

Tips and tricks for the 948 Continuous IC Module

Industry sector

Chemical; pharmaceuticals; food & beverage; personal care & cosmetics

Keywords

Tips and Tricks, 948, Continuous IC Module, Hydroxide, Hydroxide Gradients, Automatic Hydroxide Eluent Production, S01; S010; S04; S040; S07; S070; S12; S122

Summary

This is a continuously growing list of tips and tricks to help work with the 948 Continuous IC Module. If you have additional helpful tips to add to this document, please suggest them to your local Metrohm representative.

Instruments and accessories

Article No.	Product
2.948.0010	948 Continuous IC Module
2.925.0020	925 ECO IC
2.930.xxxx	930 Compact IC Flex
2.940.xxxx	940 Professional IC

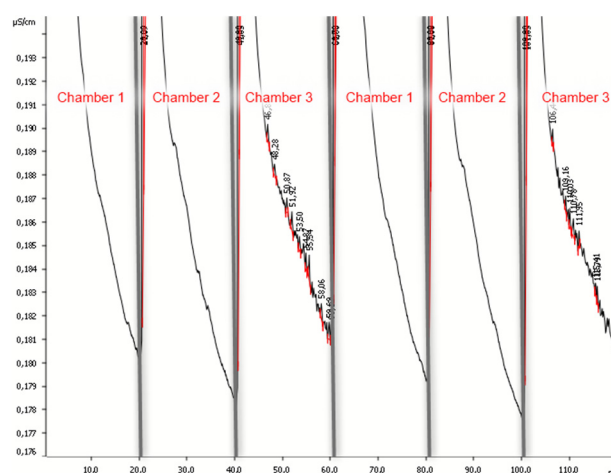
General tips

For optimal measurement results, the following points are essential to assess and, if necessary, rectify.

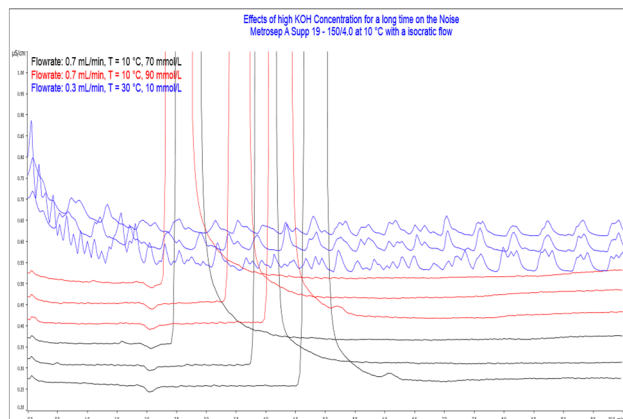
High noise in one MSM chamber

Issue: Regeneration with sulfuric acid may result in noise within one chamber of the Metrohm Suppressor Module (MSM) if the instrument remains inactive for several days. The following example shows this issue for chamber 3 in the MSM.

Example:



Example:



Solution: Use an external rinsing rate of at least 1 mL/min (up to 2 mL/min) for the MSM and CT-A. The increased flow rate will flush the hydrogen bubbles out of the CT-A more effectively.

High noise when using microbore (2 mm) systems

Like the previous issue, the reduced flow of the high-pressure pump is unable to remove all the hydrogen bubbles in the CT-A, as it is used in STREAM mode. Therefore, use the same remedy as previously discussed.

Usage of high concentrations of hydroxide (KOH, LiOH, NaOH) will reduce the concentrate lifetime

Issue: The voltage error is triggered earlier before ever receiving a warning to exchange the concentrate bottle.

Reason: Due to increased resistance in the electrolysis at higher currents, an abundance of K^+ is required for the electrolysis at higher hydroxide concentrations. Therefore, voltage errors appear earlier than the internal limit (i.e., 200 mmol/L) for exchanging the concentrate. A voltage error already appears at 80 mmol/L KOH with roughly 1 mol/L KOH remaining.

