Application Bulletin 369

Installation Instruction for ProfIC Vario 3 Cation

The ProfIC Vario 3 Cation is a Professional IC Vario system for the fully automated determination of non-suppressed cations or anions with Metrohm Inline Dialysis. «ProfIC Vario 3» safely masters all routine tasks in ion chromatography. It is easy to use and extremely reliable.

For the installation instructions for suppressed systems, please check AB-368.

Table of contents

1. Delivery Package

Delivered with ProfIC Vario 3 Cation package:

Nr	Article no.	Article designation
IC		
1	2.940.1110	Professional IC Vario ONE/Prep 1
Detector		
1	2.850.9010	IC Conductivity Detector
Accessories		
1	6.5330.100	IC Equipment: Inline Dialysis
Sample processor		
1	2.858.0020	Professional Sample Processor - Pump
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	6.2743.050	Sample tubes 11 mL
1	6.2743.070	Stopper with perforation
1	6.1050.420	Metrosep C 4 - 150/4.0
1	6.1050.500	Metrosep C 4 Guard/4.0
1	2.941.0010	Eluent Production Module
1	6.2769.110	Sensor empty 2L

2. Installation

Following is a detailed description of how to install ProfIC Vario 3 Cation.

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 block in the instrument and connect the detector cable, 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 box with the Accessory Kits 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 earring 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 stepwise 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.

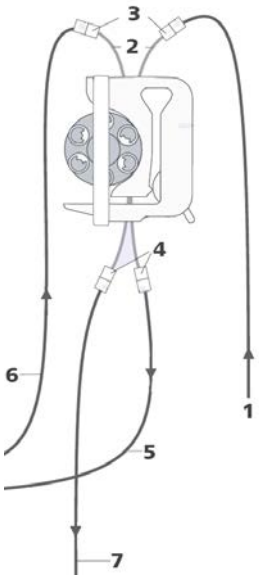
2.4.1. Dialysis cell

Inline dialysis is used for samples with a complex matrix (e.g. emulsions, samples containing fat and protein, body fluids or waste waters with high pollution loads). The dialysis is performed directly before injection of the sample into the IC. The main component of the equipment is the high-performance dialysis cell. Driven by a concentration gradient the ions diffuse out of the flowing sample through a semi-permeable membrane, into the stationary acceptor solution. After a user defined dialysis time, during which ion equilibrium between sample and acceptor solution should be reached, the acceptor solution is transferred to the sample loop and injected into the IC system. For more details, see the dialysis manual 8.110.8013.

The filter membrane is soaked in ultrapure water for preconditioning for about 5 min. After inserting the filter membrane (6.2714.010) and the o - ring (E3010111), the cell is screwed together (please refer to the manual chapters 1.4 and 2.1). Do not over tighten the screws, as this may damage the cell. The dialysis cell is placed with its assembly groove in the cell holder of the IC device, if a cell holder is present. Otherwise the dialysis cell can be placed onto an external holder (6.2057.130) in the detector chamber of your IC instrument.

A 2 L bottle of degassed 2 mmol/L nitric acid is provided as acceptor solution.

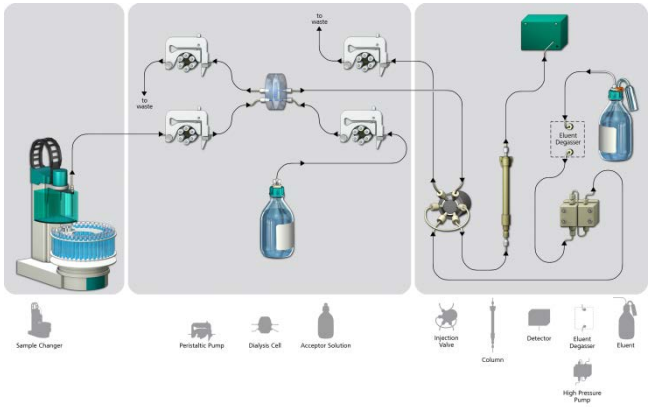
The peristaltic pump at the Sample Processor is used to transport the sample to the dialysis cell and from the cell to the waste. Capillaries are connected as shown in the following graph.



