Metrohm

Installation Instruction for ProfIC Vario 11 Anion

ProfIC Vario 11 - Anion is a Professional IC Vario system with variable sample Preconcentration, Matrix Neutralization and Matrix Elimination for trace analysis of anions. With the «ProfIC Vario 11», concentrations down to the ppt range (ng/L) in various matrixes can reliably be determined. The intelligent 800 Dosino carries out the high-precision sample preconcentration on the preconcentration column, which replaces the sample loop and removes all sample matrix using the Matrix Elimination technique.

When strongly acidic or alkaline samples are involved, Inline Neutralization is recommended. With this technique, such samples can be neutralized fully automatic during sample delivery and then analyzed directly.

This technique is also suitable for determining ultratraces. After Inline Neutralization, even strongly alkaline samples can be preconcentrated, this facilitates analyses in the ng/L concentration range.

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1. Delivery Package

Delivered with ProfIC Vario 11 Anion package:

Nr	Article no.	Article designation
IC		
1	2.940.1530	Professional IC Vario ONE/SeS/PP/Prep 3
Detector		
1	2.850.9010	IC Conductivity Detector
Sample processor		
1	2.858.0030	Professional Sample Processor - Pump - Injector
Accessoires		
1	2.800.0010	800 Dosino
1	6.2832.000	MSM - A Rotor (used for SPM)
1	6.2842.020	Adapter Vario to MSM and MSM - LC
1	6.5330.140	IC Equipment: MiPCT
1	6.1808.090	Thread adapter M8 outer / M6 inner
1	6.1014.200	Metrosep I Trap 1 – 100/4.0
Optional		
1	6.6059.312	MagIC Net 3.1 Professional CD - 1 license
1	6.6059.313	MagIC Net 3.1 Multi CD - 3 licenses
1	6.9920.191	Sample rack 3 x 60.6 mm + 35 x 41 mm
20	6.1608.100	PE bottle 50 mL
1	6.2820.000	Al foil
1	6.1030.420	Metrosep A Supp 15 - 150/4.0
1	6.1030.500	Metrosep A Supp 15 Guard/4.0
1	6.1006.310	Metrosep A PCC 1 HC/4.0
1	6.2832.000	MSM Rotor A
1	6.2842.000	MSM - HC Rotor A
1	6.2844.000	MSM - LC Rotor A
1	6.2842.020	Adapter Vario to MSM and MSM - LC
1	2.941.0010	Eluent Production Module

2. Installation

Following is a detailed description of how to install ProfIC Vario 11 Anion.

We strongly recommend that the individual steps are carried out in the order given below.

2.1. Installation of the software

All programs must be shut down first. Make sure no Metrohm instrument is connected to the PC. Install MagIC Net with the help of the MagIC Net - CD. The Microsoft Installation Wizard is accepted and executed. All the standard directories proposed by the program should be accepted. Restart the PC.

Now as soon as you connect a Metrohm instrument via USB connection to the PC, the driver is installed and a window will pop up in MagIC Net, asking you if you'd like to store this device in your configuration. If you do, please answer with yes. The names will be checked later in this installation instruction.

2.2. Accessory Kit: Vario/Flex Basic (6.5000.000)

Using the Accessory Kit Vario/Flex Basic, install the Professional IC Vario. Remove handle, place the detector block in the instrument and connect the detector cable, remove the transport locking screws, connect the leak sensor cable and connect the drainage tubing. Then please mount the holder for Dosino to IC instruments (6.2057.210) on the IC by removing the bottle holder on top of the instrument, mounting the Dosino holder on the side of the IC and remounting the bottle holder on top.

In continuation, set up the waste collector by assembling the cap and screwing it onto the vessel. Then hang the waste collector holder on the side of the IC, so you can observe the droplets coming out of the capillaries that later will be connected to the collector. Attach the waste tube to the vessel and lead it to the waste canister. When the tube is too long, please shorten it, because it is important to have a high level difference for the liquid to drain.

The power cable and USB cable (6.2151.020) are plugged into the rear of the Professional IC Vario. Please don't switch on the instrument yet. This step will follow after the completed installation.

2.3. Accessory Kit: Vario/Flex ONE (6.5000.010)

In the box with the Accessory Kit ONE, you will find all the accessories for setting up the Eluent bottle. Please lead the aspiration tube for the Eluent through the M8 stopper, the O-ring and the eluent cap. Then fix the white weight (6.2744.210), the adaptor (6.2744.210) and the aspiration filter (6.2821.090) on the eluent aspiration tube, all the while being careful not to touch the filter and its connections with bare hands in order to avoid cross contamination. Also fix the filled adsorber tube on the eluent cap. Please refer to the 940 Professional IC Vario manual for a detailed description.