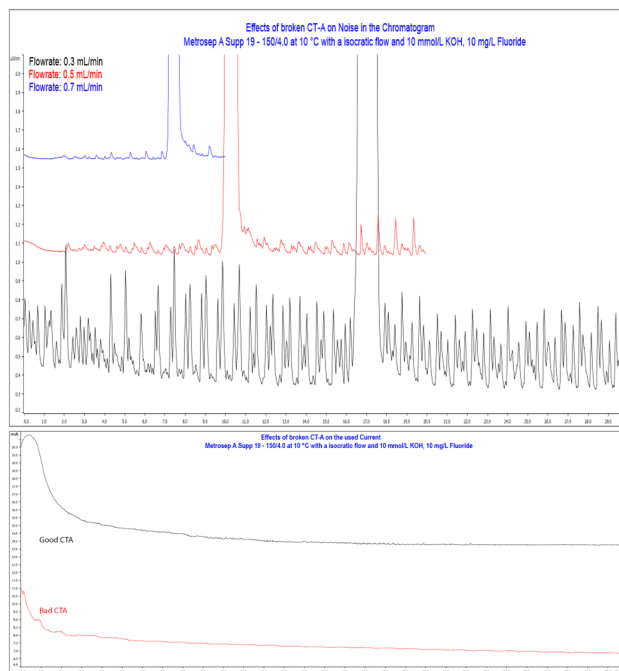
Solution: Check if the voltage error is triggered because of the bottle. To do this, create an isocratic equilibration method with the highest KOH concentration you want to reach in your gradient. If the method always triggers a voltage error, change the concentrate. Next, adapt the warning limit for the next bottle to a value slightly above the concentration that remained in the previous bottle.

CT-A is damaged

Issue: The noise increases in the system. The origin of this noise may be unclear.

Reason: The CT-A produces fewer hydrogen bubbles than before. The CT-A produces a lower current than usual. Therefore, always track the CT-A current.

Examples:



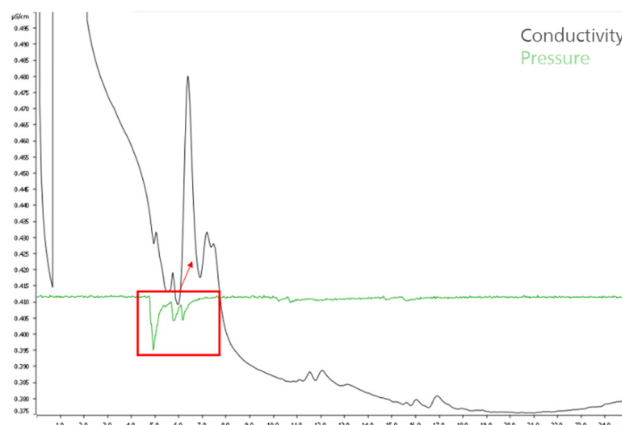
Solution: Replace the CT-A.

Positive unknown peaks in the chromatogram

Issue: Air bubbles coming from the eluent lead to positive peaks in the resulting chromatogram.

Reason: If the flow of the liquid through the high-pressure pump is disturbed due to an air bubble, a reduced flow propagates through the eluent producer cartridge (EPC). However, as the EPC still produces the same amount of K^+ and OH^- , the concentration of the KOH is higher for the eluent moving along the EPC at that time. This leads to positive peaks in the chromatogram, as shown below.

Example:



Solution: Always track the CT-A current and pressure, and the EPC voltage. These three signals indicate if it is an air bubble issue: the pressure decreases while both the EPC voltage and CT-A current spike.

Additional information:

EPC-A voltage: As K^+ concentration in the concentrate decreases, voltage must increase to maintain the current. Voltage spikes are seen here since less liquid in contact with the electrodes leads to a higher resistance.

EPC-A current: Applied current generates OH^- and drives K^+ through the membrane. Current applied is a function of the desired concentration and the eluent flow rate.

CT-A voltage: A constant voltage is applied to the CT-A.

CT-A current: If contaminations are present, they flow through the CT-A membrane causing a current. Therefore, the easier it is for something to pass the membrane, the higher the current will be. The higher the current, the more contaminations are present in the UPW source.

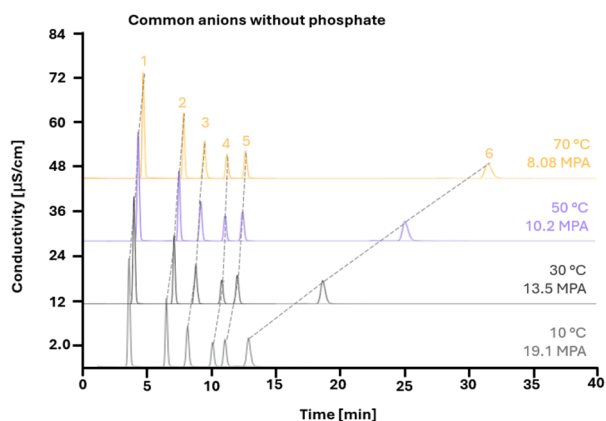
Usage of different hydroxides (Na, K, Li)

Various hydroxides (Na, K, or Li) lead to different separation behaviors for some components when using, e.g., the Metrosep A Supp 19 column. This issue especially affects carbonate, sulfate, and phosphate – but not all ions were checked. Separation of the system peak (carbonate) from sulfate on the Metrosep A Supp 19 and A Supp 21 is only possible with LiOH concentrate which needs to be prepared by hand.

