Ω Metrohm

Installation Instruction for ProfIC Vario 4 AnCat

ProfIC Vario 4 AnCat is a Professional IC Vario system with Inline Dilution for the fully automatic determination of anions and cations simultaneously in concentrated solutions. «ProfIC Vario 4» can be used to analyze samples which are too concentrated to be determined directly.

Table of contents

1. Delivery Package

Delivered with ProfIC Vario 4 AnCat package:

Nr	Article no.	Article designation
IC		
1	2.940.2500	Professional IC Vario TWO/SeS/PP
Detector		
2	2.850.9010	IC Conductivity Detector
Sample processor		
1	2.858.0020	Professional Sample Processor - Pump
Accessories		
1	6.5330.120	IC Equipment: Inline Dilution
1	2.741.0010	741 Magnetic Stirrer
1	2.800.0010	800 Dosino
Optional		
1	6.6059.302	MagIC Net 3.0 Professional CD - 1 license
1	6.6059.303	MagIC Net 3.0 Multi - 3 licenses
1	6.2041.760	Sample rack 54 x 11 mL + 1 x 300 mL
1	6.2041.440	Sample rack 148 x 11 mL + 3 x 300 mL
1	6.2743.040	Sample tubes 2.5 mL
1	6.2743.050	Sample tubes 11 mL
1	6.2743.070	Stopper with perforation
1	6.1030.420	Metrosep A Supp 15 - 150/4.0
1	6.1030.500	Metrosep A Supp 15 Guard/4.0
1	6.1050.420	Metrosep C 4 - 150/4.0
1	6.1050.500	Metrosep C 4 Guard/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
1	2.800.0010	800 Dosino
1	6.5330.090	IC Equipment: Additional eluent on the Eluent Production Module
1	6.2769.110	Sensor empty 2L

2. Installation

Following is a detailed description to install «ProfIC Vario 4 AnCat», which describes an Inline Dilution. In order to get accurate results, the sample concentration must be within the limits of the calibration. In case of samples with analyte concentrations above the highest calibration standard, dilution of the sample is one suitable answer.

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 windows.

Now as soon as you connect a new 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 blocks in the instrument and connect the detector cables (anion detector into the "Detector 1" socket, the cation detector to "Detector 2"), remove the transport locking screws, connect the leak sensor cable and connect the drainage tubing.

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 later 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 two boxes with the Accessory Kits ONE, you will find all the accessories for setting up the eluent bottles. Please lead the aspiration tubes for the eluents through the M8 stopper, the earring and the eluent caps. 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 energy chain close to the swing head by using a screwdriver to unfix it. In its place, connect the transfer tubing with holder 2 x M6 / 10 mL (6.1562.130). Then connect one end of the transfer tubing onto the swing arm, where the aspiration tip is connected with the help of the adapter UNF 10/32 outer / M6 inner (6.2744.200).

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 tube from the inside and outside. The second unit is used for dilution of samples and standards. In the present case, both units will be used.

Before adding the Liquid Handling Station onto the Sample Processor insert the stirrer into the socket of the LQH Station. This is done according to the description in the Liquid Handling Station manual (chapter 2). Subsequently, it is mounted onto the left side of the Sample Processor. For installation instructions, please refer to the Liquid Handling Station manual. All parts are included in the IC Equipment: Inline Dilution (6.5330.120). Please make sure that the Liquid Handling Station (LQH 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, a stopper (6.2744.060) is used to close the outlet of the inner compartment of the rinsing area. The waste tubing (6.1801.120) is fixed at the bottom of the rinsing station to drain the waste water.

Ultra pure water for rinsing will be supplied by a peristaltic pump. For this, please connect the coupling nozzle

(6.2744.034) on one end and the coupling nozzle with security device (6.2744.160) on the other of the pump tubing white/white (6.1826.360) supplied with the Sample Processor. This pump tubing needs to be installed at the 858 Sample Processor with the security nozzle on the pressure side after the pump. Please fill the two 2 Liter bottles with ultra pure water. One will be needed for the dilution and will be mentioned in the next paragraph.