2.4. 858 Professional Sample Processor

For a detailed description, please refer to the 858 Professional Sample Processor manual. In general, you will have to do the following: plug in the Swing Head connection cable and the power supply cable. The controller cable (6.2151.000) is plugged into the plug "Contr." on the 858 and connected to the IC device via USB. The sample processor initializes and lifts its Swing Head, once the IC device it is connected to is recognized by the software. For this, you have to plug the USB cable of the IC into the PC and turn it on. Afterwards, it is possible to mount the retaining plate, the needle and the safety shield (for detailed instructions please refer to the 858 Professional Sample Processor manual).

Please remove one element of the guide chain close to the swing head by using a screwdriver to unfix it. In its place, connect the transfer tubing PFA, 20 mL (6.1825.280).

For the following installations, please shut down the IC again and disconnect the 858 from the power supply.

2.4.1. Liquid Handling Station (LQH Station)

The Liquid Handling Station consists of two function units: The rinsing unit is equipped with a two-pipe system and is used for rinsing the sample needle from the inside and outside or for fresh ultra pure water pick-up. The second unit is used for dilution of samples and standards. In the present case, the dilution unit won't be used.

The LQH Station is mounted onto the left side of the Sample Processor. For installation instructions, please refer to the Liquid Handling Station manual. Please make sure that the Liquid Handling Station is aligned properly before continuing the setup. All angle settings will be configured in a later step.

2.4.1.1. Rinsing unit

For this application, ultrapure water for rinsing will be supplied by the peristaltic pump on the Sample Processor. The waste tubing (6.1801.120) is fixed at the bottom of the rinsing station to drain the waste water.
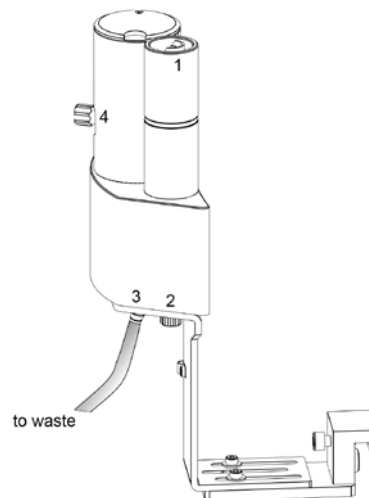

2.4.2. Peristaltic pump

Continuous fresh ultrapure water supply is very essential for this technique as every impurity is visible on the chromatogram or could accumulate in the system. Therefore, the peristaltic pump on the 858 Sample Processor is used to feed fresh ultrapure water to the Liquid Handling Station for rinsing the needle and for fresh ultrapure water supply. PEEK capillaries (i.D. 0.5 mm, 6.1831.180 included in 6.5330.140) are used to connect the black/black peristaltic pump tubing (6.1826.340) with the LQH Station, installing a Metrosep I Trap 1 - 100/4.0 column (6.1014.200) in between these two elements in order to purify the water further before use in the IC flowpath.

2.4.3. Injection valve

For this application, the injection valve at the Sample Processor is used as a direction switch valve because the sample needs to be pushed through the SPM and not pulled. This is to avoid air aspiration into the sample and to obtain a more accurate sample volume transfer. On the injection valve, close inserts 1, 2 and 3 with threaded stoppers (6.2744.060). Position 4 is connected with a PEEK capillary (i.D. 0.5 mm, 6.1831.160) to the sample needle. Position 5 is connected with a PEEK capillary (i.D. 0.5 mm) to the T-connector on the PFA transfer tubing (6.1825.280). At position 6, the SPM inlet capillary – labeled with *in-* is directly connected.
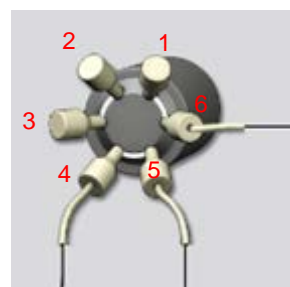
2.4.4. 800 LH Dosino

The liquid handling for this installation will be done with an 800 Dosino. The PFA transfer tubing (6.1825.280) is used as a buffer volume to avoid sample contact with the Dosing Unit. In the IC Equipment: MiPCT, the PFA transfer tubing is included. If fluoride is required to be analysed in the sub-ppb (µg/L) range, it is recommended to use the PEEK transfer tubing 10 mL, 1/8 (6.1825.270) instead, as Fluoride can leach out of the PFA material.