Effect of temperature changes on hydroxide measurements

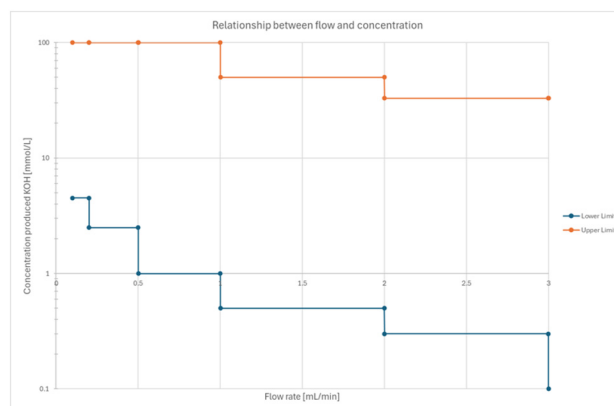
Temperature changes in the separation column lead to the «hydroxide-tilted V-effect» where all components shift to later eluting times. However, multivalent ions are more affected by this phenomenon.

Example:



Flow and concentration dependencies

Example:



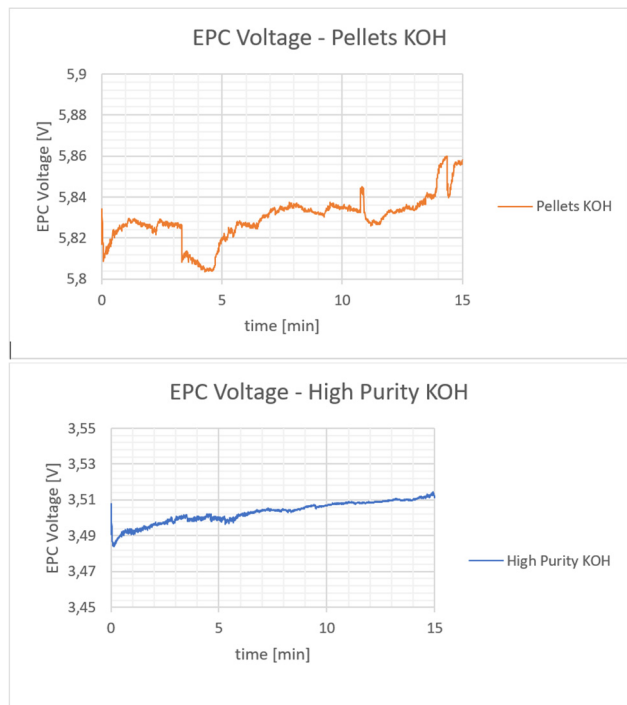
Flow and corresponding accessible KOH concentration

Flow (mL/min)	Concentration (mM KOH)
0.100–0.200	4.5–100
0.201–0.500	2.5–100
0.501–0.500	2.5–100
1.001–2.000	0.5–50
2.001–2.999	0.3–33
3.000	0.1–33

Using high-purity chemicals and hydroxides (KOH, LiOH, NaOH) will give you the longest EPC lifetime

Issue: Lower purity chemicals lead to a higher EPC voltage and more voltage noise. This will most likely lower the EPC lifetime.

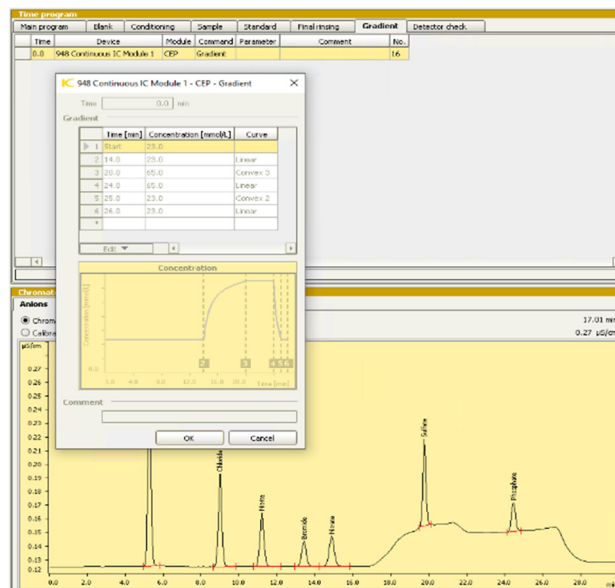
Examples:



Overshoot peaks with gradients

When using gradients with Metrohm separation columns, the chemistry will lead to a situation resembling a gradient overshoot. This can be seen as a hump at the initialization of the gradient which will eventually drop. Once the gradient lowers back again, this will lead to an additional hump. This chemistry-related issue is also known for comparable instruments from other manufacturers.