The second one should be closed with a cap (6.1602.150) and a capillary led to the aspirating side of the peristaltic pump. A capillary is lead from the pump tubing to the PEEK pressure screw on the bottom of the Liquid Handling Station (Number 2 on the following schemata). Between the peristaltic pump and the LQH Station, an I Trap (6.1014.200) can be installed into the flow path to cleanse the water before it goes into the IC system. The I-Trap is not included in the IC Equipment: Inline Dilution kit (6.5330.120). It would have to be ordered separately.
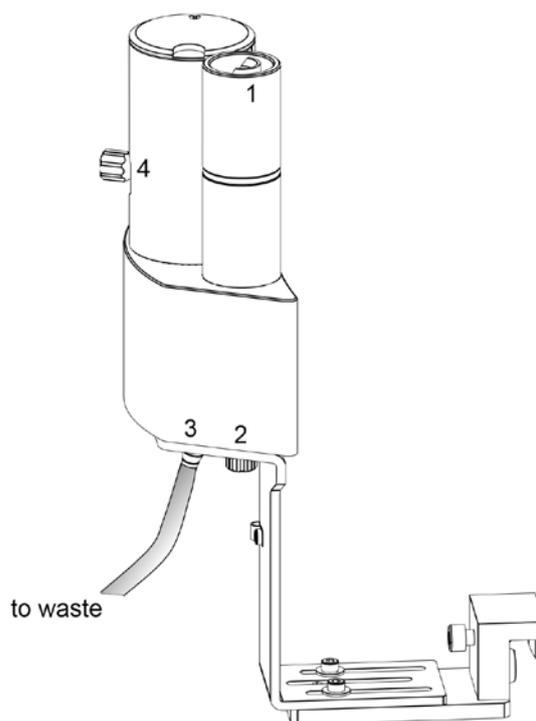
2.4.1.2. Dilution unit and 800 Dosino

At port 1 of the 2 mL Dosing Unit, please connect the loose end of the transfer tubing (6.1841.000). Port 2 is used for ultrapure water supply; therefore please screw the FEP aspiration tubing (6.1819.110) into Port 2 on the bottom of the Dosing Unit. Then fix the Dosing Unit onto the second 2 liter bottle with ultra pure water. Adjust the Dosino on top and connect its cable on the backside of the 858 Sample Processor on MSB1. Please make sure that the 858 is switched off!

The stirrer cable is plugged in at the tower into the ⚓ - socket. Capillary connections are carried out according to the following scheme.
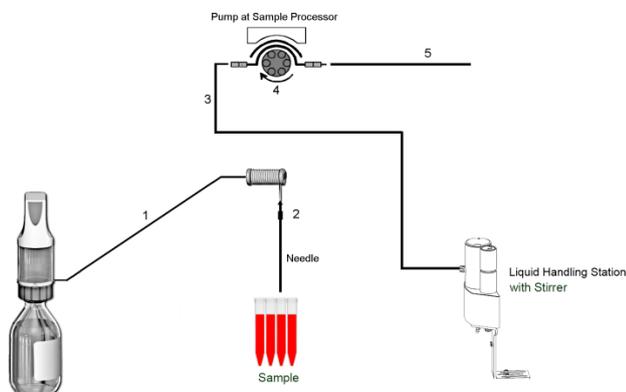
1. Transfer Tubing with holder 2 x M6 / 10 mL (6.1562.130) to be connected to Dosino port 1. The holder can be fixed as a chain element at the Tower. The other end is fixed to Adapter UNF 10/32 outer / M6 inner (6.2744.200) (see 2.)

2. Adapter UNF 10/32 outer / M6 inner (6.2744.200) for the connection between the needle and the transfer tubing

3. PTFE capillary 0.97 mm i.d. / 40 cm (6.1803.070) connected to the grey/grey pump tubing with a coupling nozzle (6.2744.034). Its other end is connected to position 4 on the side of the LQH Station with a PEEK pressure screw (6.2744.010)

4. Pump tubing LFL (gray/gray), 3 stoppers (6.1826.380)

5. The PTFE capillary 0.97 mm i.d. / 1 m (6.1803.080) is connected to the anion IC valve position 1. The other end is attached to the gray/gray pump tubing with the help of a nozzle coupling with security device (6.2744.160)