Accurate dosing is performed by the Dosino via a Trap column (port 1) to guarantee excellent water quality. Aspiration and filling steps are carried out via port 3 (no trap column) to reduce back pressure. Tight screwing of all connections at the Dosino and the transfer tubing is of uttermost importance for accurate results.

For installation details, please refer to the following schematics:
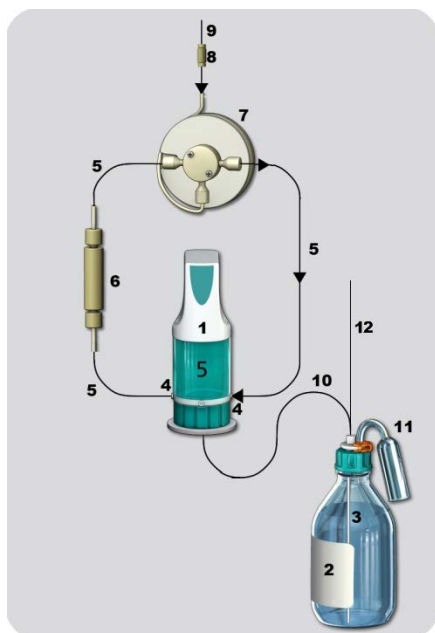
1. Dosino with 5 mL Dosing Unit: Place it on the Dosino holder that has been mounted to the side of the IC in the beginning of this installation and secure it with the help of the thread adapter (6.1618.020).

2. Ultrapure water reservoir (2 L bottle). To avoid air bubbles in the dosing unit, degas the water before use. Alternatively, when an ELGA flex ultra pure water system is used, port 2 of the Dosino can be connected directly to this system by a M6 FEP tubing.

3. FEP aspiration tubing to canister (6.1819.110) to be inserted into the M6 hole of the eluent cap and to be cut to the appropriate length for the 2 L bottle.

4. M6 threads / UNF 10/32 coupling (6.2744.080) to connect capillaries to the Dosino ports.

5. PEEK capillaries (i.D. 0.5 mm, 6.1831.180 included in 6.5330.140).

6. The (6.1014.200) Metrosep I Trap 1 - 100/4.0 column, to be connected to the Dosing Unit port 1. The flow direction is from the Dosino to the transfer tubing (n° 7).

7. Transfer tubing (20 mL, 6.1825.280) with T-connector.

8. Coupling (6.2744.310) to connect the transfer tubing to a capillary.

9. PEEK capillary (i.D. 0.5 mm, 6.1831.180 included in 6.5330.140) leading to the injection valve position 2 on the Sample Processor.

10. FEP Tubing M6 100 cm (6.1805.120) to connect the LH Dosino port 2 with the T-connector (6.1808.060) splitting the water coming from the 2 L bottle of ultrapure water.

11. The adsorber tube consists of two parts, the adapter for the adsorber tube (6.1624.000) and the adsorber tube (6.1619.000) which can be found in the accessories of the Dosing Unit. It is recommended to fill the adsorber tube with cotton and CO_2 adsorber material.

12. Here, a (i.D. 0.5 mm, 6.1831.180 included in 6.5330.140) is connected directly to the peristaltic pump of the Sample Processor for the water supply to the LQH station.

The Dosino is plugged in at the back of the Sample Processor. The method ProfIC Vario 11 Anion uses the MSB1 port of the Sample Processor for the Dosino. Be careful to have the Sample Processor disconnected from the power supply for this step!

2.5. Interconnection of devices

The whole setup of the ProfIC Vario 11 Anion packet is depicted here:
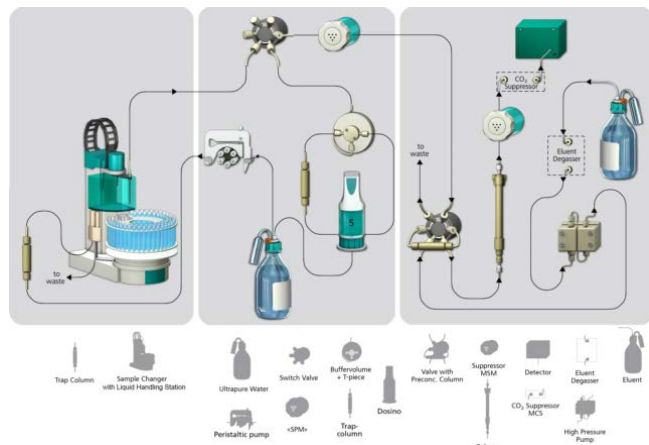
Prepare and degas an eluent suitable for the column you want to use (see column manual). For the Metrosep A Supp 15 - 150/4.0 a solution of 5.0 mM Na_2CO_3 / 0.3 mM NaOH is adequate.

