Column manual

Metrosep C Supp 2 (6.01053.XX0)

Manual

8.0107.8007EN / 2019-06-24

Metrohm

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Technical Communication
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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.

Table of contents

1 General information

This cation separation column is particularly suitable for the determination of cations, transition metals and amines with sequential chemical suppression. Detection limits in the lowest µg/L range are achieved due to low baseline noise after sequential suppression.

1.1 Ordering information

Table 1 4-mm columns

Order number	Designation
6.01053.410	Metrosep C Supp 2 – 100/4.0
6.01053.420	Metrosep C Supp 2 - 150/4.0
6.01053.430	Metrosep C Supp 2 - 250/4.0

Table 2 Guard column

Order number	Designation
6.01053.500	Metrosep C Supp 2 Guard/4.0

1.2 Technical specifications

Column material Polystyrene/divinylbenzene copolymer, with carboxyl groups

Particle size 5 µm

Dimensions

Order number	Measurements
6.01053.410	100 x 4.0 mm
6.01053.420	150 x 4.0 mm
6.01053.430	250 x 4.0 mm

pH range eluent 0 to 12

ph range sample 0 to 14

Temperature range 10 to 60 °C

Recommended standard temperature 40 °C

Maximum pressure

4 mm	25 MPa (250 bar)

Flow rate

Order number	Recommended flow rate	Maximum flow rate
6.01053.410	1.0 mL/min	3.8 mL/min
6.01053.420	1.0 mL/min	3.1 mL/min
6.01053.430	1.0 mL/min	2.0 mL/min

Standard eluent

5.0 mmol/L of nitric acid, 50 µg/L of rubidium (made of $RbNO_3$-salt)

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

Preparation

Rinse the column with eluent for 3 h.

Storage

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

Typical pressure

For columns with a guard column under standard conditions with sequential suppression (MSM-HC C and MCS):

Order number	Typical pressure
6.01053.410	7 ± 2 MPa
6.01053.420	10 ± 2 MPa
6.01053.430	14 ± 2 MPa

Column housing

PEEK

Application

Determination of monovalent and divalent cations with suppression and conductivity detection.

2 Key aspects of working with separation columns

Storage

Once the backpressure in your ion chromatograph has dissipated, remove the column at ambient temperature. Seal the column at both ends using the original stoppers (6.2744.060). Store the column in 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. We recommend 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% acetonitrile or acetone to the eluent. This is only possible if you are *not using membrane suppressors*. 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 a 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. 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 your sample automatically with one of the Metrohm Inline Sample Preparation techniques (MISP).

Sample preparation cartridges

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

Pulsation absorber

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. 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 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 3 to 4 times during the service life of the analytical column.

Water quality

Ion chromatography involves mainly aqueous media. 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 specific resistance greater than 18.2 MΩ·cm and should be free of particles. Therefore, we recommend filtering the water using a 0.45-µm filter and treating it with UV light. Modern ultrapure water systems for laboratory use ensure this level of water quality (Type I).

3 Eluent production

We recommend 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
- Rubidium nitrate, $RbNO_3$, 99.7%
  Sigma-Aldrich order number: 289299
- 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 nitric acid and 50 µg/L of rubidium:

Producing 2 L of standard eluent

1
- Pre-rinse the eluent bottle with ultrapure water several times.
- Set out 1.9 L of ultrapure water.

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

- Degas the ultrapure water for the eluent for 5-10 minutes using a vacuum pump. This prevents problems with air bubbles in the high-pressure pump.

3 Producing a stock solution of 1 g/L of rubidium (=1000 ppm):

- Weigh 86.27 mg of rubidium nitrate salt
- Dissolve in 50 mL of ultrapure water

4 Producing eluents:

- Pipette 100 µL of the rubidium stock solution into the eluent bottle.
- Pipette 5 mL of the 2 mol/L nitric acid into the eluent bottle.
- Fill the eluent bottle with ultrapure water to 2 L.

- Stir briefly.

This eluent (5.0 mmol/L of nitric acid, 50 µg/L of rubidium) and sequential suppression can be used to achieve background conductivity of < 0.3 µS/cm. The noise is typically less than 0.1 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

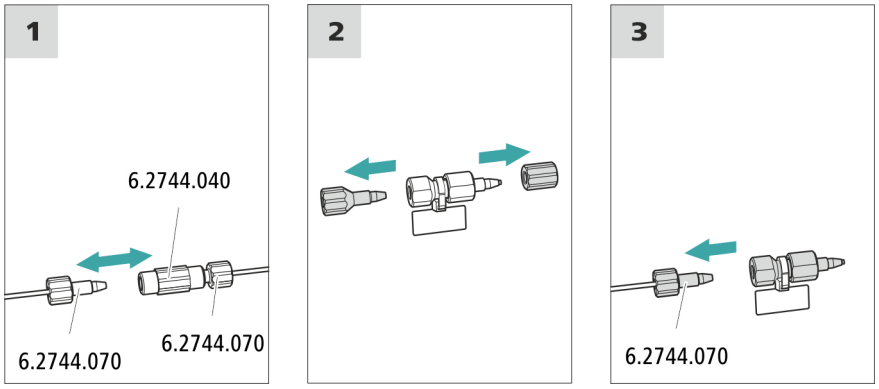
The guard column may not be connected until after the instrument has already been put into operation once . The guard column and the separation column have to be replaced by a coupling (6.2744.040) until then.

Accessories

For this step, you need the following accessories:

- Guard column (suitable for separation column)

Connecting the guard column



1 Removing the coupling

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

2 Preparing the guard column

- Remove the 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.

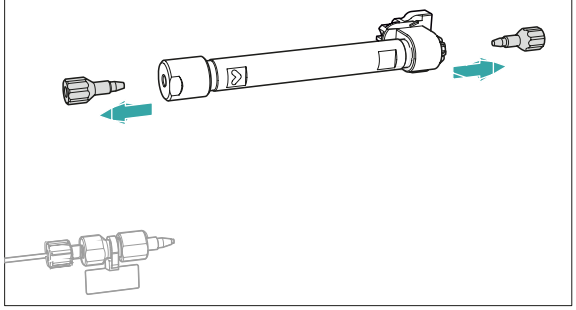
CAUTION

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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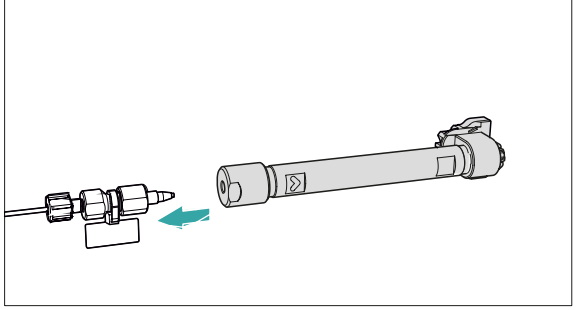

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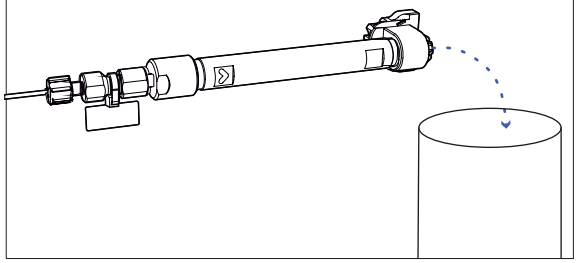

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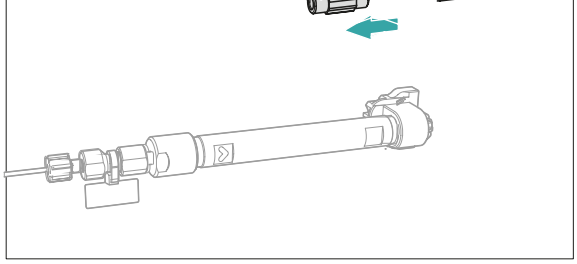

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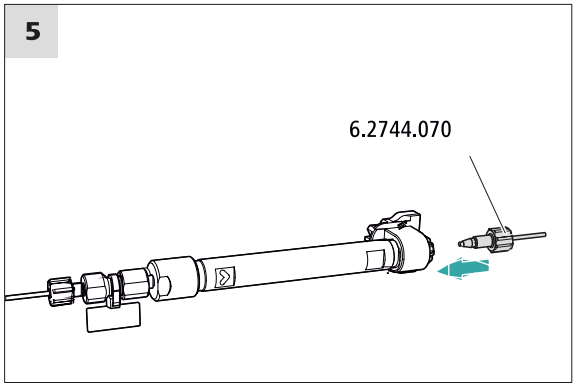

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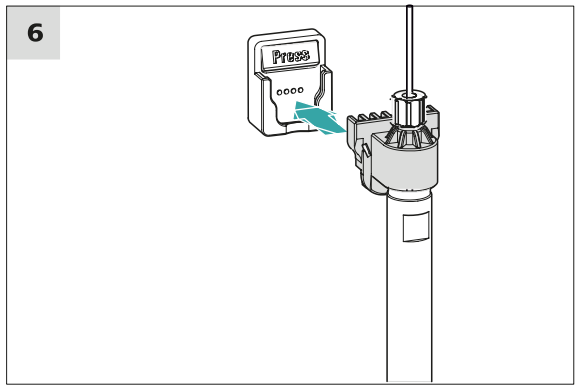