2.5. Interconnection of devices

The whole setup of the ProfIC Vario 4 AnCat packet is depicted here:
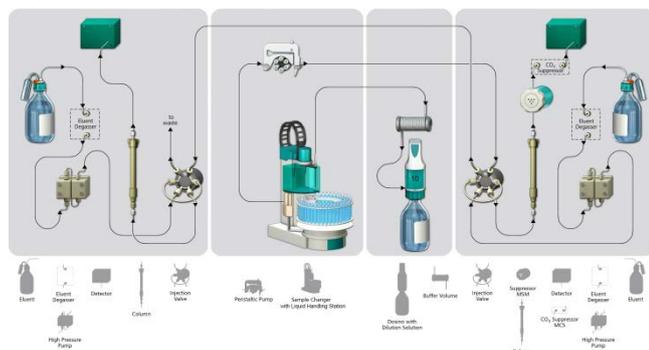
Prepare and degas two eluents suitable for the anion and cation columns you want to use (see column manual). For Metrosep A Supp 15 - 150/4.0 a solution of 5.0 mmol/L Na_2CO_3 / 0.3 mmol/L NaOH is recommended. For Metrosep C 4 - 150/4.0 a solution of 1.7 mmol/L HNO_3 / 0.7 mmol/L dipicolinic acid (DPA, 2,6-Pyridinedicarboxylic acid) is adequate.

Provide the regeneration solution for the MSM Suppressor (100 mmol/L sulfuric acid).

2.6. 940 Professional IC Vario

Capillaries are connected according to the following list and the diagram below:

1. Connection to the eluent bottle for anion determination

2. Capillary to anion separation column

3. MSM inlet capillary – labeled with *in*

4. MSM outlet capillary – labeled with *out,* which can be connected to the MCS with a long PEEK pressure screws (6.2477.090) or directly to the detector (5) using a coupling (6.2744.040).

5. Capillary from MCS to anion detector, long PEEK pressure screws (6.2744.090) are used for the MCS connection

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

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 (6.1826.420). 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 (21)

8. MSM regeneration and rinsing solution outlet capillaries – labeled with *waste reg.* and *waste rins.* – to be connected to the waste collector

9. Connection to the eluent bottle for cation determination

10. Capillary to cation separation column

11. Capillary from cation separation column to cation detector

12. Detector outlet capillary, connected to the waste collector

13. Sample aspiration capillary (PTFE, 0.5 mm ID), coming from the peristaltic pump tubing of the 858 (Nr.5 of chapter 2.4.1.2) connected to the anion valve (position 1)

14. Optional sample degasser

15. Capillary connection (PTFE, 0.5 mm ID) between anion valve (position 2) and cation valve (position 1)

16. Capillary to the waste collector

17. Coupling 2xUNF 10/32 PEEK (6.2744.040)

18. PEEK pressure screw short (6.2744.070)

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

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

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

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

23. The UNF 10/32 couplings (6.2744.040) are installed instead of the columns to rinse the system with eluent. After rinsing the columns are installed.
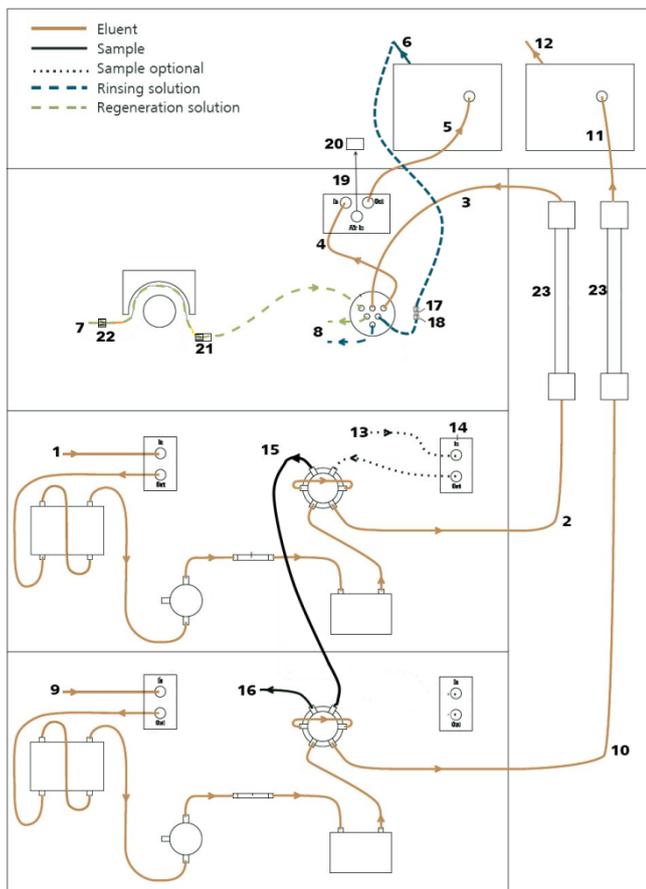
Make sure that all outlet capillaries are put into the waste collector (detector outlet, sample waste, suppressor waste, …).