Provide the regeneration solution for the MSM suppressor (100 mM sulfuric acid) and also the regeneration solution for the SPM suppressor (50 mM perchloric acid).

2.6. 940 Professional IC Vario

Capillaries are connected according to the following list and the diagram below:
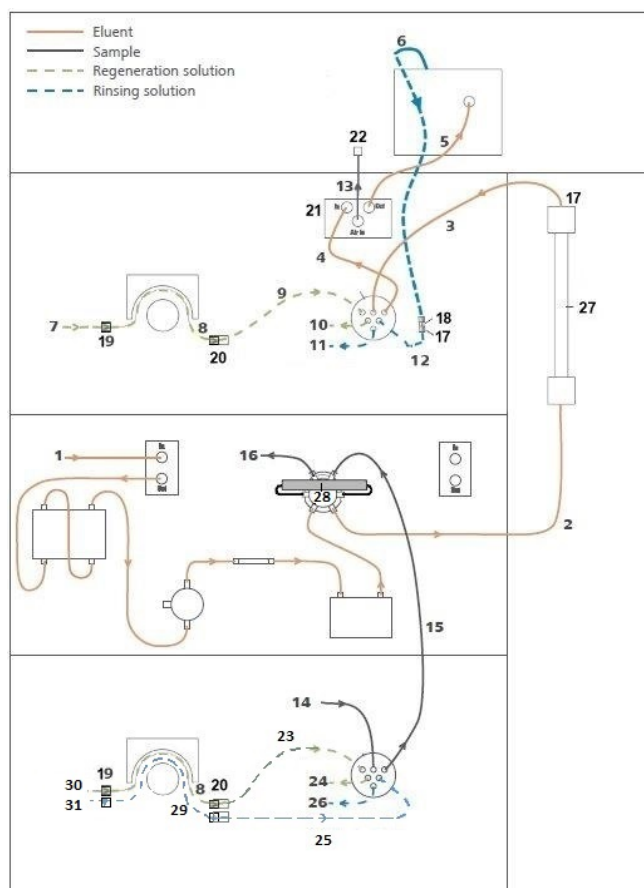
2. Capillary to Column inlet – 0.25 mm ID

3. MSM inlet capillary – labeled with *in*

4. MSM outlet capillary – labeled with o*ut* can be connected to the MCS or directly to the detector using a coupling (6.2744.040)

5. Detector inlet capillary

6. Detector outlet capillary connected to the MSM inlet capillary – labeled with *rinsing solution* with the help of a coupling (6.2744.040) for the STREAM setup

7. Regeneration solution aspiration capillary – PTFE, 0.5 mm ID - connected to the bottle with 100 mmol/L sulfuric acid and the orange/yellow pump tubing (8). This pump tubing is, on the other side, relied to the regeneration solution capillary of the MSM - labeled with *regenerant* - with a security lock and filter (20)

8. Peristaltic Pharmed pump tubing with orange/yellow stoppers (6.1826.420)

9. MSM regeneration inlet capillary – labeled with *regenerant*

10. MSM regeneration solution outlet capillary – labeled with *Waste reg.* – to be connected to the waste collector

11. MSM rinsing solution outlet capillary – labeled with *waste rins.* - to be connected to the waste collector

12. MSM rinsing solution inlet capillary - labeled with *rinsing solution* – connected to the detector outlet capillary with a coupling (6.2744.040)

13. MCS air aspiration capillary – connected to the CO_2-adsorber cartridge

14. SPM inlet capillary – labeled with *in-* connected to the injection valve of the Sample Processor

15. SPM outlet capillary – labeled with o*ut* – connected to the injection valve of the IC

16. PTFE capillary connection to the waste collector

17. PEEK pressure screw short (6.2744.070)

18. Coupling 2 x UNF 10/32 PEEK (6.2744.040)

19. Coupling nozzle - UNF 10/32 (6.2744.034)

20. Pump tubing connector with security lock and filter (6.2744.180)

21. PEEK pressure screw long (6.2744.090)

22. Luer coupling – connect with short PEEK pressure screw to CO_2-adsorber cartridge

23. SPM regeneration inlet capillary – labeled with *regenerant*

24. SPM regeneration solution outlet capillary - labeled with *Waste reg.* – to be connected to the waste collector

25. SPM rinsing solution inlet capillary - labeled with *rinsing solution*

26. SPM rinsing solution outlet capillary – labeled with *waste rins.* - to be connected to the waste collector

27 Initially, the UNF 10/32 coupling (6.2744.040) is installed instead of the column to rinse the system with eluent, after rinsing the column is installed.