1. Sample-aspirating PEEK capillary (6.1831.160) connected to the needle
2. Two pump tubings (6.1826.340) with black-black stoppers
3. PEEK pressure screws (6.2744.070) and coupling olives (6.2744.034)
4. PEEK pressure screws (6.2744.070) and and pump tube connections with safety device (6.2744.160)
1. PTFE capillary (6.1803.040) to convey sample from peristaltic pump to the cell (refers to number 8 in chapter 2.6.)
2. PTFE capillary (6.1803.040) to convey sample from the cell to the peristaltic pump (refers to number 9 in chapter 2.6)
5. PTFE capillary (6.1803.040) to convey sample from the peristaltic pump to the waste

2.5. Interconnection of devices

The whole setup of the ProfIC Vario 3 Cation packet is depicted here:



Prepare and degas an eluent suitable for the column you want to use (see column manual). 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.

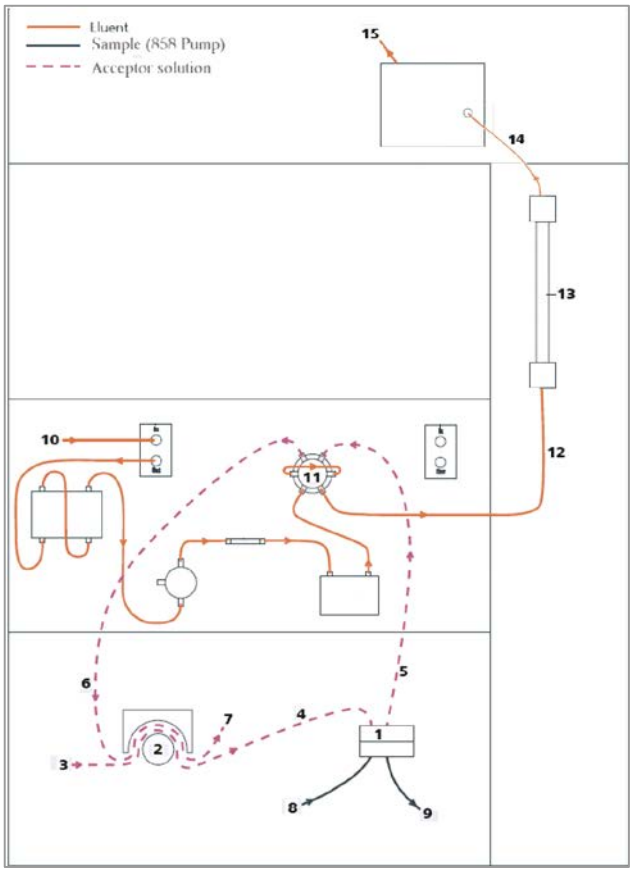
2.6. 940 Professional IC Vario

Capillary connections are shown in the illustration below:

1. Dialysis cell - all capillaries have to be connected with PVDF pressure screws to preserve the plexiglass cell.
2. Peristaltic pump for the acceptor solution, equipped with two yellow-orange pump tubings (6.1826.320)
3. PTFE capillary, 0.5 mm ID (6.1803.040) for supplying acceptor solution to the peristaltic pump
4. PTFE capillary, 0.5 mm ID (6.1803.040) to convey acceptor solution from peristaltic pump to the cell
5. **PEEK** capillary, 0.5 mm ID (6.1831.050) to convey acceptor solution from the cell to the injection valve. The length of **40 cm** is crucial for reproducible data.