Connecting the separation column

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

Sample preparation: –

Detection: Conductivity

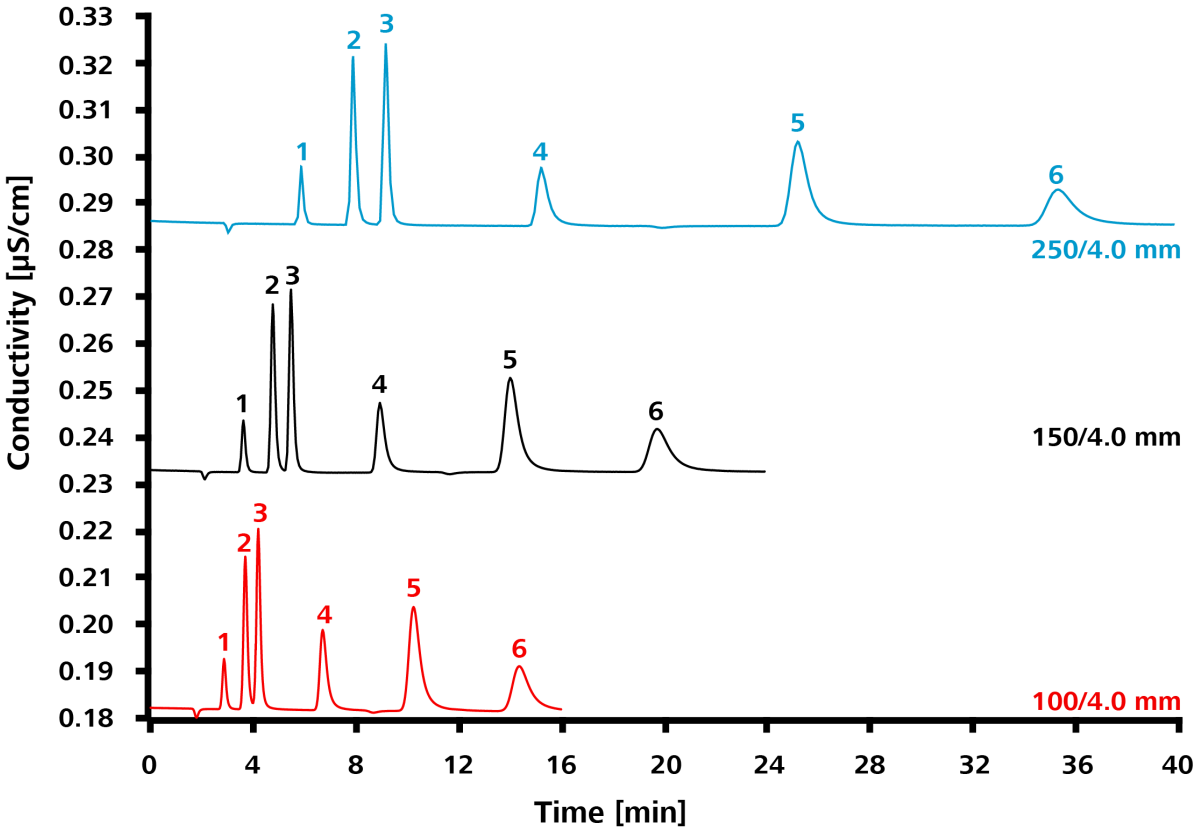
Suppression: Sequential suppression with MSM-HC C and MCS

Temperature: 40 °C

Loop: 20 µL

Flow rate: 1.0 mL/min

Eluent: 5.0 mmol/L of nitric acid and 50 µg/L of rubidium



	Metrosep C Supp 2 – xx0/4.0	mg/L
1	Lithium	0.025
2	Sodium	0.125
3	Ammonium	0.125
4	Potassium	0.250
5	Magnesium	0.250
6	Calcium	0.250

5.2 Variation of the temperature

Column: Metrosep C Supp 2 - 150/4.0

Sample preparation: –

Detection: Conductivity

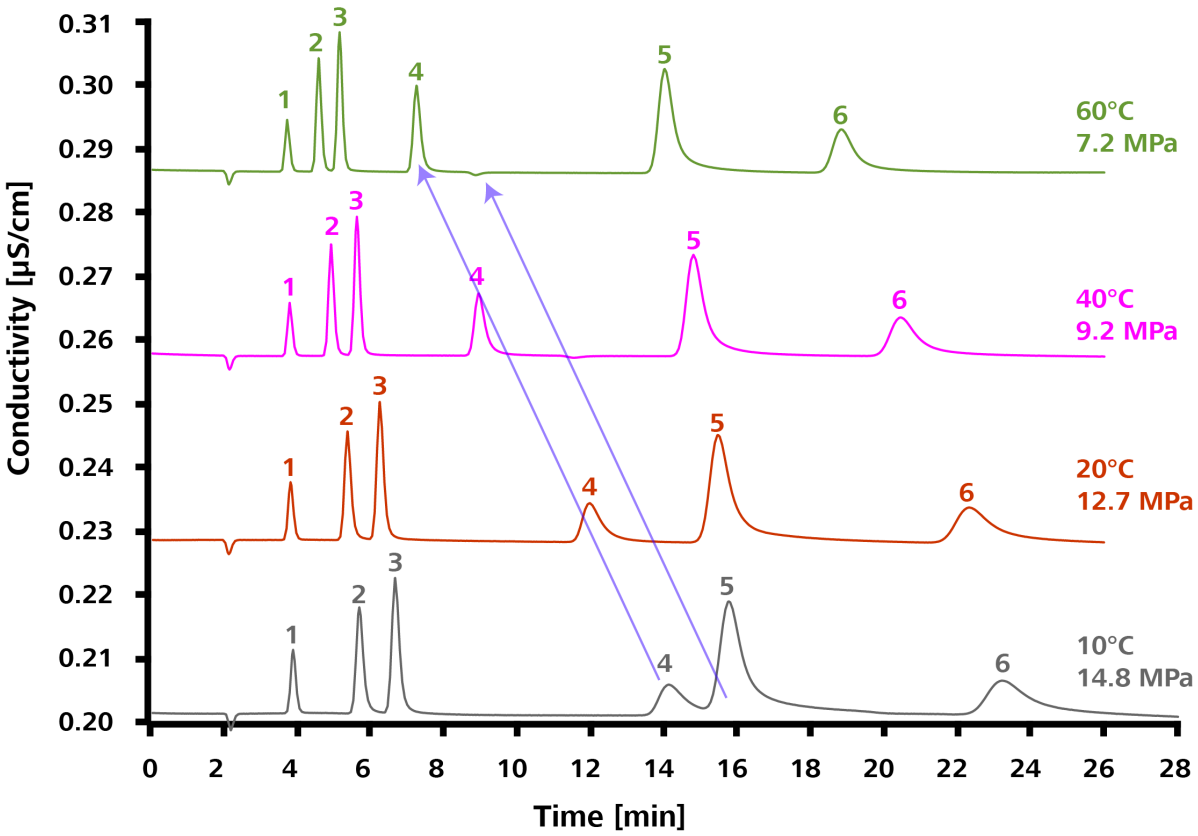
Suppression: Sequential suppression with MSM-HC C and MCS

Temperature: 10 to 60 °C

Loop: 20 µL

Flow rate: 1.0 mL/min

Eluent: 5.0 mmol/L of nitric acid and 50 µg/L of rubidium



	Metrosep C Supp 2 – 150/4.0	mg/L
1	Lithium	0.025
2	Sodium	0.125
3	Ammonium	0.125
4	Potassium	0.250
5	Magnesium	0.250
6	Calcium	0.250

When the temperature increases, the retention times of potassium and rubidium (system peak) decreases disproportionately. The retention times of potassium and rubidium are shortened at the same rate. At 10 °C rubidium and magnesium coelute.

When the temperature increases, the backpressure of the column decreases.

5.3 Variation of the eluent flow rate

Column: Metrosep C Supp 2 - 150/4.0

Sample preparation: –

Detection: Conductivity

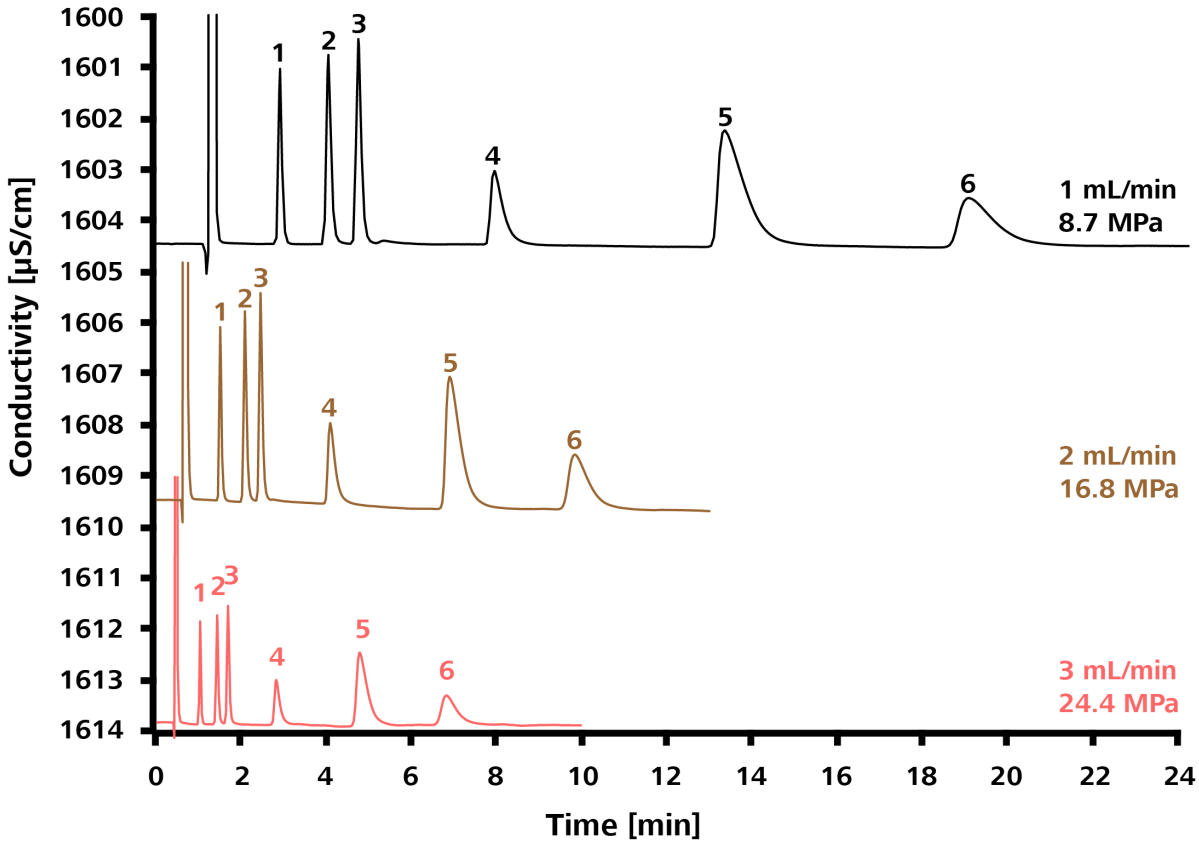
Suppression: –

Temperature: 40 °C

Loop: 20 µL

Flow rate: 1.0 up to 3.0 mL/min

Eluent: 5.0 mmol/L of nitric acid



	Metrosep C Supp 2 – 150/4.0	mg/L
1	Lithium	1

	Metrosep C Supp 2 – 150/4.0	mg/L
2	Sodium	5
3	Ammonium	5
4	Potassium	10
5	Magnesium	10
6	Calcium	10

All cations elute faster when the flow rate is higher.