28 Metrosep A PCC 1 HC/4.0 (6.1006.310) – IC preconcentration column, to be connected to the valve with PEEK capillaries (i.D. 0.25 mm), instead of a loop, flow direction should go with the eluent (left to right), and be the opposite of the loading direction of the sample

29 Peristaltic LFL pump tubing with black/black stoppers (6.1826.340)

30 Regeneration solution aspiration capillary – PTFE, 0.5 mm ID, connected to the bottle with 50 mmol/L perchloric acid and the orange/yellow pump tubing. This pump tubing is, on the other side, relied to the regeneration solution capillary of the SPM - labeled with *regenerant* - with a security lock and filter (20)

31 Rinsing solution aspiration capillary – PTFE, 0.5 mm ID, connected to the bottle with ultra pure water and the black/black pump tubing. This pump tubing is, on the other side, relied to the rinsing solution capillary of the SPM - labeled with – *rinsing solution* - with a security lock and filter (20)

Make sure that all outlet capillaries are put into the waste collector.

3. MagIC Net
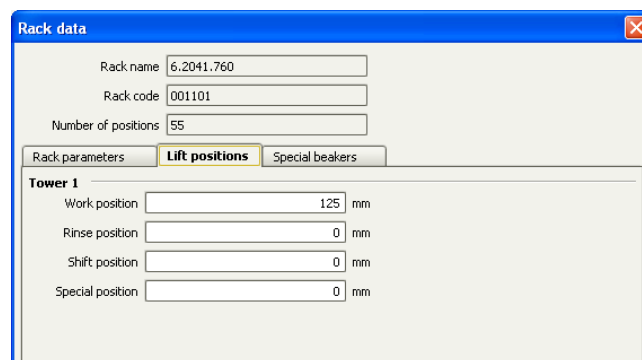
3.1 Configuration

Please adjust the "view layout" of the configuration. Please configure it in the following way (the order of the sub windows is up to you):
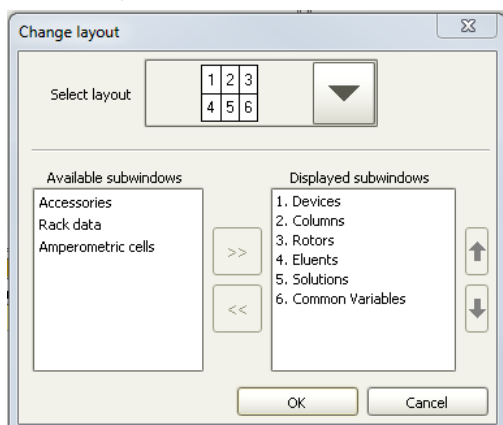

Now please connect the USB cables from the instruments and turn their power on. Connected USB devices are automatically recognized when MagIC Net is started. After confirmation of the automatically generated requests, the devices and columns are stored in the configuration. The devices are predefined as "940 Professional IC Vario 1" and "858 Professional Sample Processor 1". Name them accordingly, if other names appear in your configuration (e.g. due to changed settings on your computer). The Dosino labeled as "LH Dosino", connected to the MSB1 of the 858 can be found in the sub window "solution".

In the window configuration, the 940 Professional IC Vario, the 858 Professional Sample Processor and the column Metrosep A Supp 15 - 150/4.0 are visible. Add and define the eluent, the two suppressor solutions for the MSM and the SPM and also the MSM and SPM Rotor.

The settings for the rack require a "work position" fitting to the respective rack (e.g. 125 mm for rack 6.2041.760).