6. PTFE capillary, 0.5 mm ID (6.1803.040) to convey the acceptor solution from injection valve to peristaltic pump
7. PTFE capillary, 0.5 mm ID (6.1803.040) to convey the acceptor solution from peristaltic pump to the waste collector
8. PTFE capillary, 0.5 mm ID (6.1803.040) to convey the sample from the peristaltic pump at the 858 to the cell
9. PTFE capillary, 0.5 mm ID (6.1803.040) to convey the sample from the cell to the peristaltic pump at the 858
10. Connection to the eluent bottle
11. Loop (20 µL) connected to the valve positions 3 and 6
12. Connection between valve and column
13. The UNF 10/32 coupling (6.2744.040) is installed instead of the column to rinse the system with eluent. After rinsing, the column (with pre-column) is installed.
14. Detector inlet capillary
15. Detector outlet capillary connected to waste collector

Make sure that the flow is directed as indicated by the arrows in the illustration.



The sample injection is at position 1 of the valve. A counter current flow of the sample with regard to the eluent flow is recommended, in order to minimize diffusion and carry over. Make sure that all outlet capillaries are put into the waste collector.

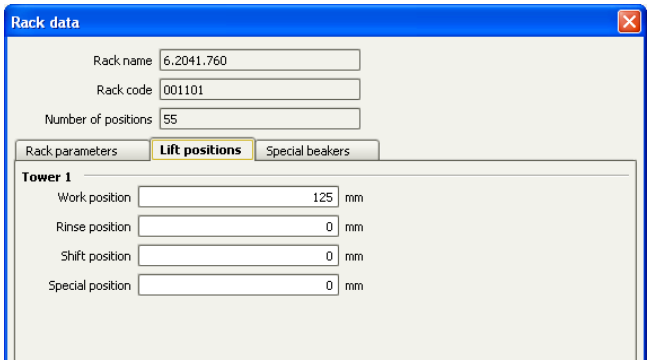
3. Software

3.1. Configuration

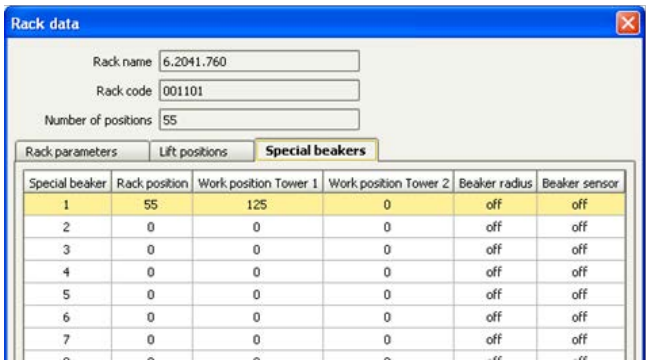
Now please connect the USB cables from the instruments and turn their power on. Connected USB devices are automatically recognized when MagIC Net is started. After confirmation of the automatically generated requests, the devices and columns are stored in the configuration. The devices are predefined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

In the window configuration, the 940 Professional IC Vario, the 858 Professional Sample Processor and the column Metrosep C 4 - 150/4.0 are visible. Add and define the eluent.

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



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.



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 3 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_3_ Cation.imet

In the window ”Method”, open the ProfIC Vario 3 Cation method and assign the following equipment: Assign the column Metrosep C 4 - 150/4.0 to the analysis cation, define the rack of the connected sample processor, and assign the eluent to the IC pump. Perform a method test and save the method.

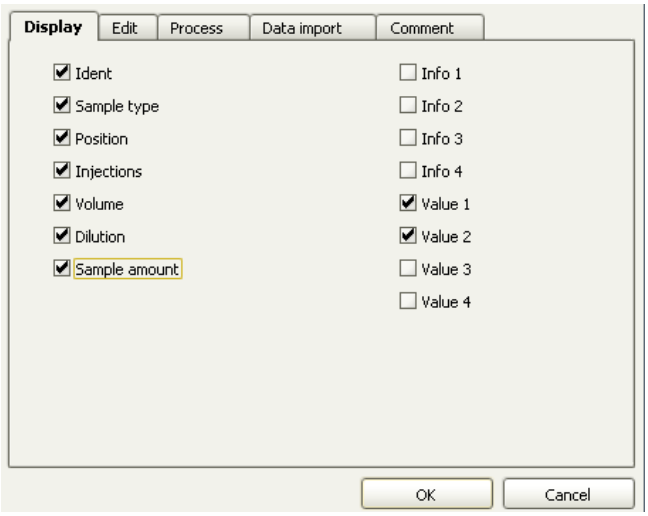
3.3. Purge of the system

Before inserting the column, flush the system for about 10 minutes and get rid of air bubbles (by using the purge valve and syringe).