5.4 Variation of the eluent strength

Column: Metrosep C Supp 2 - 250/4.0

Sample preparation: –

Detection: Conductivity

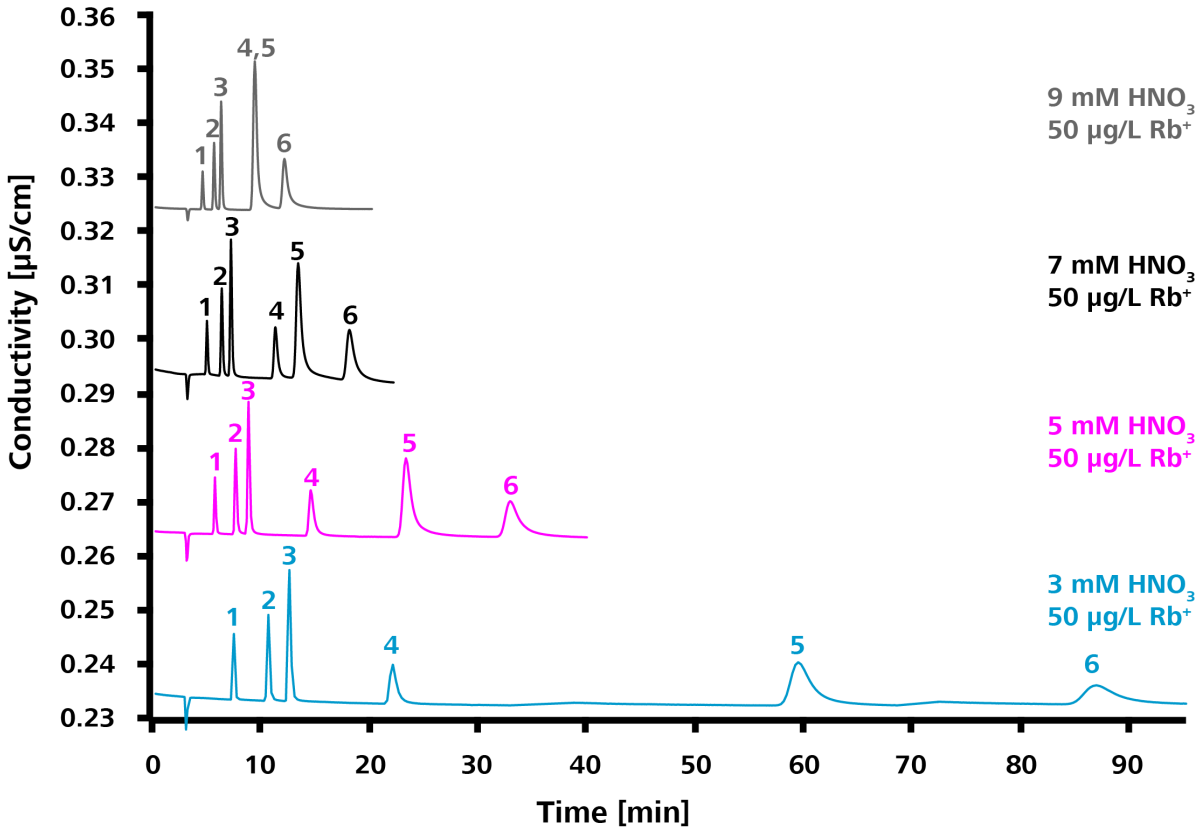
Suppression: Sequential suppression with MSM-HC C and MCS

Temperature: 40 °C

Loop: 20 µL

Flow rate: 1.0 mL/min

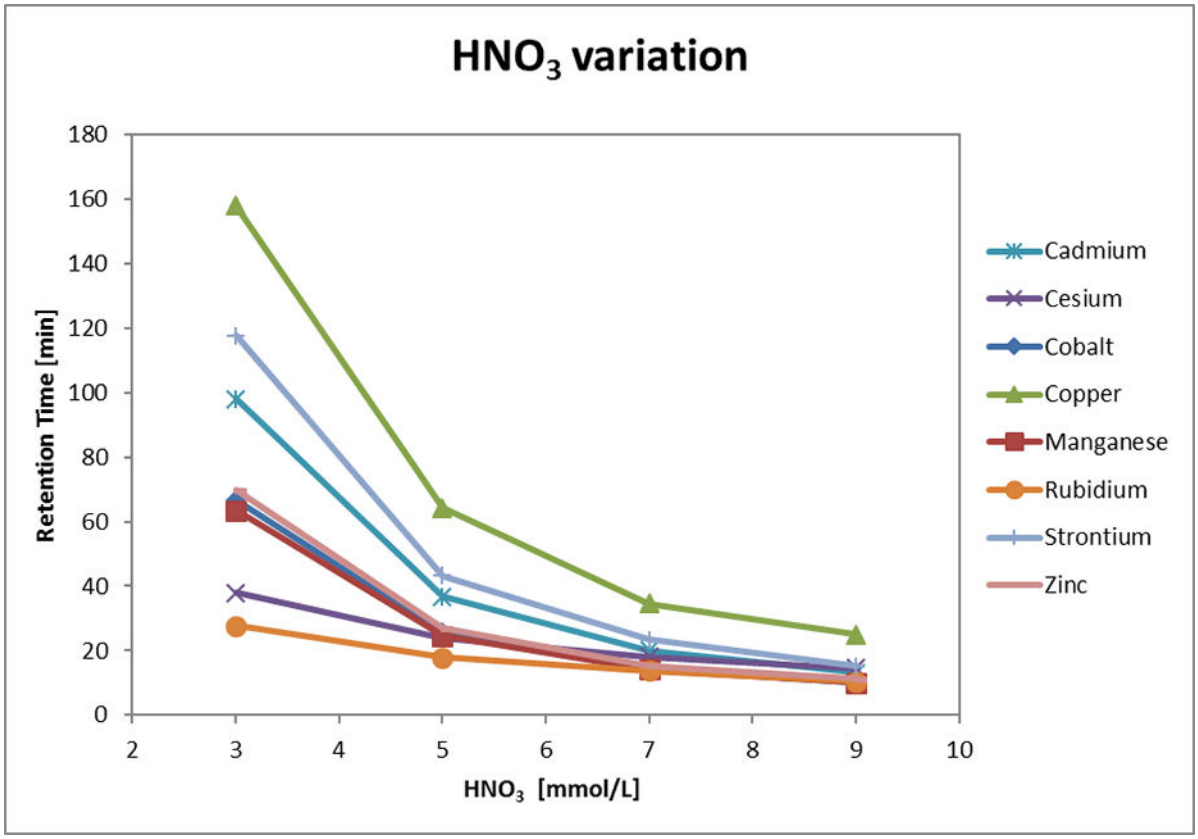
Eluent: A) 3.0 mmol/L of nitric acid and 50 µg/L of rubidium
B) 5.0 mmol/L of nitric acid and 50 µg/L of rubidium
C) 7.0 mmol/L of nitric acid and 50 µg/L of rubidium
D) 9.0 mmol/L of nitric acid and 50 µg/L of rubidium



	Metrosep C Supp 2 – 250/4.0	mg/L
1	Lithium	0.025
2	Sodium	0.125
3	Ammonium	0.125
4	Potassium	0.250
5	Magnesium	0.250
6	Calcium	0.250

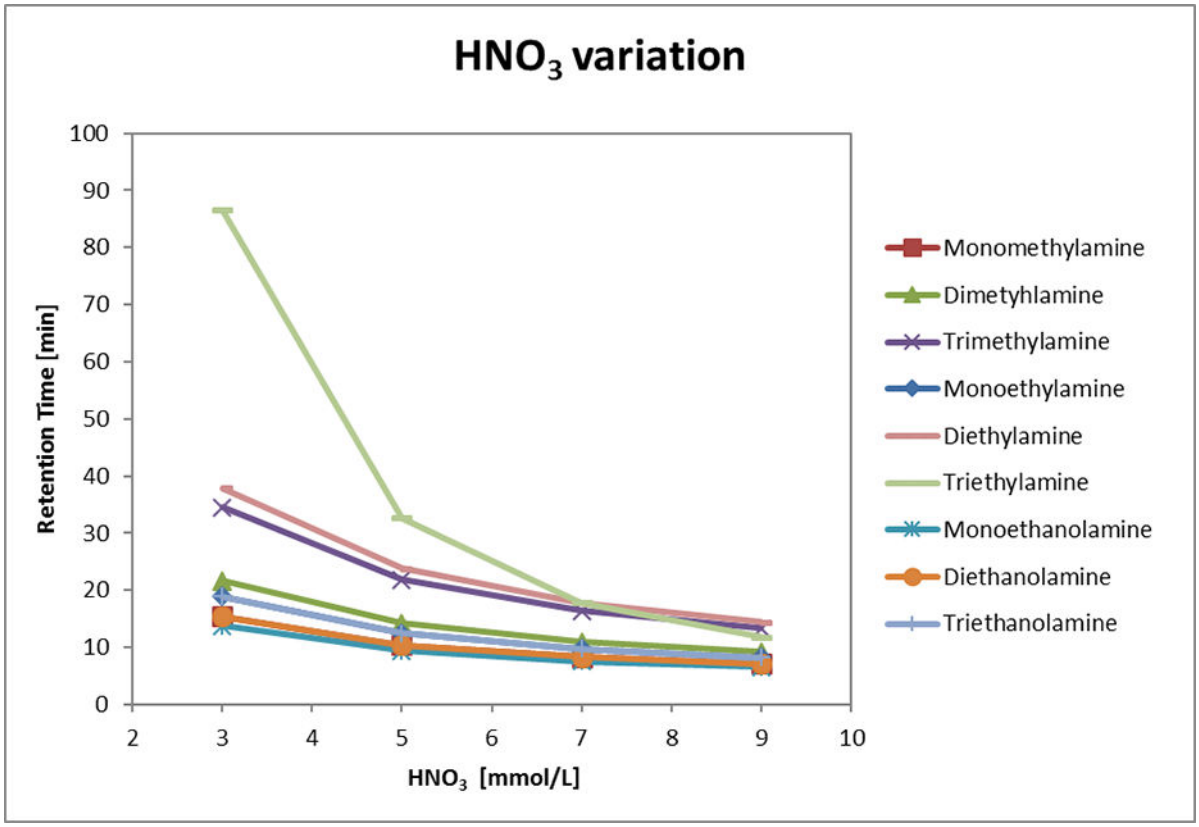
The retention times become shorter with increasing HNO_3 concentration. Especially polyvalent cations such as magnesium and calcium are accelerated disproportionately to the monovalent cations. At 9 mmol/L of nitric acid, magnesium and potassium have the same retention time.