For using the Liquid Handling Station, it is necessary to define external positions for the needle. This is done in the configuration of the Sample Processor in the sub tab "Tower". Please define the external positions 1 and 2 according to the picture below. Attention: The angles are only approximate values, for fine tuning, please use the manual control to obtain the correct values for this system and adjust them then in the configuration.
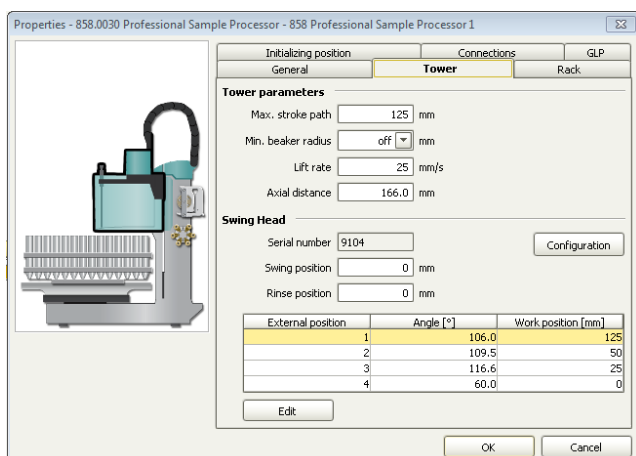
In the sub-ppb analysis all contaminations have to be avoided. To have a possible source of contamination under control, it is important to move the needle of the Sample Processor to a defined position after initialization. For this, please add an initial position which is equal to the external position 1 in your configuration:
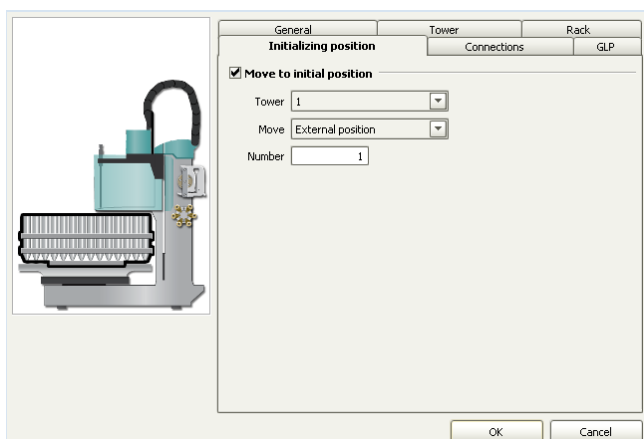
In the configuration of the solution LH Dosino, define the settings of the Dosing Unit following the example below. In the here present setup, an increased backpressure due to many capillary connections, Trap column, preconcentration column and most of all the SPM can be observed. For this reason it is important to define a maximum dosing rate for the Dosino. Also adjust length and diameter of your capillaries connected to the Dosino. This is important to ensure complete rinsing of all connections when executing the prepare command.
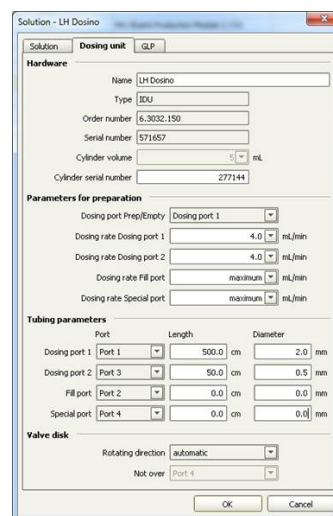
3.2 Method adjustment

The method for this configuration can be found in the example method folder on your MagIC Net - CD. In the window method, import the method for ProfIC Vario 11 Anion from the installation CD: Go to File → method manager and choose your method group. Afterwards, click on edit → import and choose the pathway on the installation CD: MagICNet\examples\methods\ ProfIC Vario \ProfIC Vario_11_Anion.imet.

In the window "Method", open the ProfIC Vario 11 Anion method and assign the following equipment: Assign the suppressor solution to the IC peristaltic pump 1 and set the speed rate to 1, add the MSM Rotor you've defined in the configuration. Similarly, assign the SPM suppressor solution to the IC peristaltic pump 2, set the speed rate to 1 and add the SPM Rotor you've defined in the configuration. Following this, link the rack of the connected sample processor, assign the eluent to the IC pump and assign the column Metrosep A Supp 15 – 150/4.0 to the analysis anion. Adjust the temperature of the column oven to 30°C. **Due to the high backpressure in the sample path created by the SPM it is very important to set the dosing speed during the sample transfer in the SPM to 1 mL/min instead of 2 mL/min as indicated here below.** Please change this in the Sample, Blank and Standard subprograms.

Perform a method test and save the method.

3.3. Purge of the system

Before inserting the column, disconnect the *out* capillary of the MSM suppressor connection piece from the MCS or from the detector and put it into a waste beaker. The suppressor needs to be rinsed with the system first and its waste should not pass through the sensitive MCS and/or detector in the beginning, as loose particles could be flushed out.

Now, please flush the system for about 10 minutes and get rid of air bubbles (by using the purge valve and syringe). During these 10 minutes, step the MSM three times in order to flush all three chambers.

As soon as the whole system is purged, reconnect the suppressor out capillary back to the MCS or the detector.

