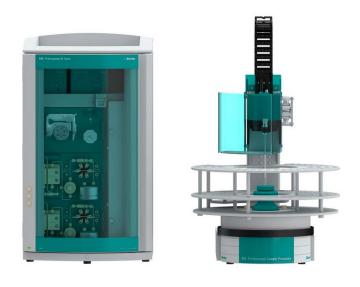
### **Application Bulletin 367**

### Installation instruction for ProfIC Vario 2 AnCat

ProfIC Vario 2 AnCat is a Professional IC Vario system for the fully automatic determination of anions and cations with Metrohm Inline Ultrafiltration. With «ProfIC Vario 2 AnCat» anions and cations can be determined both in parallel as well as independently in order to save time. Both systems use the same 858 Professional Sample Processor with pump, which is controlled by the MagIC Net software.



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#### 1. Delivery Package

Delivered with ProfIC Vario 2 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	
Acc	essories		
1	6.5330.110	IC Equipment: Inline Ultrafiltration	
San	nple processor		
1	2.858.0020	Professional Sample Processor - Pump	
Opt	tional		
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	6.2743.050	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	6.5330.090	IC Equipment: Additional eluent for 941	
1	2.800.0010	Dosino	
1	6.2769.110	Sensor empty 2L	

#### 2. Installation

Following is a detailed description of how to install ProfIC Vario 2 AnCat.

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).

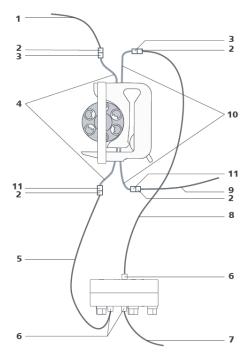
For the following installations, please shut down the IC again and disconnect the 858 from the power supply.

#### 2.4.1. Ultrafiltration cell

In this setup, an ultrafiltration cell is mounted in between the sample pickup (needle on Sample Processor) and IC system. For this, please mount the cell holder which is included in the IC Equipment: Ultrafiltration (6.5330.110) onto the Sample Processor on the right side of the tower. For detailed instructions, please refer to the Ultrafiltration Cell Manual.

The filter membrane is soaked in ultrapure water for preconditioning. After inserting the filter membrane and the sealing ring, the cell is tightly screwed together.

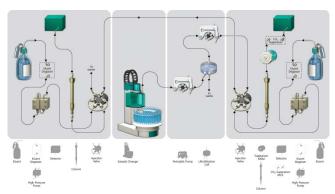
Capillary connections are shown in the illustration below. The flow must be higher at the sample side than at the filtrate side. That is why it is important to use a bigger pump tubing (yellow/yellow) and 0.97 mm i.D. capillaries on the sample side, and a thinner pump tubing (yellow/orange) and the shortest possible 0.5 mm i.D. capillaries on the filtrate side. Only use the PVDF screws in combination with the UF cell as the material of the cell is prone to break with PEEK screws.



- 1. PEEK capillary (6.1831.160) connected with the needle of the Sample Processor for aspirating the sample
- 2. PEEK pressure screw (6.2744.070)
- 3. Coupling olive (6.2744.030)
- 4. Pump tubing (6.1826.390) with yellow-yellow stoppers for conveying the sample
- 5. PTFE capillary (6.1803.060) for conveying the sample from the peristaltic pump to the ultrafiltration cell (0.97 mm i.D.)
- 6. PVDF pressure screw (6.2744.000) for connecting the capillaries to the ultrafiltration cell
- PTFE capillary (6.1803.080) for conveying the sample from the ultrafiltration cell to the waste collector (0.97 mm i.D.)
- 8. PTFE capillary (6.1803.050) for conveying the filtrate from the ultrafiltration cell to the peristaltic pump
- 9. PEEK capillary (6.1831.060) for conveying the filtrate from the peristaltic pump to the injection valve for anion analysis in the IC instrument (number 13 in chapter 2.6)
- 10. Pump tubing (6.1826.320) with yellow-orange stoppers for conveying the filtrate
- 11. Pump tubing connection with safety device (6.2744.160)

#### 2.5. Interconnection of devices

The whole setup of the ProfIC Vario 2 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<sub>2</sub>CO<sub>3</sub> / 0.3 mmol/L NaOH is recommended. For Metrosep C 4 - 150/4.0 a solution of 1.7 mmol/L HNO<sub>3</sub> / 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).