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).

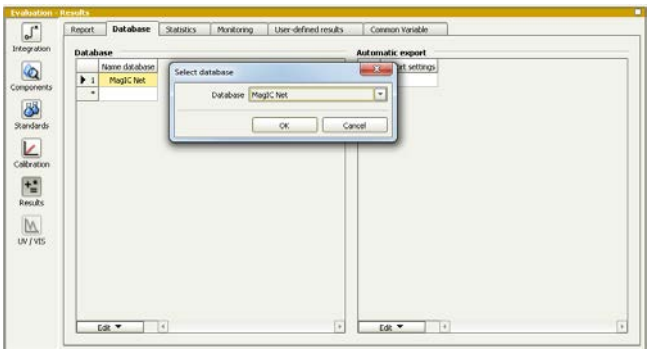
In order to start the equilibration, go to the window work place, load your method, and press “Start HW”. For the purpose of displaying value 1 and 2 in the sample table, go to “View” → properties → properties Run window and tick Value 1 and Value 2.

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.



For the dialysis setup it is imperative to determine the optimal settings for your individual setup. The dialysis time (Value 1) depends on your analytes, the matrix and the membrane used, as well as the temperature. The transfer time (Value 2) depends on the exact length of your capillary from the dialysis cell to the sample injection loop.

First choose a reasonable dialysis time at which you expect analyte signals of acceptable height, e.g. 10 minutes. Then start to optimize the transfer time: Measure the same sample with different transfer times, starting from around 20 s, and going up to around 45 s in increments equal or smaller than 5 seconds, while the dialysis time is kept constant. Screen your data for the determination with the maximum peak area of your compounds of interest, which is easiest using the detailed overview in MagIC Net. The optimum is expected at around 30 s. In a second step optimize the dialysis time: Start

with e.g. 8 min and add increments of 1 min, while the transfer time is kept constant with the previously determined optimized value. The optimal time for dialysis is reached when peak areas do not further increase. Please refer to the manual for a detailed description (8.110.8393; chapter 3).

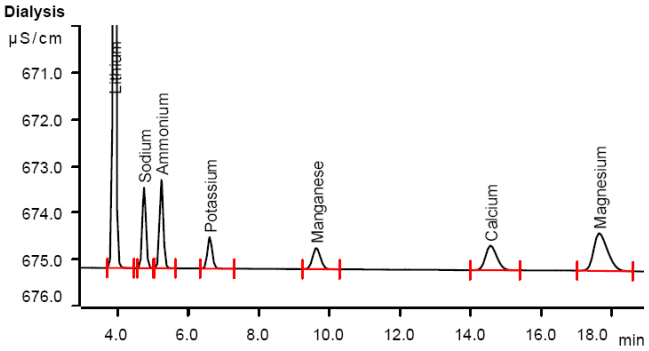
In the window work place, set up a “determination series”, describing your samples by ident, vial number, sample type (standard, blank, or sample etc.). After putting the analyte solutions onto the rack, press “start”.