Transition metals



With transition metals, the polyvalent cations are accelerated faster than the monovalent cations such as rubidium and cesium. Copper elutes as an extremely wide peak. At 5.0 mmol/L of nitric acid, the copper peak is approximately 20 minutes wide and therefore insensitive.

Amines



The retention times of the amines continuously decrease with increasing eluent strength. For triethylamine, the decrease is strongest. At 9 mmol/L of nitric acid, triethylamine elutes before diethylamine and trimethylamine.

5.5 Variation with organic modifier

5.5.1 Variation of the acetone concentration

Column: Metrosep C Supp 2 - 250/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM-HC C and MCS

Temperature: 40 °C

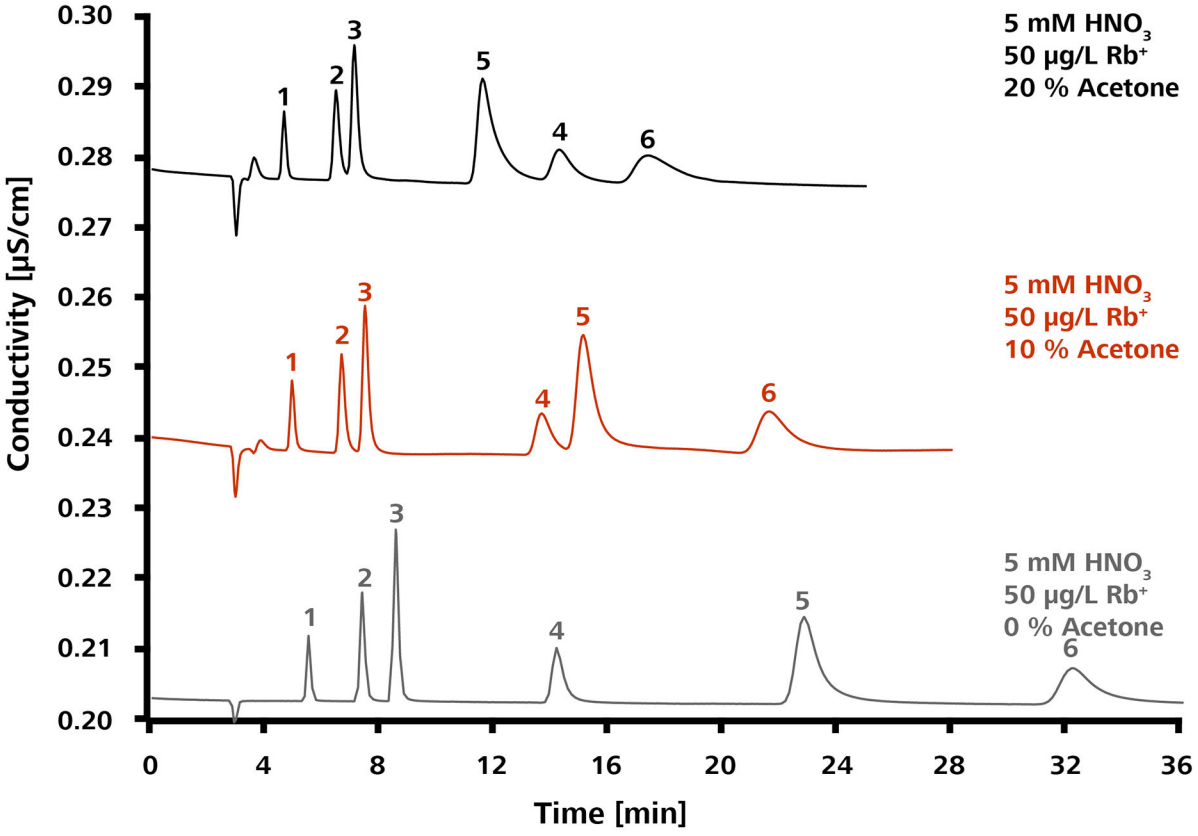
Loop: 20 µL

Flow rate: 1.0 mL/min

Eluent: A) 5.0 mmol/L of nitric acid, 50 µg/L of rubidium and 0% acetone

B) 5.0 mmol/L of nitric acid, 50 µg/L of rubidium and 10% acetone

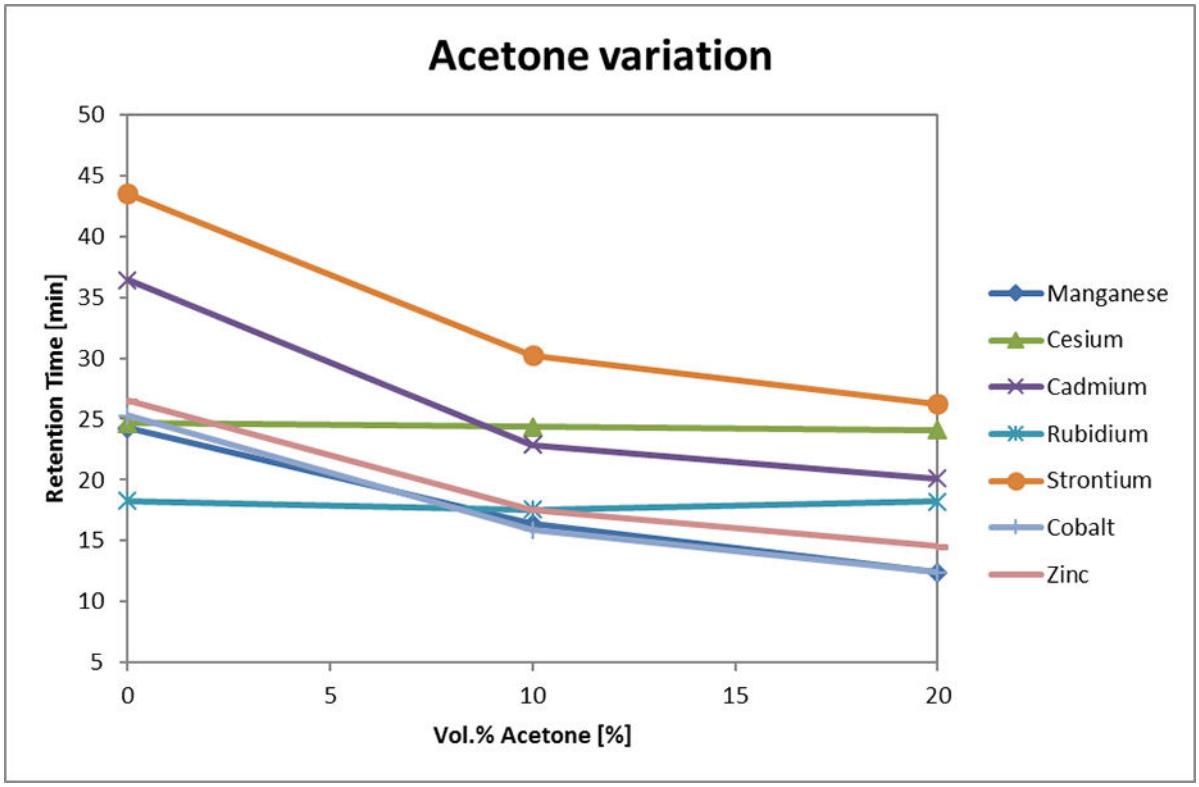
C) 5.0 mmol/L of nitric acid, 50 µg/L of rubidium and 20% acetone



	Metrosep C Supp 2 – 250/4.0	mg/L
1	Lithium	0.025
2	Sodium	0.125
3	Ammonium	0.125
4	Potassium	0.250
5	Magnesium	0.250
6	Calcium	0.250

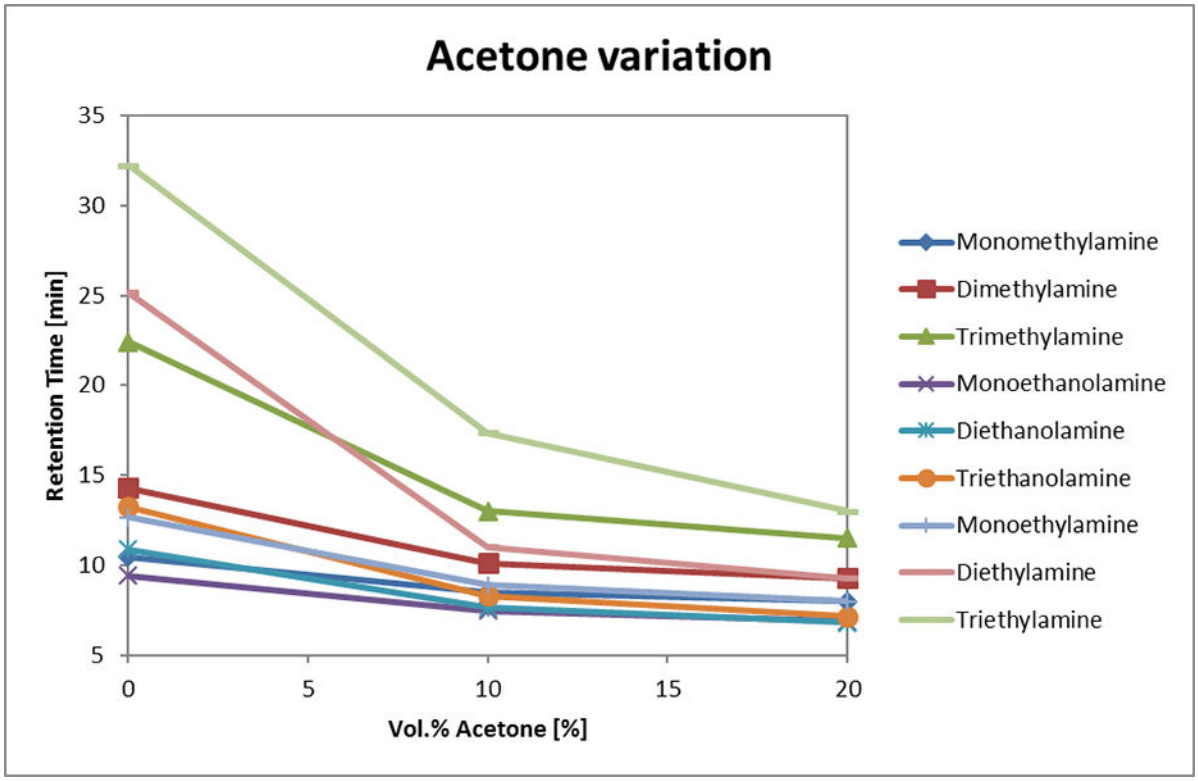
By adding acetone to the eluent, the retention time of all cations is shortened. The effect is more visible in polyvalent cations such as magnesium and calcium than in monovalent cations. From 20% acetone in the eluent, magnesium elutes before potassium. A higher proportion of acetone in the eluent leads to a deterioration of the peak form.