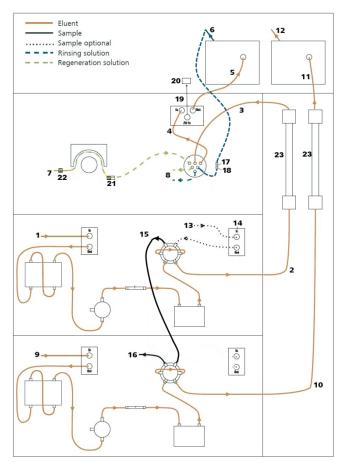
#### 2.6. 940 Professional IC Vario

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
- MSM outlet capillary labeled with *Out*, which can be connected to the MCS or directly to the detector (5) using a coupling (6.2744.040)
- Capillary from MCS to anion detector, long PEEK pressure screws (6.2744.090) are used for the MCS connection
- Anion detector outlet capillary connected to the MSM inlet capillary – labeled with *Rinsing solution* with the help of a coupling (6.2744.040)
- 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. 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
- Sample aspiration capillary (PTFE, 0.5 mm ID), coming from the ultra filtration cell and 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<sub>2</sub> adsorber cartridge
- 20. Luer coupling connect with short PEEK pressure screw to CO<sub>2</sub> 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.





The sample injection is at position 1 of the anion valve (see diagram of chapter 2.6, capillary number 13). A counter current flow of the sample with regard to the eluent flow is recommended in order to minimize diffusion and carry over. The outlet of anion valve is connected to the cation valve on position number 1. Make sure that all outlet capillaries are put into the waste collector.

#### 3. Software

#### 3.1. Configuration

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 setting on your computer).

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 and the suppressor solution.

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

Rack data			
Rack name	6.2041.760		
Rack code	001101		
Number of position:	55		
Rack parameters	Lift positions	Special beakers	
Tower 1			
Work position		125 mm	
Rinse position		0 mm	
Shift position		0 mm	
Special position		0 mm	

Also define a special beaker for rinsing the needle. Fill a beaker with ultrapure water and place it at the defined position on the rack.

Ra	k name	6.20	6.2041.760					
Rack code 001		0011	101					
Number of p	ositions	55						
Rack parameter	5	Lift po	sitions	Special b	eakers			
Special beaker	Rack p	siltion	Work pos	sition Tower 1	Work pos	sition Tower 2	Beaker radius	Beaker senso
1	55	;		125		0	off	off
2	0			0		0	off	off
3	0			0		0	off	off
4	0		0			0	off	off
5	0			0		0	off	off
6	0	(		0		0	off	off
7	0			0		0	off	off
				0		0	. 11	.07

#### 3.2. Method adjustment

In the window method, import the method for ProfIC Vario 2 AnCat from the installation CD: Go to File  $\rightarrow$  method manager and choose your method group. Afterwards, click on edit  $\rightarrow$  import and choose the pathway on the installation CD:

MagICnet\examples\methods\ ProfIC Vario\ProfIC\_Vario\_2\_ AnCat.imet).

In the window method, open the ProfIC Vario 2 AnCat method and assign the following equipment: Assign the suppressor solution to the IC peristaltic pump, define the rack of the connected sample processor, assign the eluents to the respective IC pumps, and assign the column Metrosep A Supp 15 - 150/4.0 to the analysis anion and the column Metrosep C 4 - 150/4.0 to the analysis cation. Adjust the temperature of the column oven to  $45^{\circ}$ C.

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.

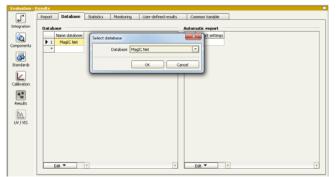
Now insert and rinse the precolumns for 10 minutes by leading the outlets directly into the waste. Afterwards connect the analytical columns and flush them likewise for 10 minutes. Only after these steps have been performed, the columns are completely connected to the flow path of the eluent (see also 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.

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

Equilibrate the system until the baseline is stable.

#### 3.4. User defined determination

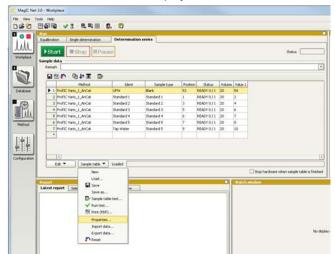
In the window method, under evaluation, enter the ions and the concentrations of the required standards. Now please add the correct Database in the Evaluation window (Evaluation - Results - Database) for the method.



