

Installation Instruction for ProfIC Vario 1 Amperometry

ProfIC Vario 1 Amperometry is a Professional IC system for the fully automatic determination of electroactive compounds. «ProfIC Vario 1» safely masters all routine tasks in ion chromatography. It is simple to use and extremely reliable.


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1. Delivery Package

Delivered with ProfIC Vario 1 Amperometry package:

Nr	Article no.	Article designation
IC		
1	2.940.1100	Professional IC Vario ONE
Detector		
1	2.850.9110	IC Amperometric Detector
Sample processor		
1	2.858.0020	Professional Sample Processor
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	6.2743.040	Sample tubes 2.5 mL
1	6.2743.050	Sample tubes 11 mL
1	6.2743.070	Stopper with perforation
1	6.5337.010	IC equipment Wall-Jet cell: Carb (Au,Pd)
1	6.1090.420	Metrosep Carb 2 - 150/4.0
1	6.1090.500	Metrosep Carb 2 Guard/4.0
1	6.1619.000	Adsorber tube for Dosing Unit
1	6.1608.120	PP eluent bottle, 2 L
1	6.1602.200	Bottle head 2 x M6, 1 x M8 to 6.1608.120
1	6.5330.130	IC Equipment: LQH Station

2. Installation

Following is a detailed description of how to install ProfIC Vario 1 Amperometry.

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 IC Amperometric 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)

When working with the Amperometric Detector, it is recommended to use the PP eluent bottle (6.1608.120) instead of the usual glass bottle for IC analysis. This is done to avoid contamination and detection of electroactive compounds leaking out of the glass.

In the box with the Accessory Kits ONE, you will find all the additional accessories for setting up the eluent bottle. For trace analysis and better chemical resistance, please use the PP bottle (6.1608.120) with its cap (6.1602.200) and the adsorber tube (6.1619.000).

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

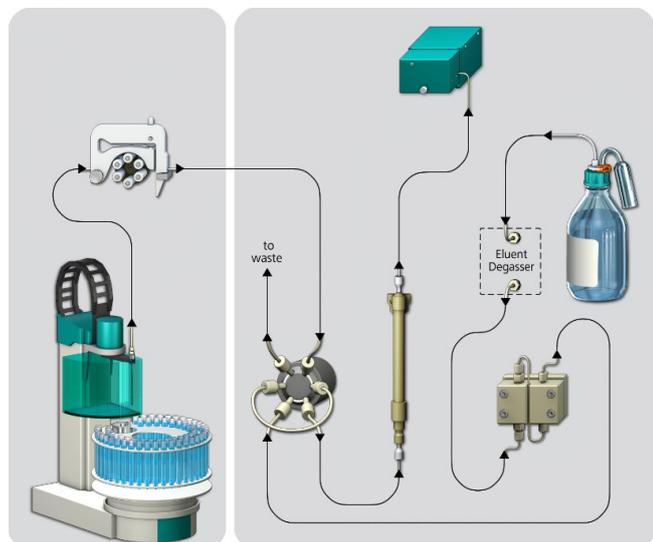
device it is connected to is recognized by the software. For this, you have to plug the USB cable of the IC into the PC and turn it on. Afterwards, it is possible to mount the retaining plate, the needle and the safety shield (for detailed instructions please refer to the 858 Professional Sample Processor manual).

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

Insert the pump tubing (white/white 6.1826.360) into the peristaltic pump and connect the aspiration side to the needle via a PEEK capillary (6.1831.160) (see also 858 manual).