Now insert and rinse the precolumn for 10 minutes by leading the outlet directly into the waste. Afterwards connect the analytical column and flush it likewise for 10 minutes. Only afterwards the column is completely connected to the flow path of the eluent (see also 940 manual). Start the peristaltic pumps and adjust the pressure of the levers on the tubing, in order to see the droplets of suppressor regeneration solutions drop into the waste collector.

In order to confirm the tightness of the sample flow path and to rinse the capillaries, a test will be performed. Please replace the outlet of the eluent at the injection valve (position 5) with the outlet capillary from the SPM. The inlet capillary of the SPM should then be disconnected from the injection valve of the Sample Processor and by a coupling (6.2744.080) connected to port 1 of the Dosino. The capillary including the I Trap column on the Dosino can be disconnected so that no eluent can get into the I Trap. If it comes in contact with the eluent, it will be contaminated and you won't be able to use it anymore in this installation.
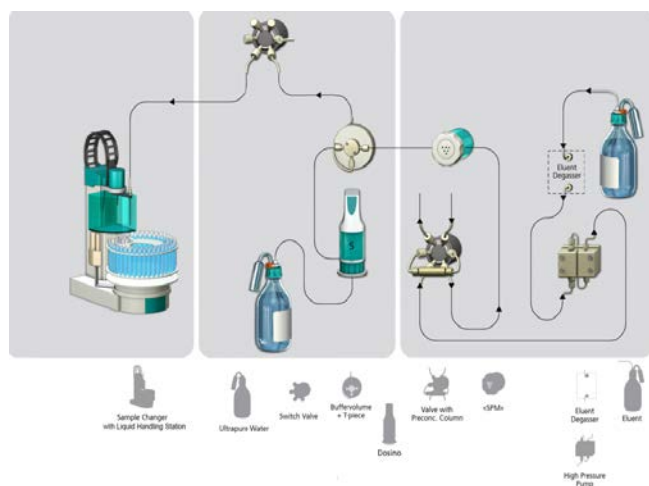
In the manual control window, make sure the needle at the Sample Processor is immersed in the Liquid Handling

Station (external position number 2). Also make sure that the injection valve position of the Sample Processor is on Fill. Set the pump speed of the high pressure pump to 1 mL/min to check the resulting backpressure. It should be below 0.5 MPa. If the pressure is too high, check your installation for any obstruction in the flow path. If no such hindrance can be found, reduce the flow of the high pressure pump gradually until the backpressure is below 0.5 MPa. The respective flow value has to be transferred to the method. In the subprograms "Sample" and "Blank" adjust the dosing rate of the time dosing program lines with the determined value.

Leave the pump running for at least 30 minutes.

If during this rinsing procedure no leaks have appeared, reconnect the capillaries where they were before and reinstall the SPM as shown in chapter 2.5. Rinse the Dosino and fill the transfer tubing by applying the "prepare" button under manual control for Dosino. Now dose 100 mL with the Dosino with a flow of 4 mL/min, through port 1 to rinse the Trap column and the flow path with ultrapure water.

Subsequently to this rinsing step, raise the needle out of the LQH Station so you can see its tip. Now dose 0.1 mL of ultrapure water through port 1 (flow of 2 mL/min). When the Dosino has finished dosing the water, observe the needle tip. If the water droplets don't stop in the following 5 seconds, but continue dripping, there is a connection in the flow path which is not completely tight and causes air gabs in the system. Retighten all connections including the needle at the Sample Processor and repeat this dosing test. When the dripping test has been passed, the installation is good to go.

In order to start the equilibration, go to the window work place, load the anion method, and press "Start HW".

Equilibrate the system until the baseline is stable.

3.3 User defined determination

In the window method, enter the ions and the concentrations of the required standards. The standard with the highest concentration (standard 1) has to be prepared when using the method ProfIC_Vario_11_Anions.imet. All other standards have to be a divisor of standard 1, e.g. all components of standard 2 have half the concentration of those in standard 1.

Now please add the correct Database in the Evaluation window (Evaluation - Results - Database) for the method.
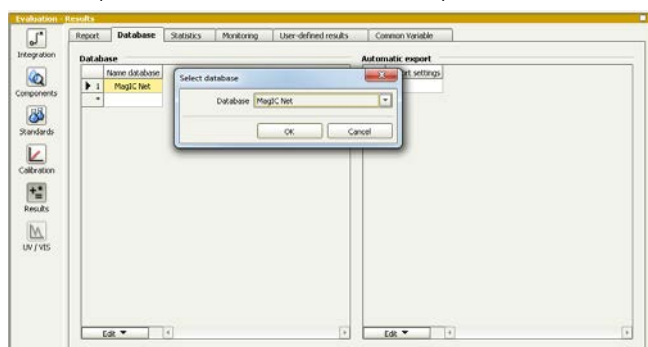
In the window work place, set up a "determination series", describing your samples by ident, vial number, sample type (standard, blank, check standard or sample etc.). Please note that for standards, always 2000 μL has to be written in the column "volume" of the sample table, because the method itself calculates the volume that actually has to be injected. After putting the analyte solutions onto the rack, press "start".

