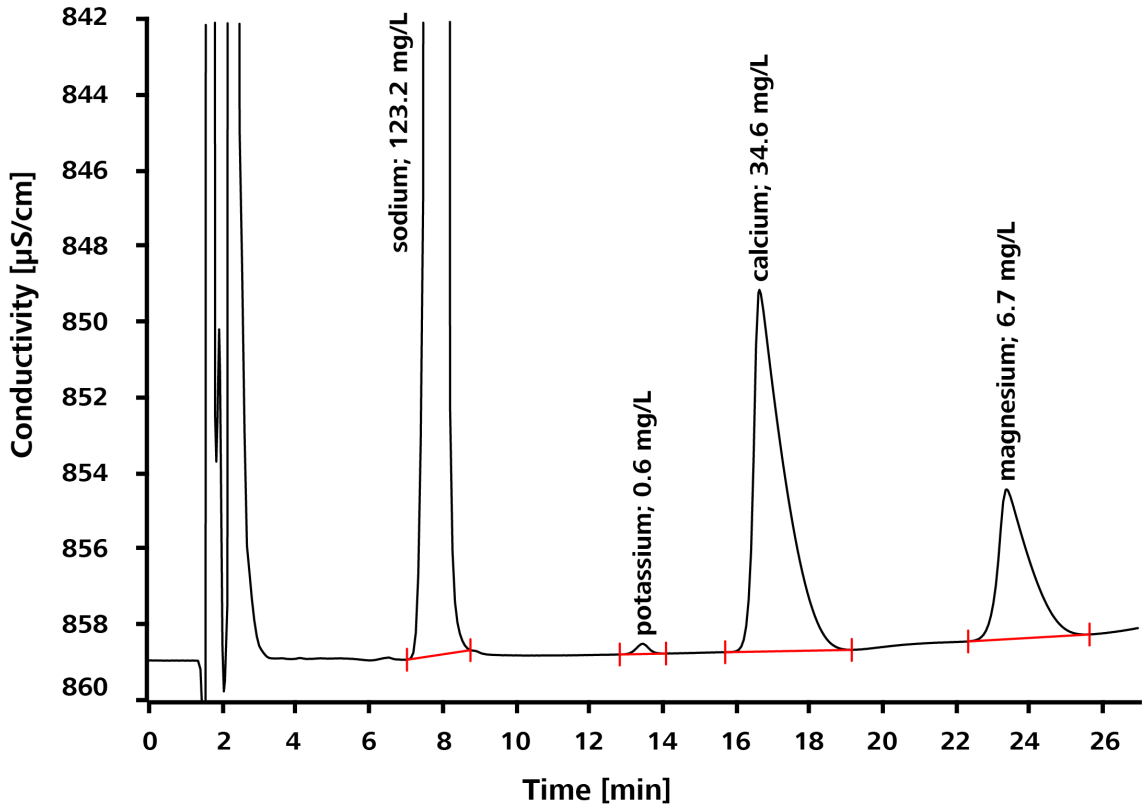
Suppression: –

Temperature: 30 °C

Loop: 5 µL

Flow rate: 0.25 mL/min

Eluent: 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid



The sodium concentration is increased because this drinking water has been treated in an ion exchanger.