Transition metals



Acetone has less impact on the retention time of rubidium and cesium. The retention times of other transition metals are shortened when the acetone concentration increases. A higher proportion of acetone in the eluent leads to a deterioration of the peak form.

Amines



The retention times of all the amines decrease with increasing acetone concentration. Diethylamine and trimethylamine as well as triethanolamine and monomethylamine change the retention order. A higher proportion of acetone in the eluent leads to a deterioration of the peak form.

5.5.2 Variation of the acetonitrile concentration

Column: Metrosep C Supp 2 - 250/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM-HC C and MCS

Temperature: 40 °C

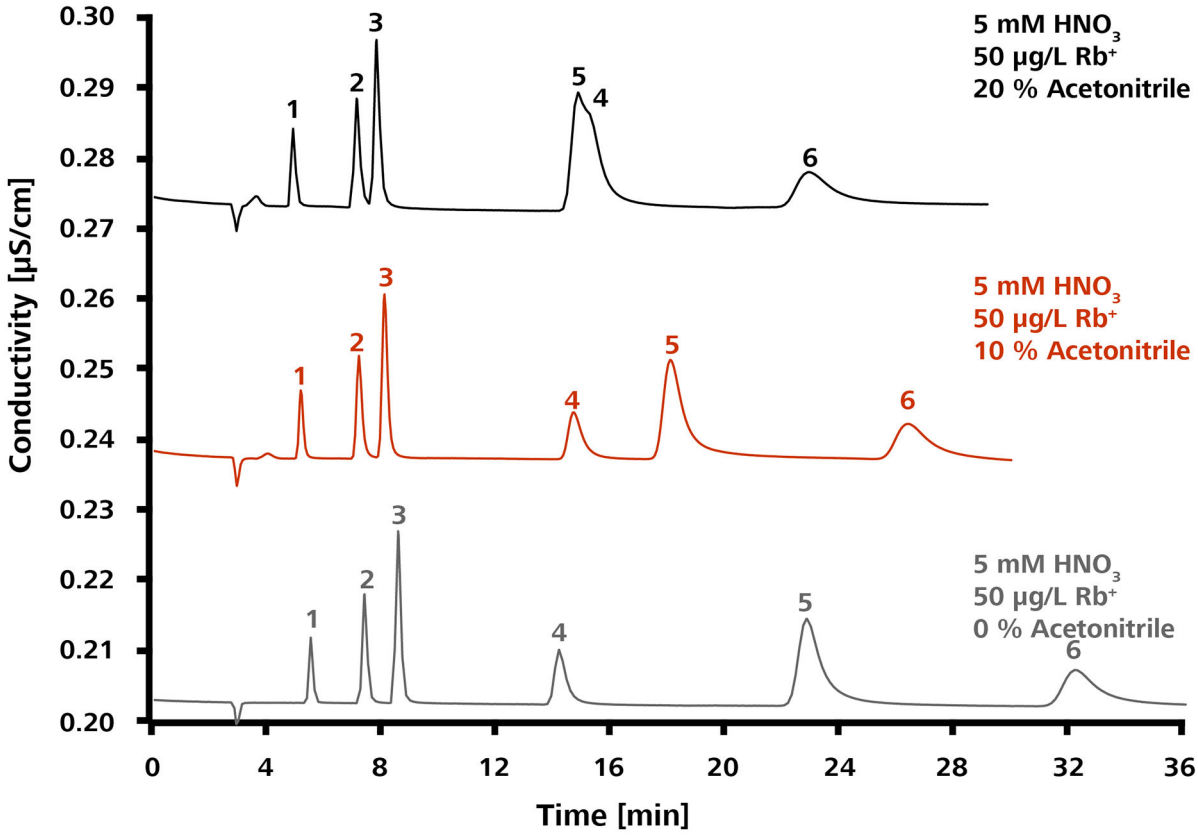
Loop: 20 µL

Flow rate: 1.0 mL/min

Eluent: A) 5.0 mmol/L of nitric acid, 50 µg/L of rubidium and 0 % acetonitrile

B) 5.0 mmol/L of nitric acid, 50 µg/L of rubidium and 10 % acetonitrile

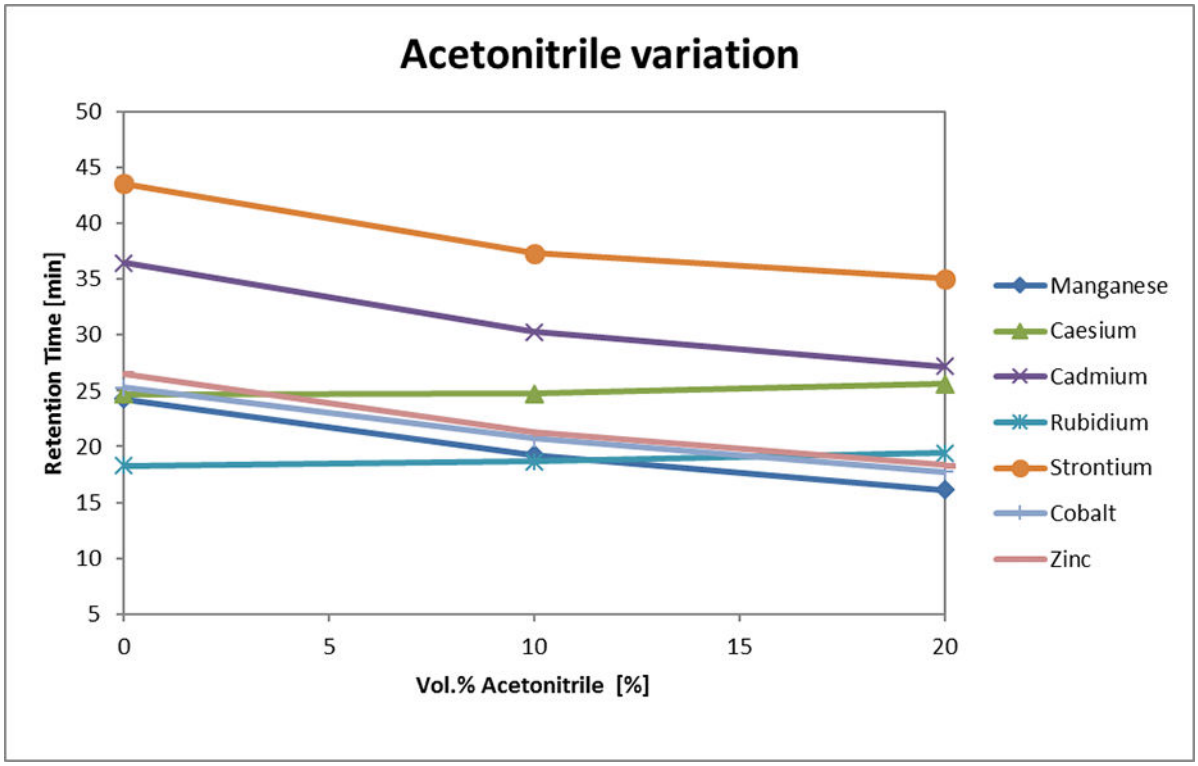
C) 5.0 mmol/L of nitric acid, 50 µg/L of rubidium and 20 % acetonitrile



	Metrosep C Supp 2 – 250/4.0	mg/L
1	Lithium	0.025
2	Sodium	0.125
3	Ammonium	0.125
4	Potassium	0.250
5	Magnesium	0.250
6	Calcium	0.250

When the acetonitrile concentration increases, the retention times of the divalent cations such as magnesium and calcium decrease disproportionately to the monovalent cations. From 20% acetonitrile in the eluent, magnesium elutes before potassium.

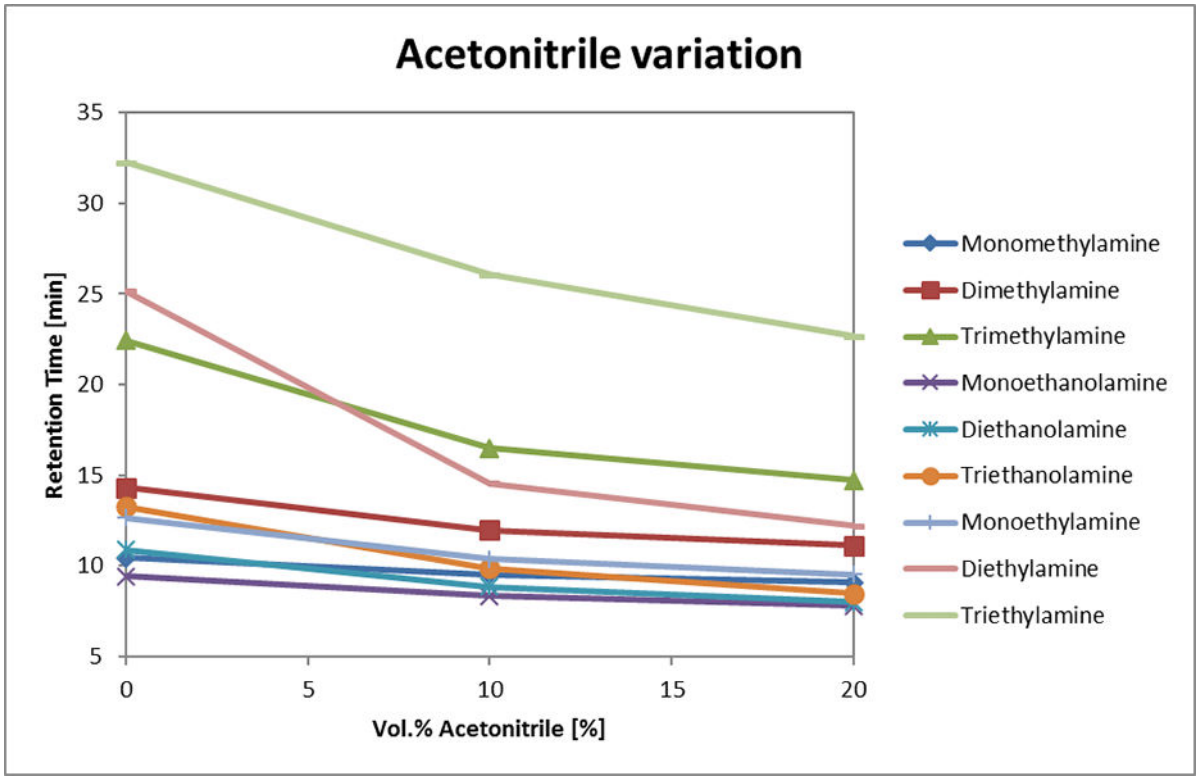
Transition metals



Acetonitrile has less impact on the retention time of rubidium and cesium.