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):
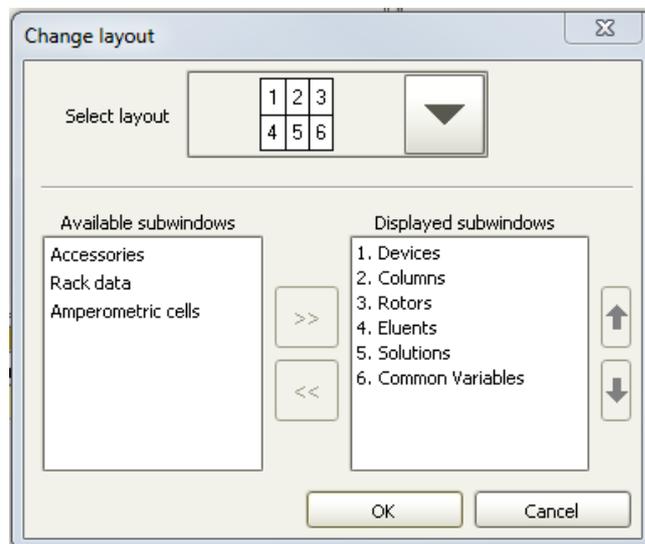
Now please connect the USB cables from the instruments and turn their power on. Connected USB devices are automatically recognized when MagIC Net is started. After confirmation of the automatically generated requests, the devices and columns are stored in the configuration. The devices are predefined 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).

Accept the request to add the Dosino connected to the 940, name the Dosino solution "UPW" (for ultra pure water) and chose "Dosino" in the "Use" dropdown menu.

In the window configuration, the 940 Professional IC Vario, the 858 Professional Sample Processor and the columns Metrosep A Supp 15 - 150/4.0 and Metrosep C 4 - 150/4.0 are visible. Add and define the eluents, the suppressor solution and the MSM Rotor.

The settings for the rack require a "work position" fitting to the respective rack (e.g. 125 mm for rack 6.2041.760).
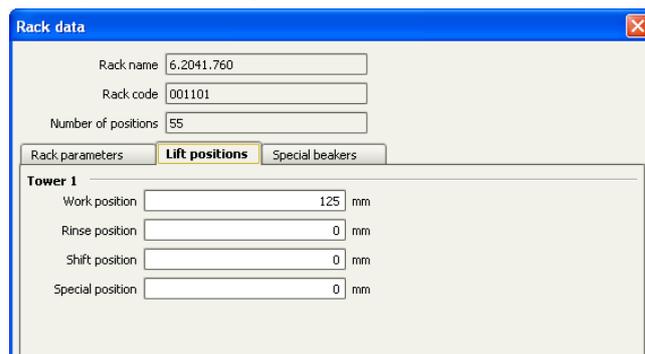
For using the Liquid Handling Station, it is necessary to define external positions for the needle. This is done in the configuration of the Sample Processor in the subtab "Tower". Please define the external positions 1, 2 and 3 according to the picture below. Attention: The angles are only approximate values, for fine tuning, please use the manual control to find the correct values for this system and adjust them then in the configuration.

For the Dosino, the exact lengths and diameters of the tubings used need to be added for it to work properly. This can be done in the configuration as follows:

Please add a common variable called: "end volume" and set its value to 7 mL.

3.2. Method adjustment

For this setup the ProfIC Vario 4 Anion and Cation methods need to be adapted to suit the parallel AnCat analysis. For this, first import the methods for ProfIC Vario 4 Anion and ProfIC Vario 4 Cation 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_4 _Anion.imet and

MagICNet\examples\methods\ ProfIC Vario \ProfIC Vario_4 _Cation.imet

In the window method, open the ProfIC Vario 4 Anion method and the ProfIC Vario 4 Cation method.

3.2.1. Anion method

For starters, your devices sub window should look like this:

Open the menu of the 940 by using the right mouse click on the 940 device. In the window "Edit", please check that the name of the instrument is the one you have given your 940.2500 as you've added it to your configuration. If the name is different, please adapt it here. Change the module names as following and save the changes.

Close the window and delete the 940 from your instrumentation.

Following this step, add a new device and chose the 2.940.2500 from your device table. Now, your method should look like this:

Now assign the following equipment: Assign the suppressor solution to the IC peristaltic pump and set the speed rate to 1, add your Rotor to the MSM and activate the automatic stepping all 10 minutes, define the rack of the connected sample processor, assign the eluent to the anion IC pump (Pump 1) and define reasonable start-up times e.g. 2 minutes. Set the temperature of the column oven to 45°C.