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

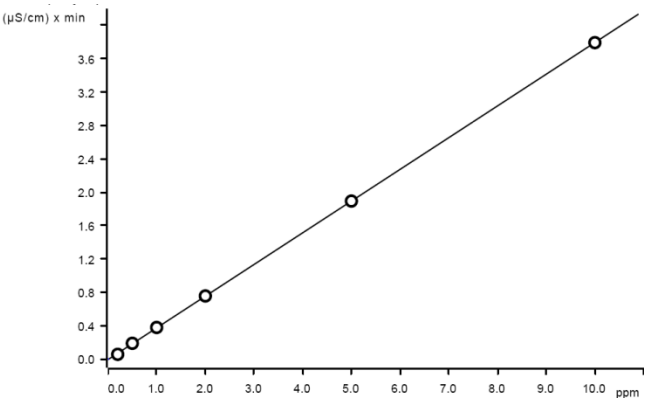
Lithium, sodium, ammonium, potassium, manganese, calcium, and magnesium (2 mg/L of each compound) were measured after Inline Dialysis.

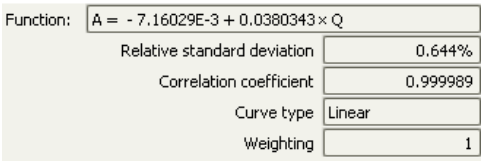
With 0.7 mM dipicolinic acid (DPA, 2,6-Pyridinedicarboxylic acid) calcium and manganese elute faster than magnesium.



The calibration was performed within the concentration range of 0.2 – 10 mg/L, a linear curve type was used for evaluation and 10 µL had been injected. Correlation coefficients and standard deviations are listed in the following table. The calibration curve for lithium is shown as an example.

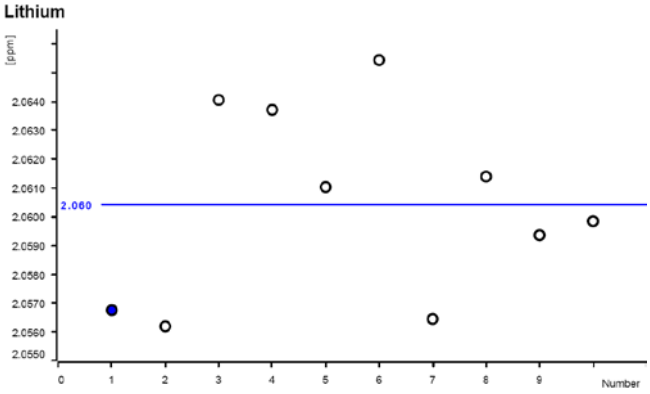
	Correlation coefficient	Percentage standard deviation [%]
Lithium	0.999989	0.644
Sodium	0.999994	0.489
Ammonium	0.999988	0.684
Potassium	0.999947	1.447
Manganese	0.999994	0.486
Calcium	0.999970	1.058
Magnesium	0.999964	1.155

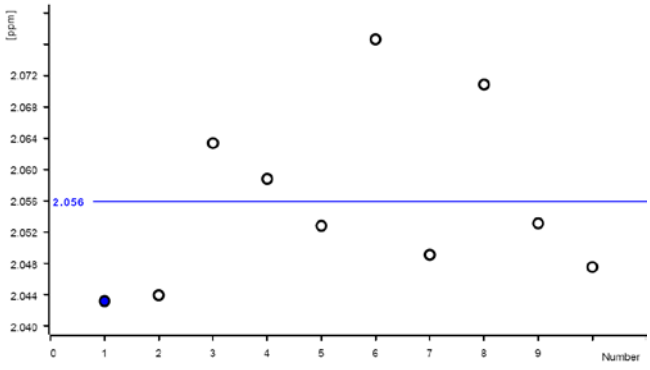




The precision of 10 injections of a 2 mg/L cation standard was determined. The results are shown in the following table and an exemplary trend chart is shown for Lithium and Manganese.