The retention times of other transition metals are shortened when the acetonitrile concentration increases. A higher proportion of acetonitrile in the eluent leads to a deterioration of the peak form.

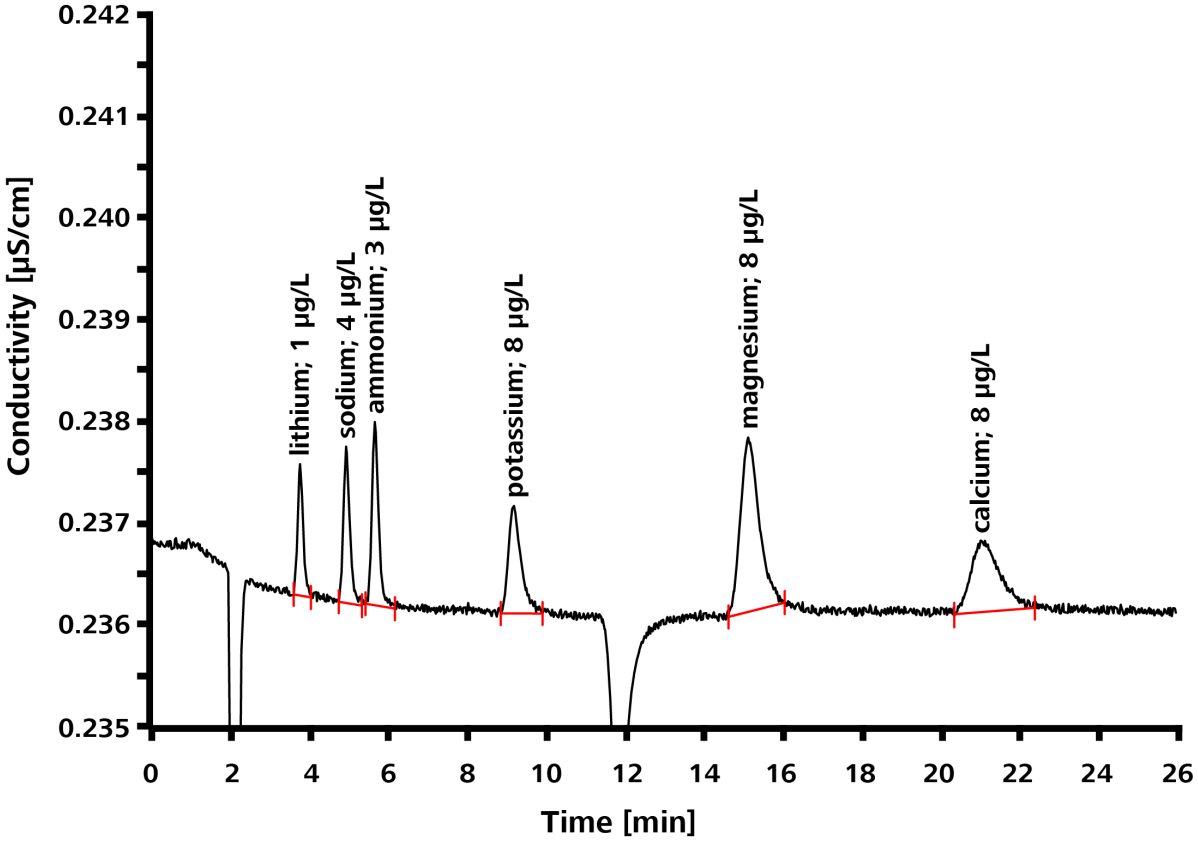
Amines



The retention times of all the amines continuously decrease with increasing acetonitrile concentration. Diethylamine and Trimethylamine as well as triethanolamine and monomethylamine change the retention order.

5.6 Trace analysis

Column:	Metrosep C Supp 2 - 150/4.0
Sample preparation:	-
Detection:	Conductivity
Suppression:	Sequential suppression with MSM-HC C and MCS
Temperature:	40 °C
Loop:	20 µL
Flow rate:	1.0 mL/min
Eluent:	5.0 mmol/L of nitric acid and 50 µg/L of rubidium



	Metrosep C Supp 2 – 150/4.0	µg/L
1	Lithium	1
2	Sodium	4
3	Ammonium	3
4	Potassium	8
5	Magnesium	8
6	Calcium	8

5.7 Not suppressed

Column: Metrosep C Supp 2 - 250/4.0

Sample preparation: -

Detection: Conductivity

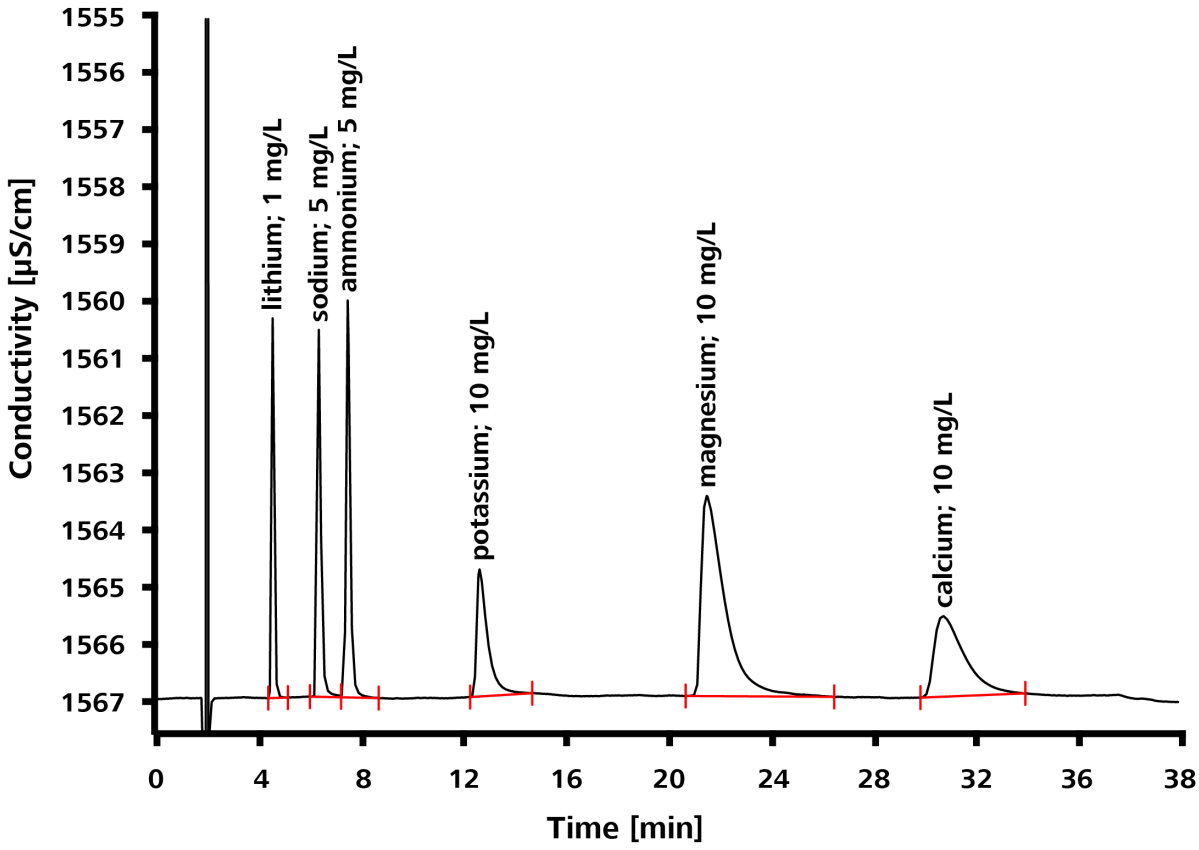
Suppression: -

Temperature: 40 °C

Loop: 10 µL

Flow rate: 1.0 mL/min

Eluent: 5.0 mmol/L of nitric acid and 50 µg/L of rubidium



	Metrosep C Supp 2 – 250/4.0	mg/L
1	Lithium	1

	Metrosep C Supp 2 – 250/4.0	mg/L
2	Sodium	5
3	Ammonium	5
4	Potassium	10
5	Magnesium	10
6	Calcium	10

The resolution of sodium and ammonium lies on the Metrosep C Supp 2 between the resolutions on the Metrosep C 4 and the Metrosep C 6.