In the analysis link the corresponding pump (Pump 1), data source (Conductivity Detector 1, Conductivity), injection valve (Injector 1) and column.

Deactivate all parts of the 940 that are not needed for the anion analysis (Detector 2, Pump 2, Injector 2)

Perform a method test and save the method.

3.2.2. Cation method

For starters, your devices sub window should look like this:

Open the menu of the 940 by using the right mouse click on the 940 device. In the window "Edit", please check that the name of the instrument is the one you have given your 940.2500 as you've added it to your configuration. If the name is different, please adapt it here. Change the module names as following and save the changes.

Close the window and delete the 940 from your instrumentation.

Following this step, add a new device and chose the 2.940.2500 from your device table. Now, your method should look like this:

Now assign the following equipment: Define the rack of the connected sample processor, assign the eluent to the IC pump (Pump 2) and define a reasonable start-up time, e.g. 2 minutes. In the analysis link the corresponding pump (Pump 2), (Conductivity Detector 2, Conductivity), injection valve (Injector 2) and column. . Set the temperature of the column oven to 45°C.

Deactivate all parts of the 940 that are not needed for the anion analysis (Detector 1, Pump 1, Injector 1, MSM, MCS, Peristaltic Pump)

Perform a method test and save the method.

3.3. Purge of the system

Before inserting the columns, disconnect the Out capillary of the 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 precolumns for 10 minutes by leading the outlets directly into the waste. Afterwards connect the analytical columns and flush them likewise for 10 minutes. Only afterward the columns are completely connected to the flow path of the eluent (see also 940 manual). Start the peristaltic pump and adjust the pressure of the lever on the tubing, in order to see the droplets of suppressor regeneration solution drop into the waste collector. Rinse the Dosino and fill the transfer tubing by applying the "prepare" button under manual control.

In order to start the equilibration, load the methods in two separate workplaces, and press "Start HW".

Equilibrate the system until the baseline is stable.

3.4. User defined determination

In the window method, under evaluation, enter the ions and the concentrations of the required standards in both, the cation and the anion method. You can either calibrate with a set of calibration standards, or you can calibrate with only one standard for anions and one for cations and diluting these two to different calibration levels.

If you wish to calibrate by diluting one standard solution, the standard with the highest concentration has to be prepared and labeled as standard 1 in this method. All other standards have to be divisors of standard 1, e.g. all components of standard 2 have half the concentration of those in standard 1, all components of standard 10 have a tenth of the standard 1 concentration.

Always use the standard number as the factor that lies between the two solutions. Make sure to always fix the dilution factor = 1 for all the standards in your determination table.

Now please add the correct Database in the Evaluation window (Evaluation - Results - Database) for the method.
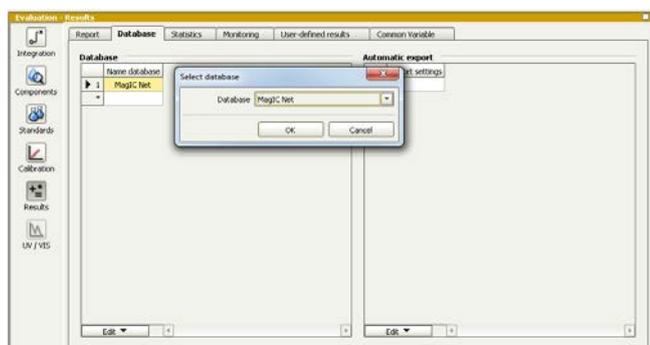
In the window work place for anions, set up a "determination series" for the anions, describing your samples by ident, vial number, sample type (standard, blank, or sample etc.).

In the field dilution, indicate the dilution you wish to apply to your sample, e.g "2" if you want to dilute your sample 2 times. If no dilution is required or a standard is measured, write 1. After putting the analyte solutions onto the rack, press "start".

In the second work place, set up a "determination series" for the cations, describing your samples by ident, vial number, sample type (standard, blank, or sample etc.).

In the field dilution, indicate the dilution you wish to apply to your sample, e.g "2" if you want to dilute your sample 2 times. If no dilution is required or a standard is measured, write 1. After putting the analyte solutions onto the rack, press "start".

For evaluation and after recording the first chromatograms, check the retention times of your compounds. Since they depend on the performance of your columns, you may have to adjust them in your methods.

4. Exemplary measurement

4.1. Anion method

This data was recorded using a MSM Rotor A with the Adapter Vario on a Metrosep A Supp 15 - 150/4.0 column.