5.16 Determination of standard cations in snow

Column: Metrosep C 6 - 100/2.0

Sample preparation: 0.20 µm particle filter

Detection: Conductivity

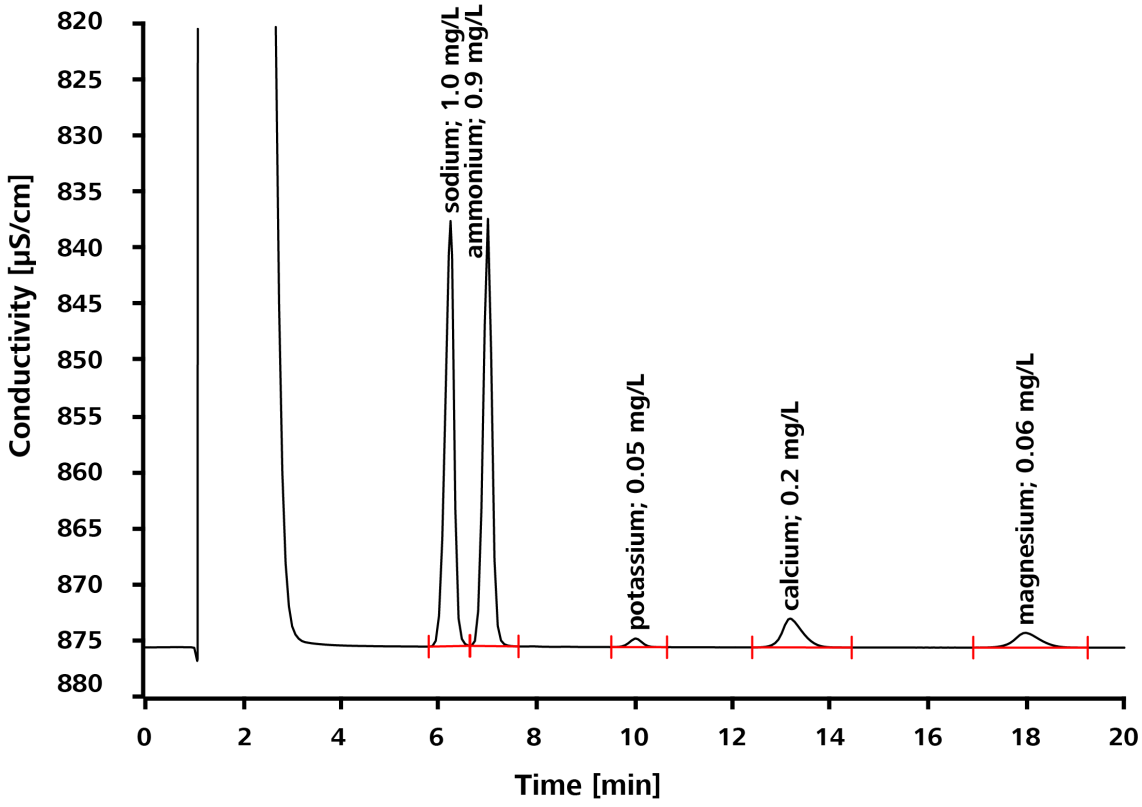
Suppression: –

Temperature: 30 °C

Loop: 250 µL

Flow rate: 0.25 mL/min

Eluent: 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid



5.17 Determination of standard cations in roadside snow

Column: Metrosep C 6 - 250/2.0

Sample preparation: 0.20 µm particle filter, dilution 1:25

Detection: Conductivity

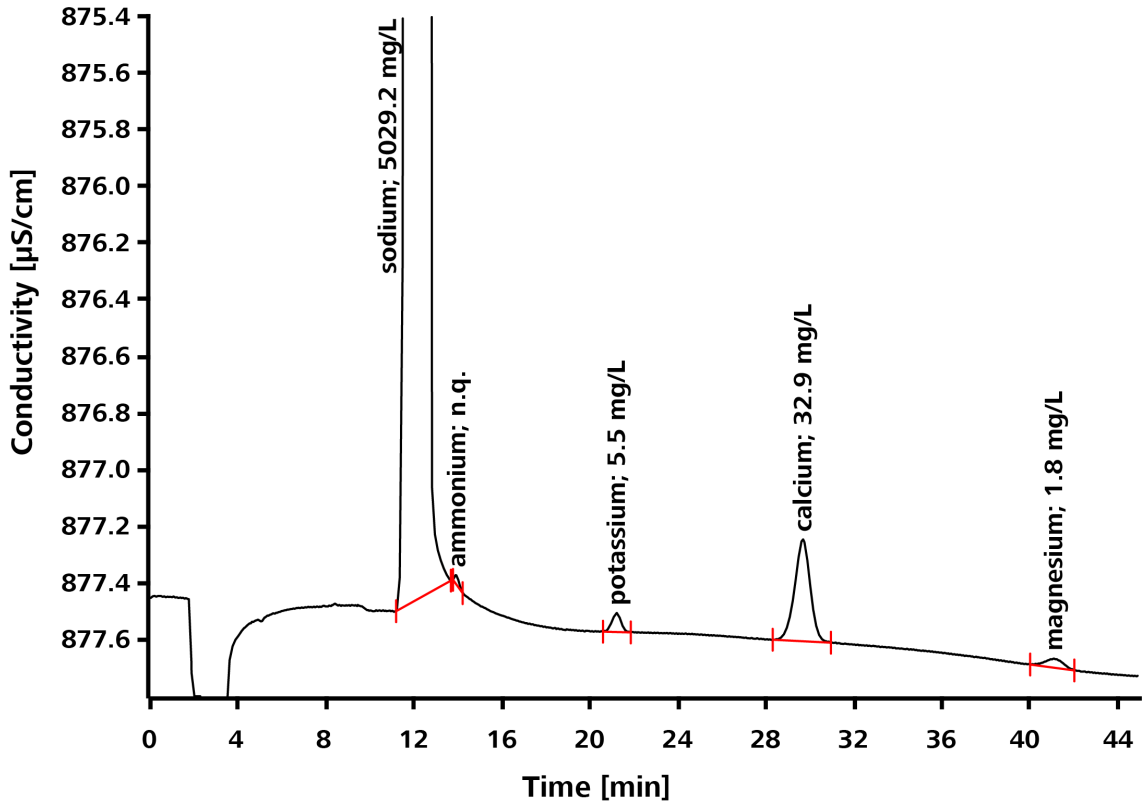
Suppression: –

Temperature: 30 °C

Loop: 5 µL

Flow rate: 0.25 mL/min

Eluent: 1.7 mmol/L nitric acid, 1.7 mmol/L dipicolinic acid



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

1 Disconnecting the separation column from the IC system

Disconnect the separation column outlet from the detector inlet.

2 Regenerating the separation column

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

- Contamination with organic components *(see table 4, page 46)*.
- Contamination with inorganic components *(see table 5, page 46)*

Table 4 Contamination with organic components

	Rinse with	Duration [min]	Flow rate [mL/min]	
			2-mm columns	4-mm columns
1	Ultrapure water, opposite to flow direction	60	0.25	0.9
2	Acetonitrile/water (40/60), opposite to flow direction	60	0.25	0.9
3	Ultrapure water	60	0.25	0.9

Table 5 Contamination with inorganic components

	Rinse with	Duration [min]	Flow rate [mL/min]	
			2-mm columns	4-mm columns
1	10 mmol/L nitric acid and 4 mmol/L dipicolinic acid, opposite to flow direction	60	0.25	0.9

6.2 Decreasing resolution / peak shapes

Problem

The resolution of peaks deteriorates or peak shapes are asymmetrical.