In the window work place, set up one or two "determination series", depending on your method choice describing your samples by ident, vial number, sample type (standard, blank, or sample etc.). For quantitative analysis, samples subjected to cation analysis are normally acidified, whereas anion analysis is normally carried out with unmodified samples.

Hence for ProfIC Vario 2 AnCat, two sample vials are posed on the rack for each sample (an acidified one for cation determination, and an unmodified one for anion determination). According to the method ProfIC Vario 2 AnCat.imet, the position of the vial for anion determination should be defined in the column "sample position" and the position of the vial for cation determination should be defined the in the column "Value 1".

To be able to see and use the Value 1 of the determination table, click on the subcategory "Properties..." under Sample table in the run window. In the newly opened window, check the "Value 1" case under "Display".



Now you'll be able to add the cation vial position in your determination series.

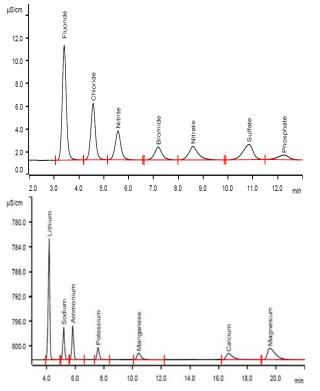
For evaluation and after recording the first chromatogram, check the retention times of your compounds. Since they depend on the performance of your column, you may have to adjust them in your method.



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#### 4. Exemplary measurement

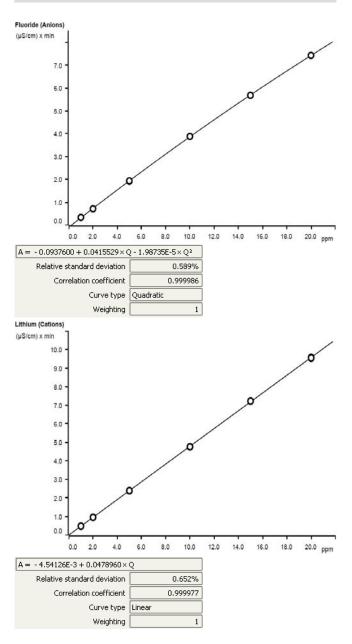
An exemplary measurement was performed, including the ions fluoride, chloride, nitrite, bromide nitrate, sulfate, phosphate, lithium, sodium, ammonium, potassium, manganese, calcium, and magnesium (5 mg/L each). Anions and cations were measured from separate sample vials with the sample for cation measurement being acidified by 100  $\mu$ L nitric acid (2 mol/L) per 100 mL. The two following chromatograms were recorded in parallel.



For the calibration, the average peak areas of 3 injections were evaluated. Within the concentration range of 1 - 20 mg/L, a quadratic curve type was used for evaluation of anions, and a linear curve type was used for evaluation of cations. Correlation coefficients and standard deviations are listed in the following table. The calibration curves for fluoride and lithium are shown as examples.

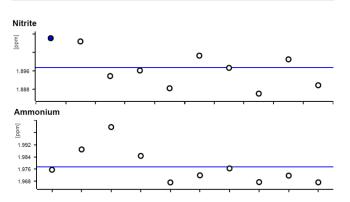
	Correlation coefficient	Percentage standard deviation [%]
Fluoride	0.999986	0.589
Chloride	0.999992	0.451
Nitrite	0.999979	0.754
Bromide	0.999968	0.940
Nitrate	0.999949	1.175
Sulfate	0.999824	2.241

Phosphate	0.999982	0.733
Lithium	0.999977	0.652
Sodium	0.999962	0.827
Ammonium	0.999992	0.374
Potassium	0.999994	0.339
Manganese	0.999962	0.914
Calcium	0.999964	0.910
Magnesium	0.999957	0.892



The precision of 10 injections of 2 mg/L standard ions was determined. The results are shown in the following table and an exemplary trend chart is shown for nitrite and ammonium.

lon	Standard deviation		
	relative [%]	absolute [mg/L]	
Fluoride	0.292	0.006	
Chloride	2.160	0.042	
Nitrite	0.647	0.013	
Bromide	0.176	0.003	
Nitrate	0.496	0.01	
Sulfate	0.306	0.006	
Phosphate	0.349	0.007	
Lithium	0.258	0.005	
Sodium	0.476	0.009	
Ammonium	0.488	0.01	
Potassium	0.353	0.007	
Manganese	2.918	0.06	
Calcium	3.175	0.063	
Magnesium	0.786	0.016	





#### 5. Optional equipment

#### 5.1. Liquid Handling Station

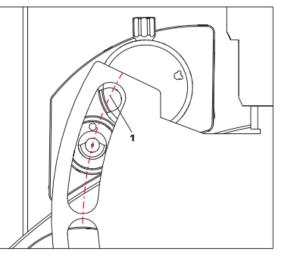
The Liquid Handling Station (LQH Station) consists of two function units: The rinsing and the dilution unit. As an addon it is mainly useful for rinsing the needle on the inside and outside, thus minimizing contamination.