2.5. Interconnection of devices

The whole setup of the ProfIC Vario 1 Amperometry packet is depicted here:

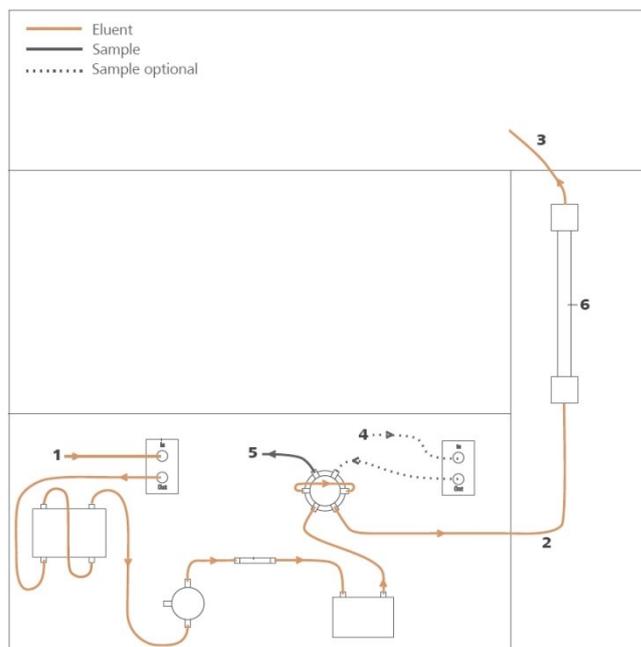


Prepare and degas an eluent suitable for the column you want to use (see column manual). For Metrosep Carb 2-150/4.0 a solution of 100.0 mmol/L sodium hydroxide and 10 mmol/L sodium acetate is adequate.

2.6. 940 Professional IC Vario

Capillaries are connected according to the following list and the diagram below (also see 940 IC manual).

1. Connection to the Eluent bottle
2. Capillary to column inlet – 0.25 mm ID
3. Detector input capillary – 0.25 mm ID
4. Sample aspiration capillary – PTFE, 0.5 mm ID. The sample degasser is only used if necessary
5. Sample outlet capillary – PTFE, 0.5 mm ID, ca. 1 m, connected to waste container
6. 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.

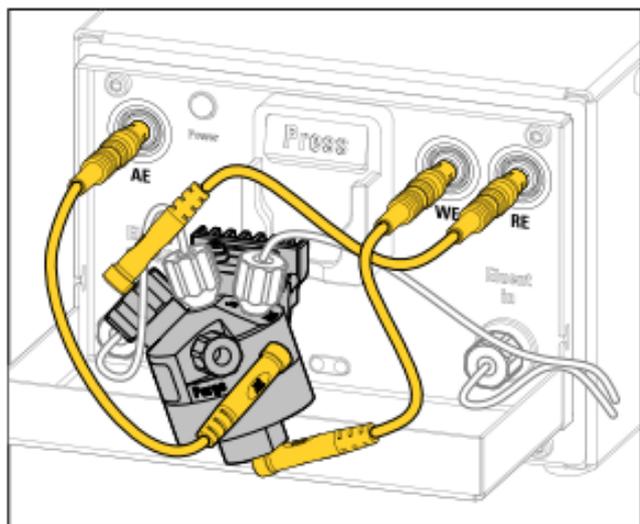


In order to transfer the sample to the IC device, place a PTFE capillary (6.1803.030) of suitable length (~1 m) between the peristaltic pump tubing outlet of the sample processor and the valve of the IC instrument. The sample injection is at position 1 of the valve (see diagram, capillary number 4). 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 (detector outlet, sample waste).

The “detector input capillary” (number 3 in the previous image) connects the column outlet with the “Eluent in” connector of the detector. A short piece of 0.25 mm ID capillary has to be connected between the “Eluent to cell” connector and the “In” connector on the measuring cell. Install the capillary as short as possible to avoid peak broadening. The detector output capillary needs to be at least 1.5 - 2.0 m long (0.25 mm ID) to guarantee a stable pressure in the flow-through cell. Please refer to the IC Amperometric Detector manual for more details. For assembling the measuring cell check the measuring cell manual.

Insert the chip of the measuring cell into the cell holder so that you can hear it locking in. Do not move the cell for at least 5 seconds after having inserted it.

After connecting the cables the cell should look like the following picture. Be careful to connect the correct cables to the right electrode (AE= auxiliary electrode, WE= working electrode, RE= reference electrode).



The purging of the cell will be performed later on in this instruction.