Fluoride, chloride, nitrite, bromide, nitrate, sulfate, and phosphate were measured by direct injection of 0.2 ppm (upper chromatogram) and by 10-fold dilution of 2 ppm (lower chromatogram).
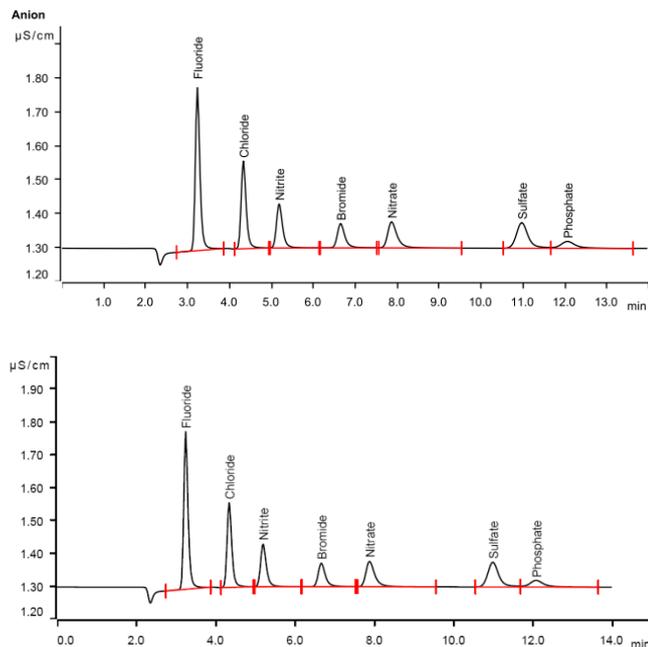
The calibration was performed with one standard containing standard anions of 20 ppm each. Within the concentration range of 0.2 – 10 ppm, a quadratic curve type was used for evaluation.

	Std. 2	Std. 4	Std. 10	Std. 20	Std. 40	Std. 100
Fluoride	10	5	2	1	0.5	0.2
Chloride	10	5	2	1	0.5	0.2
Nitrite	10	5	2	1	0.5	0.2
Bromide	10	5	2	1	0.5	0.2
Nitrate	10	5	2	1	0.5	0.2
Sulfate	10	5	2	1	0.5	0.2
Phosphate	10	5	2	1	0.5	0.2

Correlation coefficients and standard deviations are listed in the following table. The calibration curve for fluoride is shown as an example.

	Correlation coefficient	Percentage standard deviation [%]
Fluoride	0.99998	0.988
Chloride	0.99996	1.479
Nitrite	0.999935	1.899
Bromide	0.999975	1.17
Nitrate	0.999976	1.161
Sulfate	0.99997	1.288
Phosphate	0.999959	1.52

Function:	$A = -0.0306375 + 0.0214180 \times Q - 6.95047\text{E-}6 \times Q^2$
Relative standard deviation	0.988%
Correlation coefficient	0.999980
Curve type	Quadratic
Weighting	1
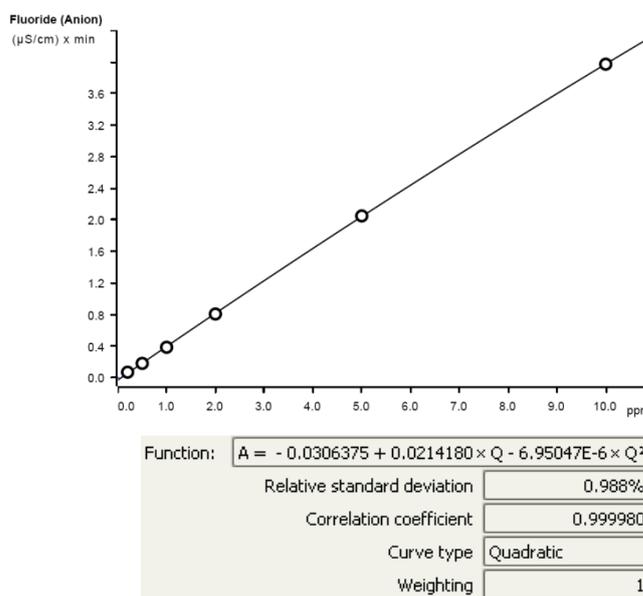
The precision of 10 injections of the 10 ppm standard was determined (Dilution factor 2). The results are shown in the following table and an exemplary trend chart is shown for Nitrite and Phosphate.