5.8 Standard cations with zinc, manganese, cobalt and nickel

Column: Metrosep C Supp 2 - 150/4.0

Sample preparation: –

Detection: Conductivity

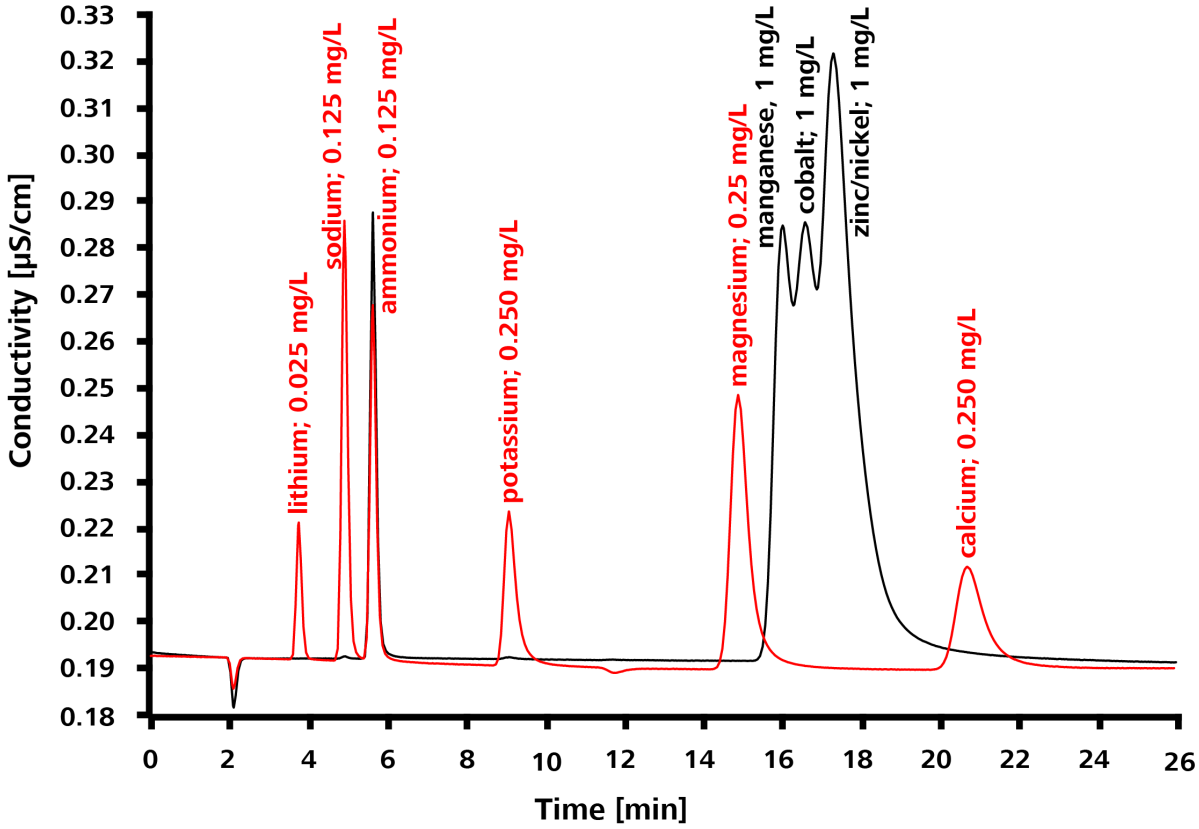
Suppression: Sequential suppression with MSM-HC C and MCS

Temperature: 40 °C

Loop: 20 µL

Flow rate: 1.0 mL/min

Eluent: 5.0 mmol/L of nitric acid and 50 µg/L of rubidium



	Metrosep C Supp 2 – 150/4.0	mg/L
1	Lithium	0.025
2	Sodium	0.125
3	Ammonium	0.125
4	Potassium	0.250
5	Magnesium	0.250
6	Calcium	0.250

Manganese, cobalt, zinc and nickel do not impact the measurement of standard cations.

5.9 Dose-in gradient

Column: Metrosep C Supp 2 - 250/4.0

Sample preparation: –

Detection: Conductivity

Suppression: Sequential suppression with MSM-HC C and MCS

Temperature: 40 °C

Loop: 20 µL

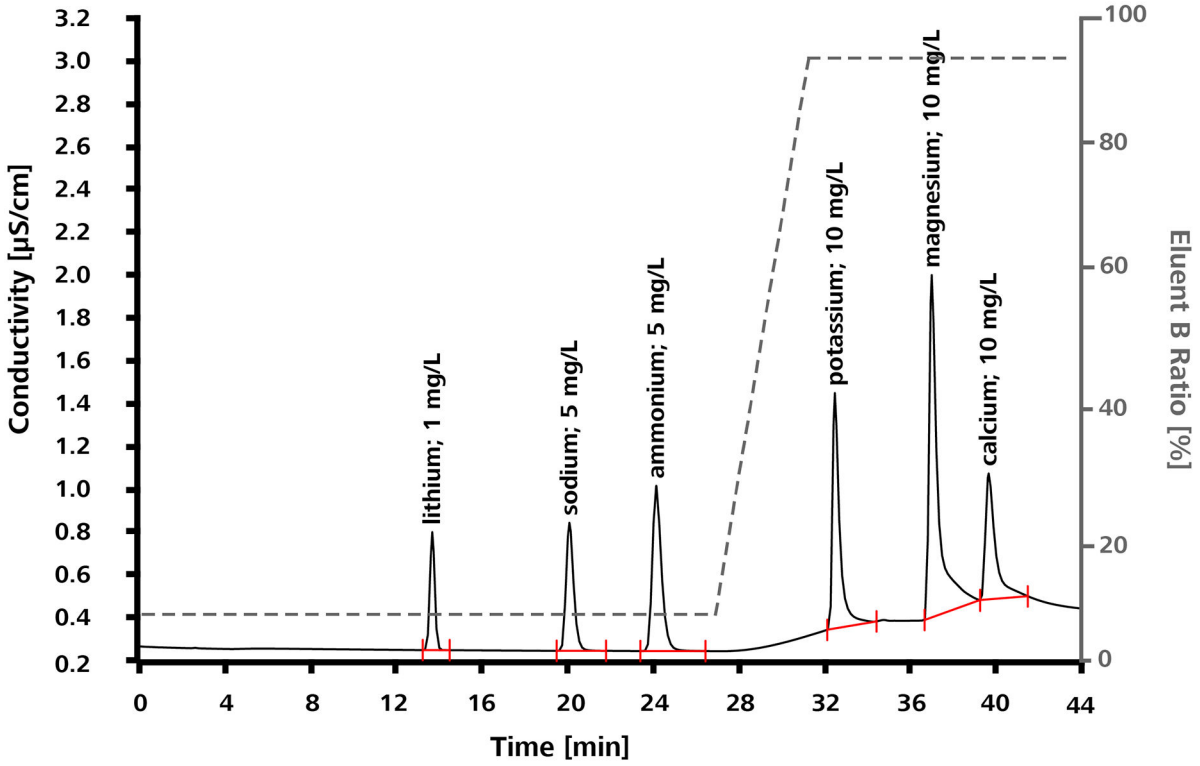
Flow rate: 1.0 mL/min

Eluent: A) 0.5 mmol/L of nitric acid and 50 µg/L of rubidium

B) 10 mmol/L of nitric acid and 50 µg/L of rubidium

Linear gradient

- 0-23 min: A: 90%, B: 10%
- 23-28 min: A: 90-5%, B: 10-95%
- 28-40 min: A: 5%, B: 95%



	Metrosep C Supp 2 – 250/4.0	mg/L
1	Lithium	1
2	Sodium	5
3	Ammonium	5
4	Potassium	10
5	Magnesium	10
6	Calcium	10

The resolution between sodium and ammonium is very high at 5.7. By using the gradient the chromatogram duration can be limited to 44 minutes, even at a resolution this high.

5.10 40 ppm ethanolamine to 10 ppm standard cations

Column: Metrosep C Supp 2 - 150/4.0

Sample preparation: –

Detection: Conductivity

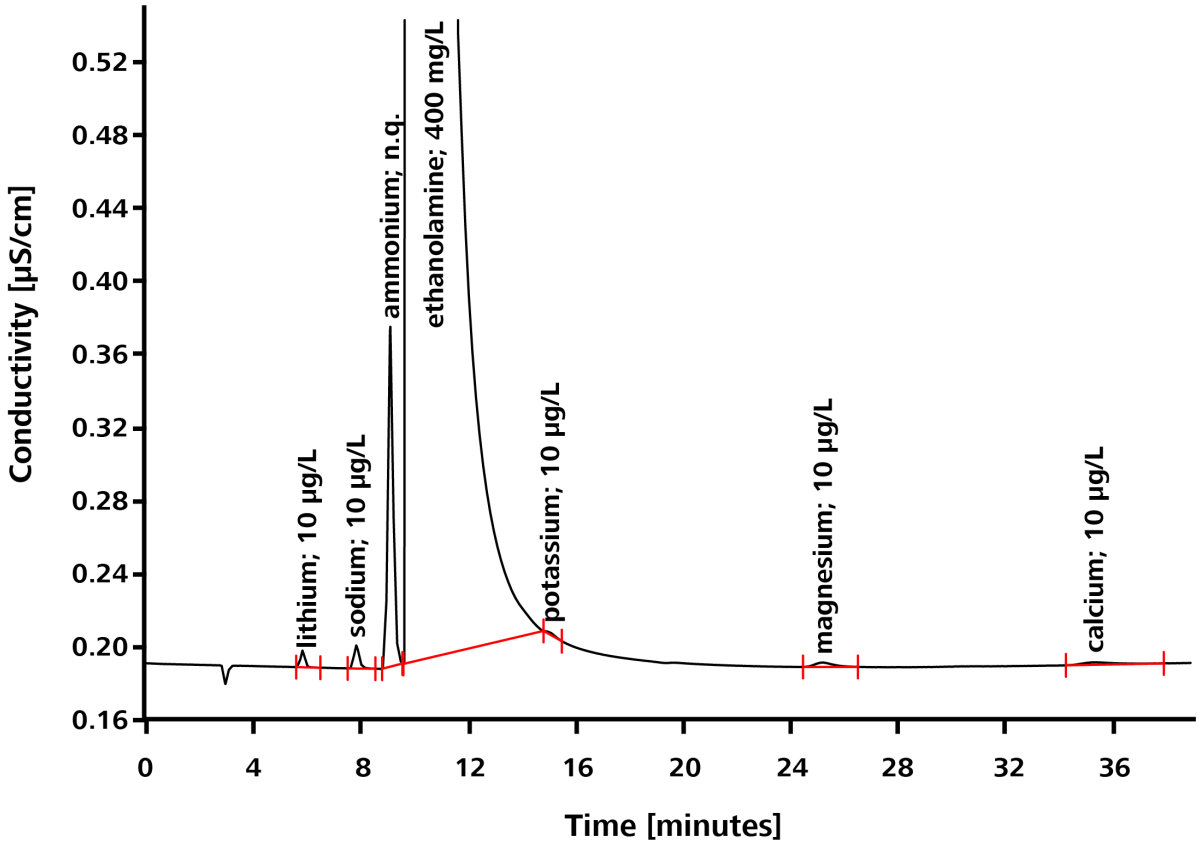
Suppression: Sequential suppression with MSM-HC C and MCS

Temperature: 40 °C

Loop: 20 µL

Flow rate: 1.0 mL/min

Eluent: 5.0 mmol/L of nitric acid and 50 µg/L of rubidium



	Metrosep C Supp 2 – 150/4.0	µg/L
1	Lithium	10
2	Sodium	10
3	Ammonium	not quantifiable
4	Ethanolamine	400,000
5	Potassium	10
6	Magnesium	10
7	Calcium	10

When carrying out a trace analysis with the preconcentration technique and an injection volume of 2000 µL, the indicated values correspond to concentrations of 4 ppm ethanolamine and 0.1 ppb of the standard cations.