3. MagIC Net

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 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, change the layout and add the subwindow "Amperometric cells" as a fifth displayed subwindow. The 940 Professional IC Vario ONE, the 858 Professional Sample Processor, the Wall-Jet cell and the column Metrosep Carb 2 - 150/4.0 should now be visible. Add and define the eluent.

In the subwindow "Amperometric cells", click on the appearing cell, give it a name and fill in the required information for the electrodes and the monitoring.

Working electrode (WE)

Type WE: Au

Order number WE: 6.1257.240

Serial number WE: 01195797

Set to work WE: 2011-05-25

Comment:

Reference electrode (RE)

Type RE: Pd

Order number RE: 6.1257.740

Serial number RE: 10202324

Set to work RE: 2011-05-25

Comment:

Spacer

Type Spacer: 50 um

Order number Spacer: 6.1257.810

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

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.

Special beaker	Rack position	Work position Tower 1	Work position Tower 2	Beaker radius	Beaker sensor
1	1	125	0	off	off
2	0	0	0	off	off
3	0	0	0	off	off

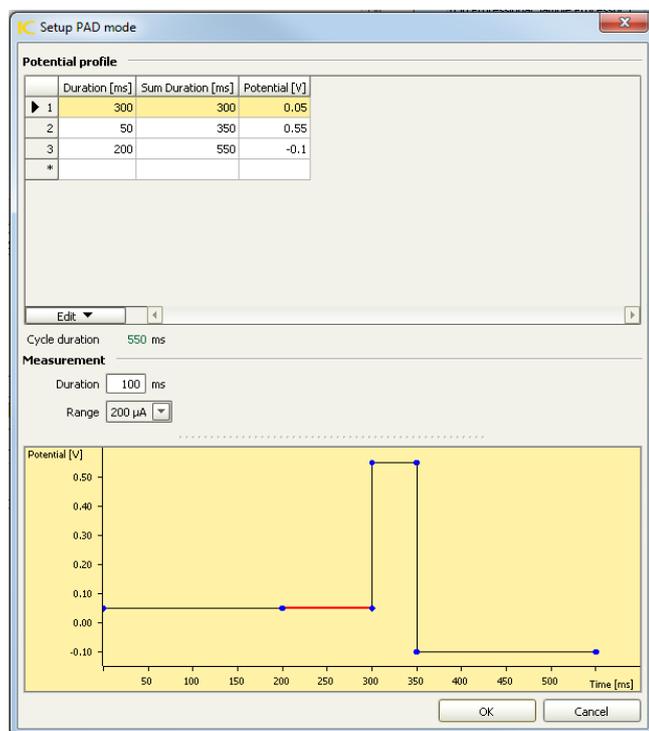
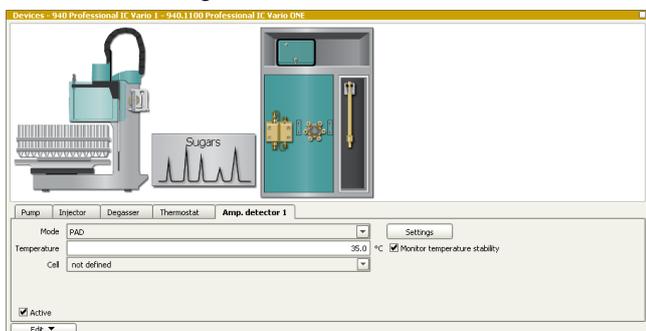
3.2. Method adjustment

In the window method, import the method for ProfIC Vario 1 Amperometry 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_1_Amperometry.imet). In the window method, open the ProfIC Vario 1 Amperometry method and assign the following equipment: Assign the column to the analysis Sugars, define the rack of the connected sample processor, assign the eluent to the IC pump and allocate the cell in the Amperometric Detector of the IC.

Perform a method test and save the method.

In the example method, the cell will be measuring in the PAD mode with the standard Metrohm waveform. If you want to perform a different analysis, you will have to change the mode and/or the waveform in the method. In the tab Amp.Detector 1 of the 940 IC Professional Vario, you can choose the mode with which you would like to measure and under settings you can change the conditions for the different measuring modes.