Example:



Reason: This is most likely due to trace amounts of carbonate eluting from the column during the gradient phase and then re-absorbing after the gradient is finished.

Increased system backpressure

Issue: During the installation this will often lead to capillaries «popping out». Check all connections when installing them and clean each connector with acetone before the installation. Additionally, tighten the connections with the plastic wrench (article no. 6.2739.000).

Reason: Because of the backpressure of the 948 Continuous IC Module (EPC, CT-A, and H-DEG) the system backpressure itself will be higher.

The backpressure required for the H-DEG to work properly needs to be >10 MPa

Issue: At lower pressures the H-DEG will not remove all the hydrogen gas produced. The bubbles will create noise in the detector.

Effect: This will lead to slight delays with your gradient (depending on the flow and dead volume introduced). If you work isocratically, then there is no impact on the chromatogram.

Solution: Use a backpressure column or an additional backpressure capillary placed after the H-DEG and in front of the injector. This is needed especially when working with high concentrations of produced hydroxide (KOH, LiOH, NaOH). If you encounter noise during an application with >60 mmol/L OH⁻ produced and a low backpressure (around 10 MPa), then increase the backpressure.

Normal usage details

- The voltage at maximum power (either 161 mA or 100 mmol/L at 1 mL/min) with a new 4 mol/L KOH concentrate should be around 10–12 V. A voltage above 20 V may limit the amount of current that can be applied (i.e., the eluent concentration produced may be lower than what was programmed).
- Note that the EPC has an inline filter. This filter (as with all inline filters) needs regular replacement.
 - Reason: The filter guards the analytical column from any contamination leaching from the membranes.
- Over long periods, slight crystallization can form on the EPC. This is normal; simply clean it with UPW.
- The longer the 948 Continuous IC Module is used, the cleaner the system becomes. Therefore, the background conductivity will drop even more over time. The background conductivity is usually at a minimum when the KOH concentrate is almost done. Once the concentrate bottle is replaced, background conductivity will slightly rise again.
- Never remove the stopper from the cable. The stopper is not supposed to block the air ventilation of the concentrate bottle.
- Only use methanol as an organic solvent, and at a maximum concentration of 25%.
- Usage of MCS is only needed to improve carbonate influence from samples. If there is a noticeable impact from carbonate but no MCS present, then use LiOH to separate carbonate from sulfate.
- Eluent concentrate bottles can be prepared in whatever «concentration» suits your requirements. The concentration should be above 1 mol/L, up to 8 mol/L of hydroxide.
- If there are other issues with the baseline, then check the following instrument parts:

Article No.	Product/description
6.2821.090	Aspiration filter of the eluent (UPW)
6.1826.420	Peristaltic pump tubing (Regeneration tubing)
6.1826.360/ 6.1826.380	Peristaltic pump tubing (External Rinsing)
6.2755.000	Tubing cartridge of the peristaltic pump
6.2821.130	Inline filters (HPP, Regenerant PP, EPC)

MSM

- Since the new MSM is higher in capacity, use higher concentrations of regenerant solution.
 - Dosino Regeneration (DR): Minimum 4.5 mL at 500 mmol/L H₂SO₄
 - PP (peristaltic pump) Regeneration: PP Rate 4 at 200 mmol/L H₂SO₄
- Ask your local Metrohm representative to calculate the capacity of the MSM and determine if your gradient stays within the MSM limit.
- During equilibration, the new MSM takes a bit longer to reach a stable baseline after stepping chambers. This is a KOH behavior that cannot be eliminated. Therefore, this drift is common for every user.
- The MSM Rotor must be stored in 15 mmol/L KOH. Additionally, if the instrument is shut down for multiple days, all chambers should be rinsed with 15 mmol/L KOH (e.g., to overcome the issue of **High noise in one MSM chamber**).
- Sometimes ghost peaks may appear in the chromatogram. They are attributed to the chemical interactions between hydroxide eluents and the process of chemical suppression. This phenomenon is an inherent chemical behavior that cannot be entirely eliminated.

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