Ion	Mean value [ppm]	Standard deviation	
		relative [%]	absolute [ppm]
Fluoride	9.982	0.110	0.011
Chloride	9.981	0.130	0.013
Nitrite	9.927	0.504	0.050
Bromide	9.988	0.140	0.014
Nitrate	9.991	0.150	0.015
Sulfate	9.972	0.130	0.013
Phosphate	9.967	0.130	0.013

Nitrite

Phosphate
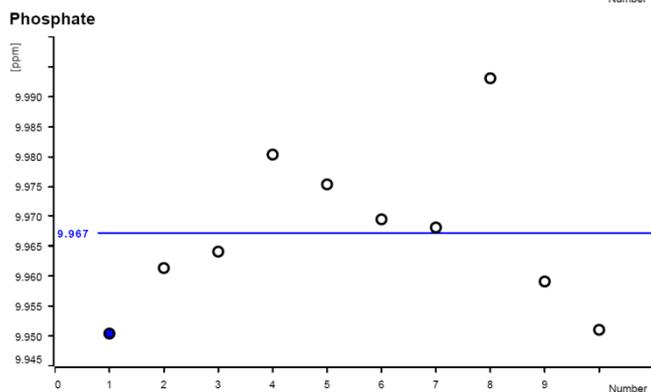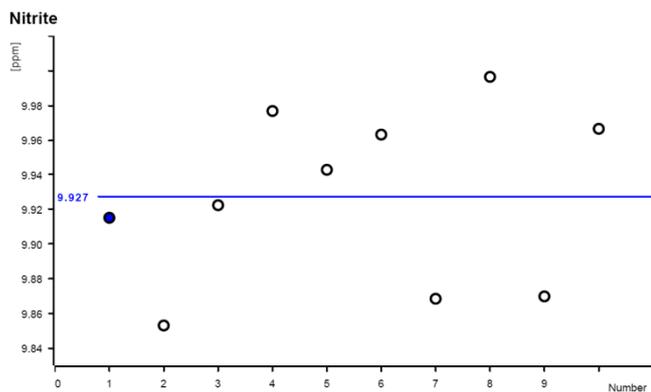
4.2. Cation method

Lithium, sodium, ammonium, potassium, manganese, calcium, and magnesium were measured by direct injection of 0.5 ppm (upper chromatogram) and by 10-fold dilution of 5 ppm (lower chromatogram).
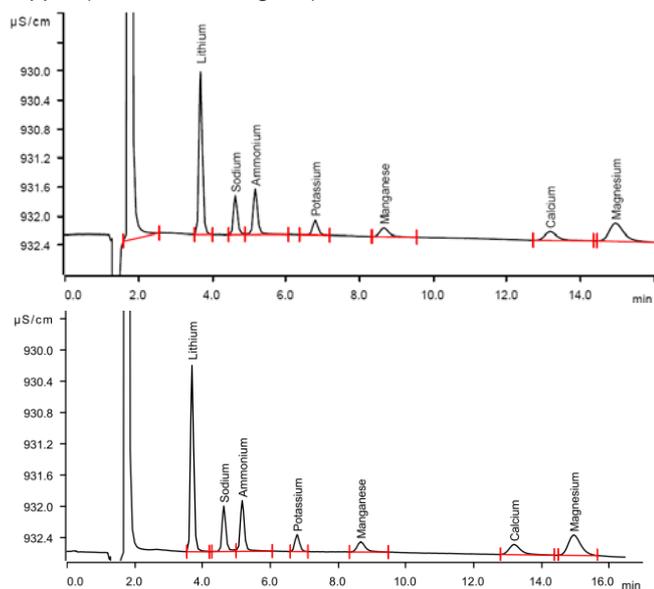
The calibration was performed with one standard containing standard cations of 20 ppm each. Within the concentration range of 0.2 – 10 ppm, a linear curve type was used for evaluation. Correlation coefficients and standard deviations

are listed in the following table. The calibration curve for lithium is shown as an example.