5.11 Quick determination of sodium, potassium, magnesium and calcium

Column: Metrosep C Supp 2 – 100/4.0

Sample preparation: –

Detection: Conductivity

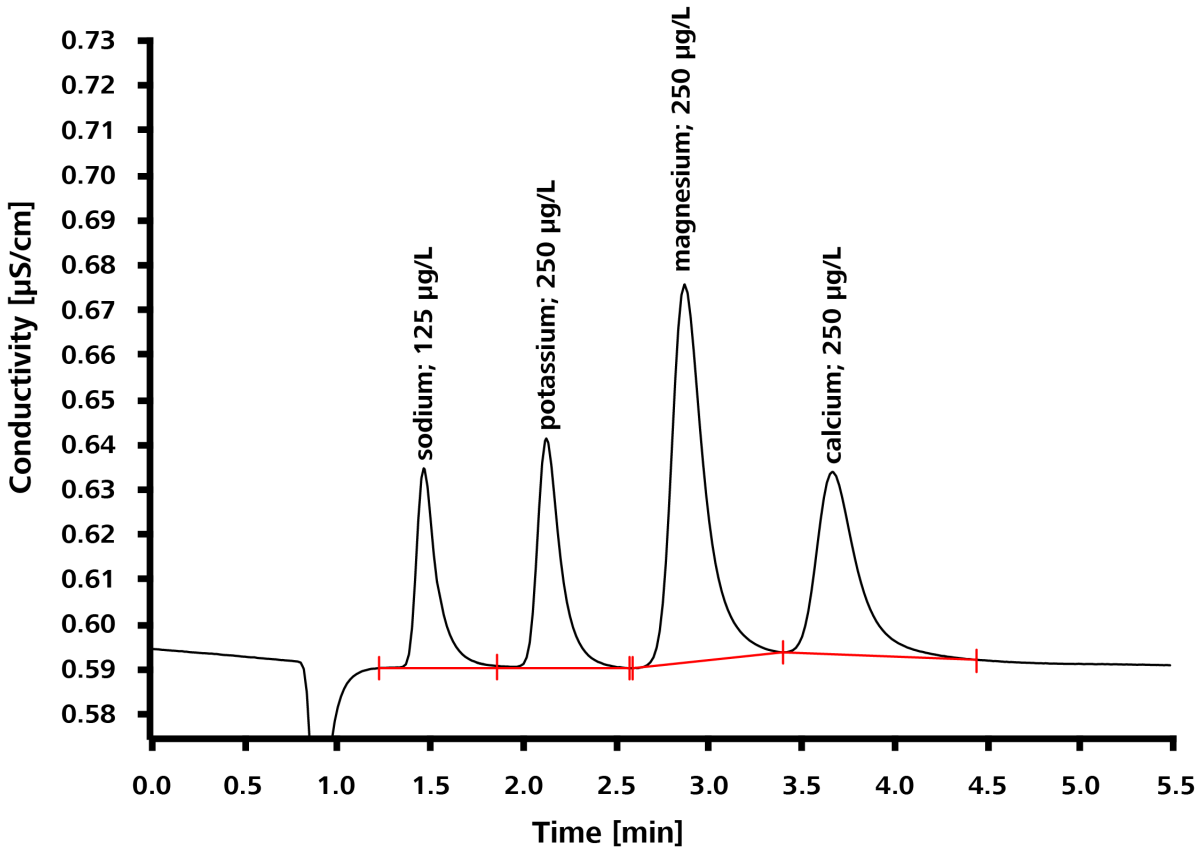
Suppression: Sequential suppression with MSM-HC C and MCS

Temperature: 60 °C

Loop: 20 µL

Flow rate: 2.0 mL/min

Eluent: 7.0 mmol/L of nitric acid and 50 µg/L of rubidium



	Metrosep C Supp 2 – 100/4.0	mg/L
1	Sodium	0.125
2	Potassium	0.250
3	Magnesium	0.250
4	Calcium	0.250

Sodium, potassium, magnesium and calcium can be determined in less than 5 minutes.

5.12 Determination of amines

Column: Metrosep C Supp 2 - 150/4.0

Sample preparation: -

Detection: Conductivity

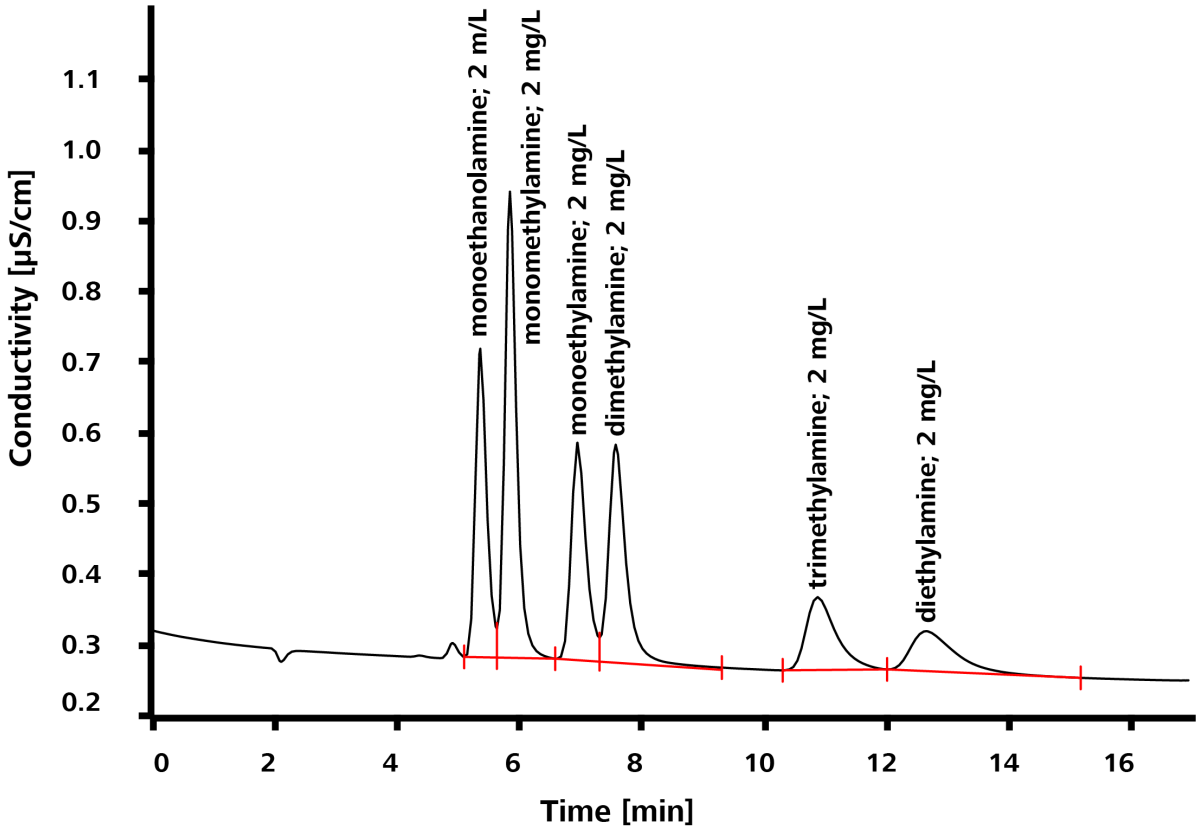
Suppression: Sequential suppression with MSM-HC C and MCS

Temperature: 60 °C

Loop: 20 µL

Flow rate: 1.0 mL/min

Eluent: 5.0 mmol/L of nitric acid and 50 µg/L of rubidium



	Metrosep C Supp 2 – 150/4.0	mg/L
1	Monoethanolamine	2
2	Monomethylamine	2
3	Monoethylamine	2
4	Dimethylamine	2
5	Trimethylamine	2
6	Diethylamine	2

6 Troubleshooting

6.1 Regeneration

CAUTION

Do not regenerate the column as a preventive measure!

Each regeneration process causes stress on the separation column and reduces its service life *see "Regenerating separation columns", page 5*.

Problem

- The backpressure increases.
- Double peaks occur.
- Tailing effects occur.
- The retention times become shorter.
- The resolution deteriorates.

Correction

Regenerating the separation column

Start by replacing the guard column if the above problems occur. Regenerate the separation column as described below if this measure does not help.

1 Disconnecting the separation column from the IC system

Disconnect the column outlet from the downstream functional units such as suppressor or detector.

Collect the flow of liquid in a beaker.

2 Regenerating the separation column

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

- Contamination with organic components *(see table 3, page 41)*.
- Contamination with inorganic components *(see table 4, page 41)*.

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

Table 3 Contamination with organic components

	Rinse with	Duration [min]	Flow rate [mL/min]
1	Ultrapure water, opposite to flow direction	60	1.0
2	Acetonitrile/water (40/60), opposite to flow direction	60	1.0
3	Ultrapure water, opposite to flow direction	60	1.0

Table 4 Contamination with inorganic components

	Rinse with	Duration [min]	Flow rate [mL/min]
1	Ultrapure water, opposite to flow direction	30	1.0
2	50 mmol/L nitric acid, opposite to flow direction	60	1.0
3	Ultrapure water, opposite to flow direction	30	1.0

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 the eluent degasser.

6.4 Unknown peaks

Problem

The chromatogram contains wide, unknown peaks.

Causes and prevention

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

6.5 Increasing backpressure

Problem

The backpressure increases.

Causes and prevention

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

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:

- Brochure: Cation suppression in ion chromatography – Cation determination in the trace range (8.000.5163)
- Application Note CS-016: Metrosep C Supp 2 – 150/4.0: Amines applying suppressed cation chromatography
- Application Note CS-017: Metrosep C Supp 2 – 250/4.0: Ammonium in acidic absorption solution – proof of concept
- Application Note CS-018: Metrosep C Supp 2 – 250/4.0: Cations in wastewater applying a Dose-in gradient
- Application Note CS-019: Trace ammonium and trimethylamine in 30% hydrogen peroxide applying sequential suppression
- Application Note CS-020: Fast IC with Metrosep C Supp 2 – 100/4.0: Four cations in five minutes

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