For evaluation and after recording the first chromatogram, check the retention times of your compounds. Since they depend on the performance of your column, you may have to adjust them in your method.
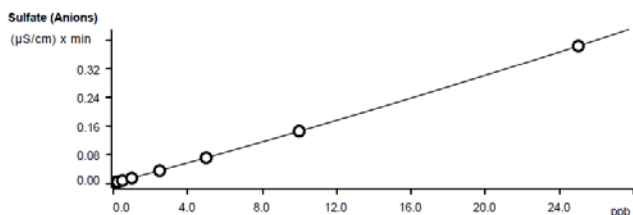
4. Exemplary measurement

Fluoride, chloride, nitrite, bromide, nitrate, sulfate, and phosphate (0.5 µg/L of each compound) were measured with preconcentration of 2000 µL.
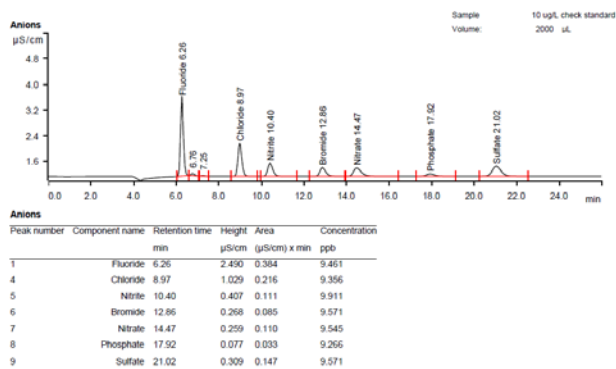
The calibration was performed within the concentration range of 0.1 – 25 µg/L and the quadratic curve type was used for evaluation. Correlation coefficients and standard deviations are listed in the following table. The calibration curve for sulfate is shown as an example.

	Correlation coefficient	Percentage standard deviation [%]
Fluoride	0.999941	2.13
Chloride	0.999993	0.73
Nitrite	0.999975	1.35
Bromide	0.999994	0.66
Nitrate	1.000000	0.18
Phosphate	0.999952	1.83
Sulfate	0.999998	0.36

The precision of 5 injections of 10 ppb standard anions was determined. For these measurements, a volume of 2000 µL was preconcentrated. The results are shown in the following table.

10 µg/L, 2000 µL

Ion	Mean value [µg/L]	Relative standard deviation [%]	Recovery [%]
Fluoride	9.50	0.3	95.0
Chloride	9.39	0.3	93.9
Nitrite	9.76	2.0	97.6
Bromide	9.61	0.2	96.1
Nitrate	9.58	0.3	95.8
Phosphate	9.13	1.6	91.3
Sulfate	9.62	0.3	96.2

5. Optional equipment

5.1. Eluent Production Module

The 941 Eluent Production Module creates fresh new eluent out of eluent concentrate and ultrapure water. For installation instructions and further information, please refer to the 941 Eluent Production Module Manual. The 941 can be connected to an ELGA flex ultra pure water system so that continuous water supply can be guaranteed. The ELGA can be further integrated into the system as well for all the ultra pure water supplies as visible below.
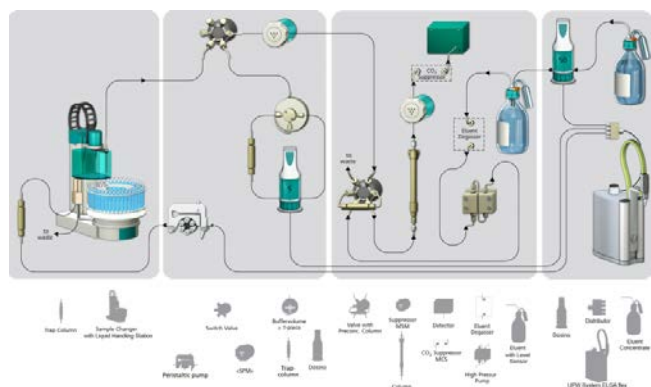
5.2. Alternative MSM rinsing and regeneration methods

For alternative suppressor rinsing and regeneration methods and setups, please refer to the AB-395.