Causes and prevention

Causes	Prevention/correction
The separation column has been overloaded.	The separation column can be overloaded by factors such as a high salt content in the sample matrix. ▪ Dilute the sample. ▪ Inject less sample.
There are dead volumes in the IC system.	▪ Check that all of the capillaries have a diameter of ≤ 0.25 mm (6.1831.010). If not, replace the larger capillaries. ▪ Check that all of the capillaries have been installed correctly. The step-by-step installation process is shown in the IC Maintenance multimedia guide.

6.3 Unstable retention times

Problem

The retention times are unstable.

Causes and prevention

Causes	Prevention/correction
Air bubbles in the eluent	Air bubbles make the eluent flow rate unstable. Backpressure is one indicator of an unstable flow rate. Backpressure should remain stable within ±0.1 MPa. ▪ Deaerate the high-pressure pump. ▪ Use an eluent degasser.

6.4 Unknown peaks

Problem

The chromatogram contains wide, unknown peaks.

Causes and prevention

Causes	Prevention/correction
Analytes eluting late	Some wider, unknown peaks can be the result of sample components eluting late. In these cases, this is the result of the previous injection. ▪ Extend the chromatogram duration.

6.5 Increasing backpressure

Problem

The backpressure increases.

Causes and prevention

Causes	Prevention/correction
Particles on the guard column	▪ Replace the guard column.
Particles on the separation column	Rinse the separation column in the direction opposite to the flow direction. ▪ Hold the column outlet in a beaker. ▪ Rinse the separation column for approximately 1 h. ▪ Install the separation column back in the flow direction.