Ion		Standard deviation	
	Mean value [2 mg/L]	relative [%]	absolute [mg/L]
Lithium	2.060	0.146	0.003
Sodium	2.030	0.148	0.003
Ammonium	2.053	0.438	0.009
Potassium	2.044	0.636	0.013
Manganese	2.056	0.535	0.011
Calcium	2.053	0.682	0.014
Magnesium	2.052	0.682	0.014





5. Optional equipment

5.1. Liquid Handling Station

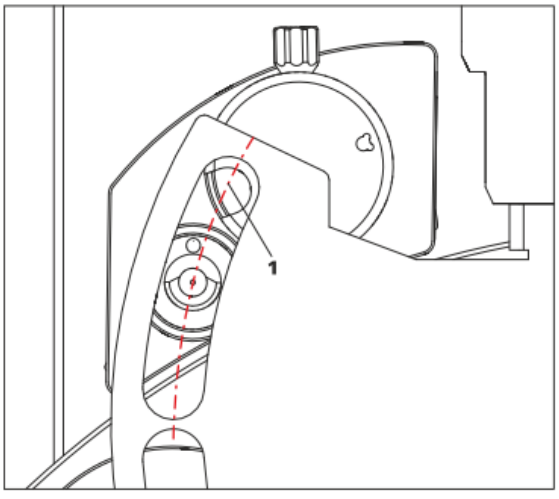
The Liquid Handling Station (LQH Station) consists of two function units: The rinsing and the dilution unit. As an add-on it is mainly useful for rinsing the needle on the inside and outside, thus minimizing contamination.

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.

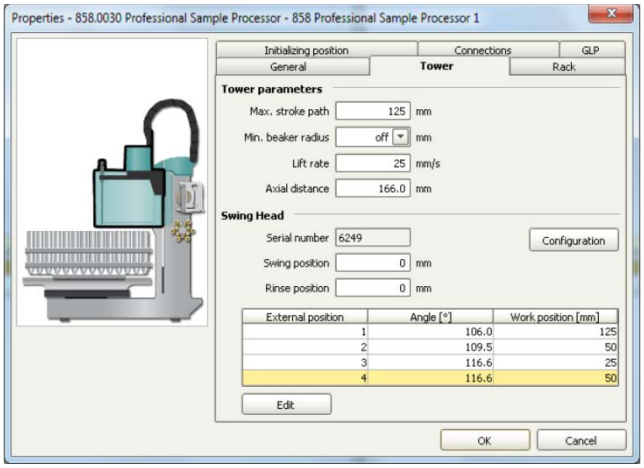
Now the Liquid Handling Station needs to be aligned with the retaining plate. For this, loosen the big screw again and move the Station underneath the retaining plate. Looking from above you should now be able to see the small hole of the rinsing unit and part of the big hole of the dilution vessel,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

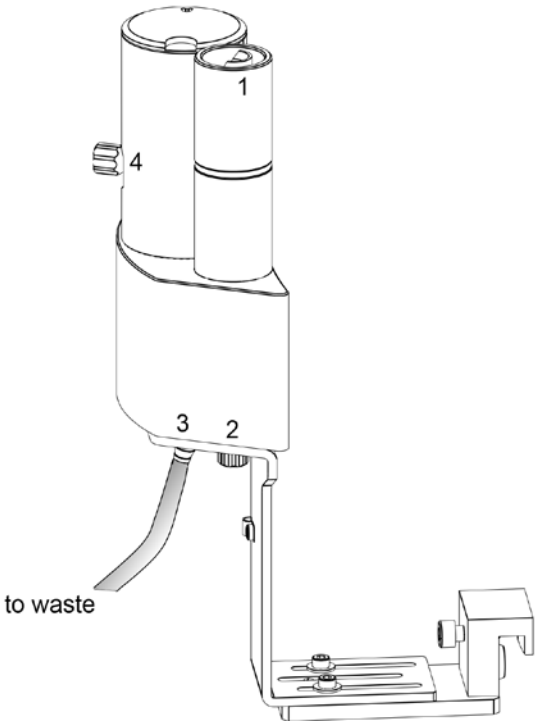


Mount the Dosino on the 5 mL Dosing Unit and connect the FEP aspiration tube M6 (6.1819.100) on Port 2 of the Dosino. Then, fill the 2 L bottle with ultrapure water and fix the Dosino with the Dosing Unit on it.

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.