The following accessories are needed:

Nr	Article no.	Article designation
1	6.1819.100	FEP aspiration tubing to 2 L bottle
1	2.800.0010	800 Dosino
1	6.3032.150	5 mL Dosing Unit
1	6.1014.200	Metrosep I Trap 1 - 100/4.0
1	6.1608.070	Eluent bottle /2 L / GL 45
1	6.1831.180	PEEK capillary i.D. 0.5 mm, 3 m
1	6.2744.010	Pressure screw 5x
1	6.2841.120	Liquid Handling Station left
1	6.2744.080	M6 thread/UNF 10/32 coupling

The installation of the LQH Station is done in two steps: First mount and align the Liquid Handling Station on the left hand side of the Sample Processor. To accomplish this, remove the sample rack and place the Liquid Handling Station on the black rail of the Sample Processor. Secure it temporarily in place with the screw and then loosen the small screws of the foot. The rack is replaced on the Sample Processor and the small screws are fixed in a way that the approximate distance between LHS and rack is 0.5-1 mm.

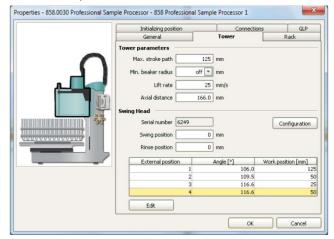
Now the Liquid Handling Station needs to be aligned with the retaining plate. For this, loosen the big screw again and move the Station underneath the retaining plate. Looking from above you should now be able to see the small hole of the rinsing unit and part of the big hole of the dilution vessel, similar to the following image.



When you have found the correct position, please tighten the screw to fix the LQH Station completely to the Sample Processor.

In a second step, the work positions for the sample tube need to be defined in the configuration.

Please go to the configuration of the Sample Processor. Under Tower it is possible to define external positions of the swing head. For rinsing purposes, only the External Position 1 (small inner tube) and the External Position 2 (outer tube of rinsing unit) have to be defined. The proposed angles are only guiding values; they need to be adapted for every system separately. This can be done easily by adjusting the angles in the manual control window. As soon as you have found the correct angles, make sure to save them in the configuration. The work positions are fixed for all of the different setups.



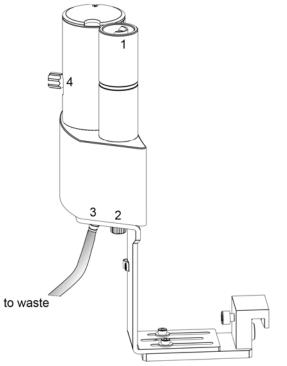
Mount the Dosino on the 5 mL Dosing Unit and connect the FEP aspiration tube M6 (6.1819.100) on Port 2 of the Dosino. Then, fill the 2 L bottle with ultrapure water and fix the Dosino with the Dosing Unit on it.

Disconnect the 858 Sample Processor from the energy source and plug in the Dosino at an MSB port of the 858. Reconnect the Sample Processor to the energy source.



Now, please connect the Dosino port 1 with an adapter (6.2744.080) and a capillary to the PEEK pressure screw (Nr. 2) on the Liquid Handling Station. Then use the Dosino to push water from below into the rinsing unit when needed. Here, it is recommended to install an I-Trap (6.1014.200) into the flow path of the water, in order that the water is cleansed from all impurities that could build in the water reservoir. During the rinsing the inner tube of the rinsing unit is meant to be filled constantly with fresh Ultrapure Water, so the aspiration needle can be thoroughly cleaned from the outside.

Please fix the PVC tubing (6.1801.120) on the disposal connector on the bottom of the Liquid Handling Station and lead its other end into a waste canister.



Number 4 of the picture above shows the attachment point of a capillary in case of a dilution feature and Number 1 indicates the rinsing entity.

The rinsing time program with the Liquid Handling Station works similar to the one for rinsing with a special beaker: Just swing to the wanted angle, go into work position and start the Dosino. Make sure to always dispose the waste in the external position 2.

#### 5.2. 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.

### 5.3. Alternative MSM rinsing and regeneration methods

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