3.3. Purge of the system

Before turning the cell “on”, be careful to purge the amperometric cell and remove all air bubbles from the system. The measuring cell must be deaerated after the installation and after each subsequent opening of the cell (see the IC Amperometric Detector manual). To do this, open the purge screw and observe the eluent that emerges through the deaeration opening out of the cell. Wipe up the liquid with a cloth. As soon as no more bubbles are visible, screw the stopper back on the Purge connector and tighten the screw by hand.

Put the front lid back in place, this is especially important when analysis in DC mode is done. Be careful not to pinch any capillaries or cables.

Observe the measuring cell: No liquid should emerge from the body of the measuring cell.

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

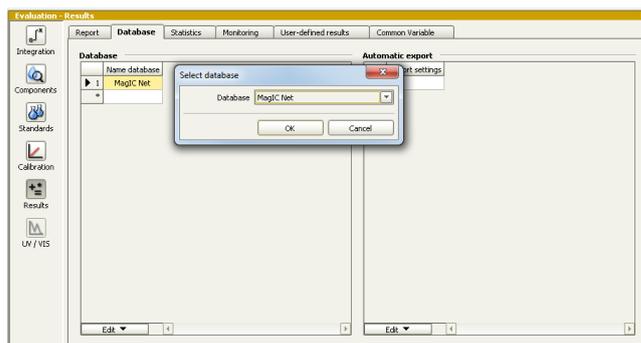
In the program part “Manual control” of MagIC Net, set the flow of the high pressure pump to 0.2 mL/min and start the high pressure pump. Watch the detector output capillary: Liquid must emerge from the end of the detector output capillary. Then raise the flow of the high pressure pump to the appropriate column flow.

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). Rinse the whole analytical system for additional 30 min without putting the cell on to condition and clean the electrodes of the cell.

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

3.4. User defined determination

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

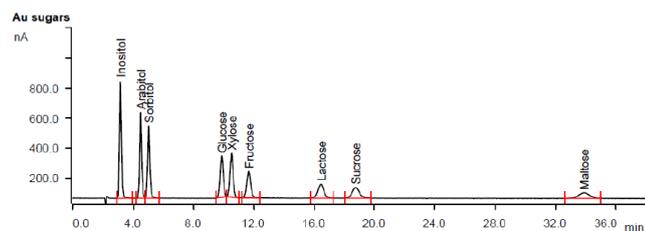


Set up a “determination series”, describing your samples by ident, vial number, sample type (standard, blank, or sample etc.). After putting the analyte solutions onto the rack, press “start”.

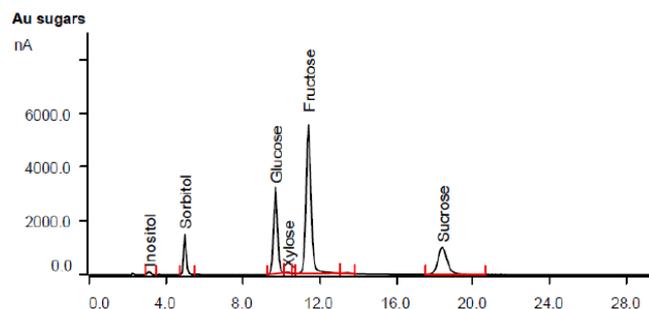
For evaluation and after recording the first chromatogram, check the retention times of your compounds. Since they depend on the performance of your column, you may have to adjust them in your method.

4. Exemplary measurement

Sugar alcohols and sugars were analyzed in ultrapure water and apple juice on the Carb 2 - 150/4.0 and an eluent of 100 mmol/L sodium hydroxide and 10 mmol/L sodium acetate. The first chromatogram shows a 20 µL injection of 1 mg/L of each compound and the second one an injection of apple juice, diluted 1:1000 and passed through an ultrafiltration cell.