Causes	Prevention/correction
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 C-119 Bethanechol chloride and calcium in tablets
- Application Note C-142 Separation of the standard cations on the high-capacity Metrosep C 6 separation columns
- Application Note C-143 Ammonia in addition to standard cations in maritime pore water
- Application Note M-007 Determination of urea in ultrapure water using IC-MS
- Application Note C-146 Bethanechol and HPTA (2-hydroxy-propyl-trimethyl ammonium) besides sodium and calcium (Metrosep C 6 - 250/4.0)
- Application Note C-144 Variable Inline Preconcentration including matrix elimination for trace cation determination (MiPCT-ME)
- Application Note C-154 Fast IC: cations in drinking water on a high capacity column in eleven minutes
- Application Note C-156 Temperature dependency of the cation separation on the Metrosep C 6 - 150/4.0 column
- Application Note C-158 Column stability of the Metrosep C 6 - 250/4.0 with Inline Ultrafiltration and Inline Eluent Preparation
- Application Note C-161 Trimethylamine N-oxide and biogenic amines in addition to standard cations in white wine
- Application Note C-164 Amine analysis in gas scrubber solutions from refineries with direct conductivity detection
- Application Note C-163 Cations in brine with minimal dilution and sub-µL injection
- Application Note C-155 Selectivity of the high-capacity Metrosep C 6 - 150/4.0 cation column
- Application Note C-172 Cations in snow collected on an open field
- Application Note C-173 Cations in snow collected at a roadside
- Application Note C-174 Rapid determination of cations in drinking water on a microbore column
- Ismail, L.; Rifai, A.; Ferronato, C.; Fine, L.; Jaber, F.; Chovelon, J.M., Towards a better understanding of the reactive species involved in the photocatalytic degradation of sulfaclozine, Applied Catalysis B: Environmental, 2016, Issue 185, p.88-99
- Lopes Pereira, E.; Brazil de Paiva, T.C:; Teixeira da Silva, F., Physico-chemical and Eco-toxicological Characterization of Slaughterhouse Wastewater Resulting from Green Line Slaughter, Water, Air, & Soil Pollution, 2016, Issue 227, Volume 199, Influences of Asian Dust, Haze, and Mist Events on Chemical Compositions of Fine Particulate Matters at Go-san Site, Jeju Island in 2014

- Song, J.M.; Bu, J.O.; Yang, S.H.; Lee, J.Y.; Kim, W.H.; Kang, C.H., Journal of the Korea Air Pollution, 2016, Volume 32, Issue 1, p. 67-81
- Sgier, L.; Freimann, R.; Zupanic, A.; Kroll, A., Flow cytometry combined with viSNE for the analysis of microbial biofilms and detection of microplastics, 2016, Volume 7, Issue 11587
- Kroll, A.; Matzke, M.; Rybicki, M.; Obert-Rauser, P.; Burkart, C.; Jurkscha, K.; Verweij, R.; Sgier, L.; Jungmann, D.; Backhaus, T.; Svendsen, C., Mixed messages from benthic microbial communities exposed to nanoparticulate and ionic silver: 3D structure picks up nano-specific effects, while EPS and traditional endpoints indicate a concentration-dependent impact of silver ions, Environmental Science and Pollution Research, 2016, Volume 23, Issue 5, p. 4218-4234
- Vaiopoulou, E.; Provijn, T.; Prévoteau, A.; Pikaar, I.; Rabaey, K., Electrochemical sulfide removal and caustic recovery from spent caustic streams, Water Research, 2016, Volume 92, p. 29-43
- De Vrieze, J.; Coma, M.; Debeuckelaere, M.; Van der Meeren, P.; Rabaey, K., High salinity in molasses wastewaters shifts anaerobic digestion to carboxylate production, Water Research, 2016, Volume 98, p. 293-301
- Deusdara, K.R.L.; Forti, M.C.; Borma, l.S.; Menezes, R.S.C.; Lima, J.R.S.; Ometto, J.P.H.B., Rainwater chemistry and bulk atmospheric deposition in a tropical semiarid ecosystem: the Brazilian Caatinga, Journal of Atmospheric Chemistry, 2017, Volume 74, Issue 1, p. 71-85
- Vilanova, J.A., Novel Catalytic Materials and Strategies for Hydrogen Production and Hydrogenation Reactions based on PEM Electrolysis, Thesis, UNIVERSITAT ROVIRA I VIRGILI, Tarragona, ES, 2014
- Gildemyn, S.; Verbeeck, K.; Jansen, R.; Rabaey, K., The type of ion selective membrane determines stability and production levels of microbial electrosynthesis, Bioresource Technology, 2017, Volume 224, p. 358-364
- Scoma, A.; Coma, M.; Kerckhof, F.M.; Boon, N.; Rabaey, K, Efficient molasses fermentation under high salinity by inocula of marine and terrestrial origin, Biotechnology for Biofuels, 2017, Volume 10, Issue 23
- Monograph: Analysis of water samples and water constituents with Metrohm instruments, p. 116 ff (8.038.5003)
- Column catalog, 8.000.5194

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