	Std. 2	Std. 4	Std. 10	Std. 20	Std. 40	Std. 100
Fluoride	10	5	2	1	0.5	0.2
Chloride	10	5	2	1	0.5	0.2
Nitrite	10	5	2	1	0.5	0.2
Bromide	10	5	2	1	0.5	0.2
Nitrate	10	5	2	1	0.5	0.2
Sulfate	10	5	2	1	0.5	0.2
Phosphate	10	5	2	1	0.5	0.2

	Correlation coefficient	Percentage standard deviation [%]
Lithium	0.999993	0.513
Sodium	0.999977	0.915
Ammonium	0.999998	0.298
Potassium	0.999985	0.76
Manganese	0.999755	3.133
Calcium	0.999917	1.798
Magnesium	0.999975	0.971

Lithium (Cation)
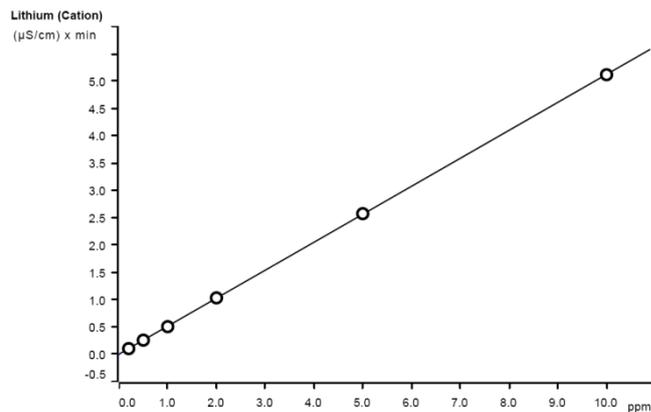
Function:	$A = 2.96510E\text{-}3 + 0.0255841 \times Q$	
	Relative standard deviation	0.513%
	Correlation coefficient	0.999993
	Curve type	Linear
	Weighting	1

The precision of 10 injections of a 10 ppm standard solution was determined (Dilution factor 2). The results are shown in the following table and an exemplary trend chart is shown for Lithium and Ammonium.

Ion	Mean value [ppm]	Standard deviation	
		relative [%]	absolute [ppm]
Lithium	10.756	0.033	0.307
Sodium	10.690	0.055	0.514
Ammonium	10.572	0.100	0.946
Potassium	10.661	0.044	0.413
Manganese	10.744	0.050	0.465
Calcium	11.058	0.046	0.416
Magnesium	11.055	0.038	0.344

Lithium
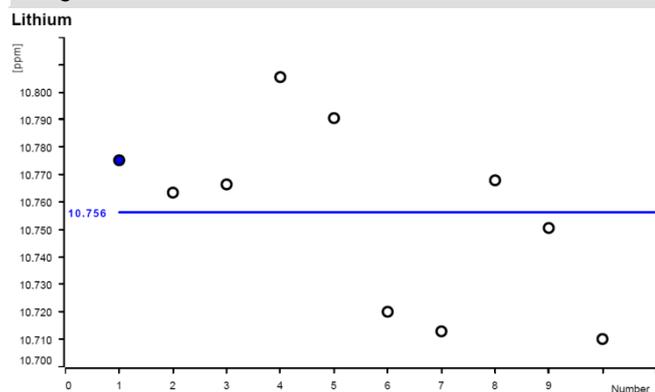
Ammonium

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.

5.2. Alternative MSM rinsing and regeneration methods

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

5.3. Acidification of cation samples

In the AB 378 no acidification of cation samples is performed. If such an acidification is wanted, it is possible to adapt the setup according to AW CH6-0929-032008 and add a defined quantity of nitric acid relative to the diluted volume of the sample for cation analysis. For this, you will need the following equipment:

Quantity	Article no.	Article designation
1	2.800.0010	800 Dosino
1	6.3032.120	Dosing Unit 2 mL
1	6.2744.200	Adapter UNF 10/32 outer / M6 inner
1	6.1808.030	Round glass bottle / 1000 mL / GL 45

The 1 L round glass bottle has to be filled with 100 mmol/L nitric acid. Please screw the FEP aspiration tube M6, which can be found in the equipment of the Dosing Unit, on port 2 of the 2 mL Dosing Unit, then fix the DU on top of the bottle with nitric acid. The Dosino is placed on top and plugged into an MSB port at the 858 Professional Sample Processor (858 must be turned **off**!).

The bottle with the Dosino should be placed on the left side of the Sample Processor, close to the Liquid Handling Station. For transfer of the nitric acid into the dilution compartment, please connect a PEEK capillary (i.D. 0.5 mm; 6.1831.180, delivered with the 6.5330.180 IC Equipment: MiPT) cut to an appropriate length, with the help of a PEEK pressure screw and the adapter (6.2744.200) to port 1 of the Dosing Unit. The other end of the capillary has to be entered through the "Mickey Mouse"-hole in the cover of the dilution unit and fixed in one of the ears. (See following image).

Following the hardware installations, the method will need to be adapted according to the before mentioned AW.