Peak number	Component name	Height nA	Area (nA) x min	Concentration mg/L	Retention time min
1	Inositol	779.515	106.937	1.023	3.16
2	Arabitol	574.721	86.554	1.009	4.50
3	Sorbitol	484.952	82.382	0.985	5.03
4	Glucose	278.130	65.895	0.984	9.87
5	Xylose	293.718	71.905	0.983	10.52
6	Fructose	177.233	53.100	1.024	11.67
7	Lactose	94.483	43.294	0.940	16.45
8	Sucrose	71.168	39.915	0.911	18.72
9	Maltose	39.487	33.227	0.961	33.85



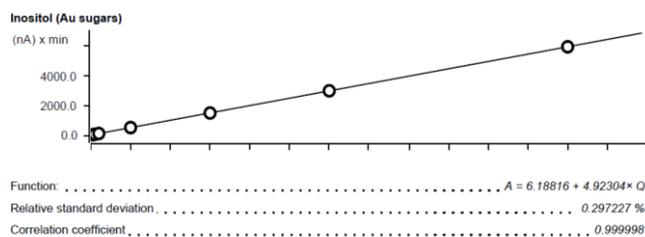
Peak number	Component name	Height nA	Area (nA) x min	Concentration mg/L	Retention time min
1	Inositol	118.124	20.625	146.627	3.13
2	Sorbitol	1514.553	246.786	2971.375	4.99
3	Glucose	3224.624	770.113	11723.981	9.71
4	Xylose	36.227	7.203	72.412	10.34
5	Fructose	5526.066	1684.063	30873.059	11.43
7	Sucrose	1027.524	581.810	14506.154	18.39

Component name	Retention time [min]	Sugar concentration [g/100 mL]
Inositol	3.13	0.01
Sorbitol	4.99	0.30
Glucose	9.71	1.17
Xylose	10.34	0.01
Fructose	11.43	3.09
Sucrose	18.39	1.45

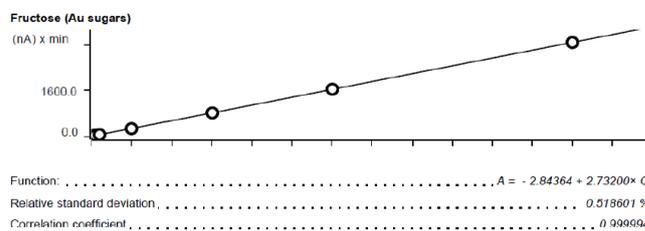
For the determination a multipoint calibration was performed. The calibration is linear over a wide range (see table below). Correlation coefficients and standard deviations are also listed in the following table.

Component name	Conc. [mg/L]	Correlation coefficient	Relative standard deviation [%]
Inositol	0.25 - 60	0.9999	0.30
Arabitol	0.25 - 60	1.0000	0.12
Sorbitol	0.25 - 60	0.9999	0.31
Glucose	0.25 - 60	0.9999	0.25
Xylose	0.25 - 60	0.9999	0.39
Fructose	0.25 - 60	0.9999	0.52
Lactose	0.25 - 60	0.9999	0.97
Sucrose	0.25 - 60	0.9999	1.34
Maltose	0.25 - 60	0.9999	0.92

The calibration curves for inositol and fructose are shown as examples.



Sample type	Index	Conc.	Volume	Dilution	Sample amount	Area	Ident
Standard 1	1	0.250	20.0	1.0	1.0	28.221	0.25 ppm mix std 1
Standard 2	1	0.500	20.0	1.0	1.0	54.699	0.5 ppm mix std 1
Standard 3	1	1.000	20.0	1.0	1.0	106.937	1.0 ppm mix std 1
Standard 4	1	5.000	20.0	1.0	1.0	502.207	5 ppm mix std 1
Standard 5	1	15.000	20.0	1.0	1.0	1476.116	15 ppm mix std 1
Standard 6	1	30.000	20.0	1.0	1.0	2965.748	30 ppm mix std 1
Standard 7	1	60.000	20.0	1.0	1.0	5912.385	60 ppm mix std 1



Sample type	Index	Conc.	Volume	Dilution	Sample amount	Area	Ident
Standard 1	1	0.250	20.0	1.0	1.0	12.731	0.25 ppm mix std 1
Standard 2	1	0.500	20.0	1.0	1.0	26.960	0.5 ppm mix std 1
Standard 3	1	1.000	20.0	1.0	1.0	53.100	1.0 ppm mix std 1
Standard 4	1	5.000	20.0	1.0	1.0	264.189	5 ppm mix std 1
Standard 5	1	15.000	20.0	1.0	1.0	812.844	15 ppm mix std 1
Standard 6	1	30.000	20.0	1.0	1.0	1642.235	30 ppm mix std 1
Standard 7	1	60.000	20.0	1.0	1.0	3274.067	60 ppm mix std 1

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 kit is needed:

Nr	Article no.	Article designation
1	6.5330.130	IC Equipment: LQH Station



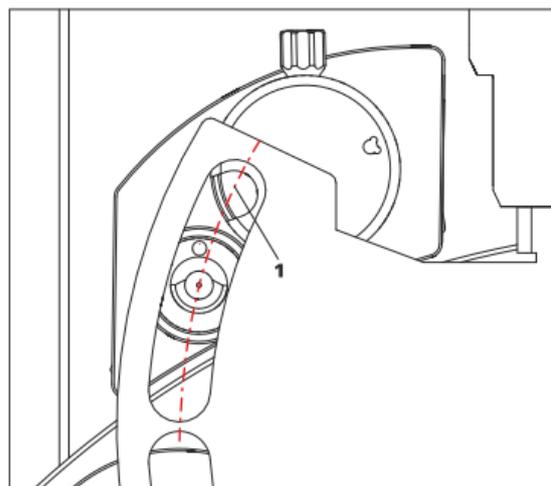
The kit includes:

Nr	Article no.	Article designation
1	6.1014.200	Metrosep I Trap 1 - 100/4.0
1	6.1602.160	Eluent bottle cap GL 45
1	6.1608.070	Eluent bottle /2 L / GL 45
1	6.1619.000	Adsorber tube for Dosing Unit
1	6.1624.000	Adapter SGJ 14
1	6.1826.390	Pump tubing LFL (yellow/yellow) 3 stoppers
1	6.1831.180	PEEK capillary i.D. 0.5 mm, 3 m
1	6.2744.010	Pressure screw 5x
1	6.2744.034	Coupling nozzle UNF 10/32
1	6.2841.120	Liquid Handling Station left

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.

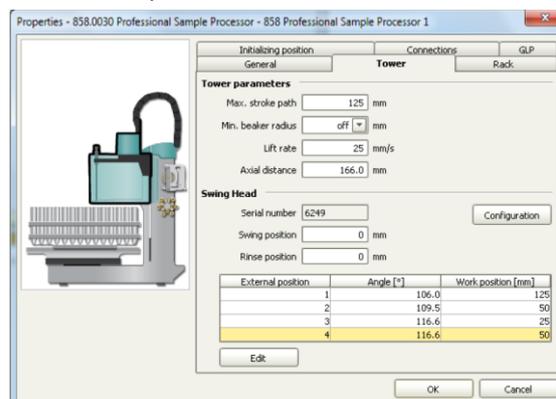
Now the Liquid Handling Station needs to be aligned with the retaining plate. For this, loosen the big screw again and move the Station underneath the retaining plate. Looking from above you should now be able to see the small hole of the rinsing unit and part of the big hole of the dilution vessel,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



Depending on the available parts, the water supply to the Liquid Handling Station is managed differently. 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.

All possibilities have the waste tube in common, Number 3 in the following image. Please fix the PVC tubing (6.1801.120) on the disposal connector on the bottom of the Liquid Handling Station.

If there is a peristaltic pump canal free, there is the possibility to connect a water bottle with a peristaltic pump tubing and lead a capillary 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, please install an I-Trap (6.1014.200) into the flow path to cleanse the water before it goes into the IC system.

Another possibility arises with a Dosino:

Similar to the setup with the peristaltic pump before, connect one of the Dosino ports with 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.

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.

In the time program you can treat the external positions like a special beaker: Just move to the wanted angle, go into work position and start the Dosino or the peristaltic pump. Make sure to always dispose the waste in the external position 2.

