Metrohm

Application Bulletin 800105002EN

Installation Instruction for a Dose-in Gradient System

The Dose-in Gradient can be used together with Compact IC as well as with Professional IC systems for the fully automatic determination of anions or cations with suppression. The Dose-in Gradient setup is highly flexible and up to five different solutions can be combined into one eluent for the IC system. The gradient profile is created by using one high pressure pump working at a constant flow rate and one or more Dosinos which deliver the additional solutions to be mixed into the main eluent flow. The portion of solution delivered by the Dosino(s), which ultimately determines the eluent concentration, can be easily programmed in MagIC Net. The Dose-in Gradient is simple to use, flexible, and very reliable.

Table of contents

1. Recommended equipment

No.	Article no.	Article designation
Instruments		
1	2.930.2560*	930 Compact IC Flex Oven/Ses/PP/Deg
1	2.850.9010	IC Conductivity Detector
1	2.858.0020	858 Professional Sample Processor - Pump
1	2.800.0020**	800 Dosino
Software		
1	6.6059.4***	MagIC Net 4*** Compact (*** newest MagIC Net version)
Accessories		
1	6.5330.150	IC equipment: Dose-in Gradient Anions
or		
1	6.05300.000	IC equipment: Dose-in Gradient Cations
1	6.1580.xx0**	807 Dosing Unit
Optional depending on analysis		
1	6.2041.440	Sample rack 148 × 11 mL + 3 × 300 mL
1	6.2041.760	Sample rack 54 × 11 mL + 1 × 300 mL
1	6.2743.050	Sample tubes 11 mL
1	6.2743.070	Stopper with perforation
1	6.xxxx	Metrosep A Supp column depending on application
1	6.xxxx	resp. Metrosep A Supp Guard column
1	6.xxxx	Metrosep C Supp column depending on application
1	6.xxxx	resp. Metrosep C Supp Guard column
1	6.2832.000	MSM Rotor A
1	6.2842.000	MSM-HC Rotor A
1	6.2842.200	MSM-HC Rotor C
1	6.2842.020	Adapter sleeve, MSM
1	2.941.0010	Eluent Production Module
1	6.5330.090	IC Equipment: Additional eluent for 941
1	6.5330.130	IC Equipment: LQH Station
1	6.5330.190	IC Equipment: Dosino Regeneration

** Can be any 930 Compact IC Flex or 930 Compact IC Flex system with suppression module*

*** Quantity depends on the number of solutions to be added—the right choice of the 807 Dosing Unit volume is best to be defined in accordance with the information of your Metrohm representative, which is the starting point of the application.*

2. IC Equipment: Dose-in Gradient Anions/Cations

IC equipment: Dose-in Gradient Anions (6.5330.150) IC equipment: Dose-in Gradient Cations (6.5300.0000)		
1 or 1	6.1014.000 6.1015.000	Metrosep A Trap 1 - 100/4.0 Metrosep C Trap 1 - 100/4.0
1	6.1602.160	Eluent bottle cap GL 45
1	6.16008.070	Eluent bottle / 2 L / GL 45
1	6.1618.020	Thread adapter S 40 to GL 45
1	6.1624.000	Adapter SGJ 14 for 6.1619.xxx Adsorber tube
1	6.1805.120	FEP tubing / M6 / 100 cm
1	6.1829.020	FEP aspiration tube M6, 0.5 m
1	6.1831.030	PEEK capillary 0.75 mm ID, 3 m
1	6.1831.120	PEEK capillary 0.25 mm ID, 45 cm
1	6.1834.090	Connecting tubing for degasser pump, 125 cm
1	6.2053.030	Mounting ribbon to 6.2744.340, 25 × 60 mm
1	6.2057.210	Holder for Dosino to IC instruments
1	6.2744.070	Pressure screw short
1	6.2744.080	M6 thread / UNF 10/32 adapter
1	6.2744.340	6-port adapter 4 × UNF 10/32, 2× UNF 1/4-28

1	6.2744.350	PEEK pressure screw 14 mm, 3 pcs.

The 807 Dosing Unit and Dosino for the addition of the second solution into the main eluent are not included in the kit. This is because the volume of the 807 Dosing Unit depends on the application (the smaller the volume the more accurate the dosing, but the volume must also be large enough so no refill is required during the chromatogram) and as the Dosino is an instrument, it can never be included in kits.

For the junction of more than two solutions, more 807 Dosing Units, Dosinos, and accessories are required. For each additional solution, the following parts must be ordered:

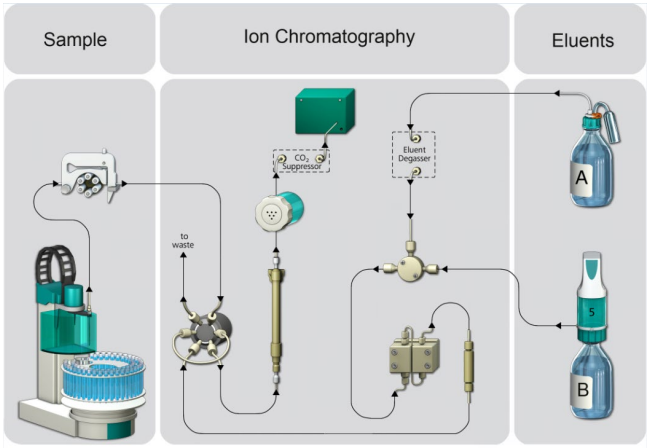
1	2.800.0020	800 Dosino
1	6.1580.xx0	807 Dosing Unit
1	6.1602.160	Eluent bottle cap GL 45
1	6.16008.070	Eluent bottle / 2 L / GL 45
1	6.1618.020	Thread adapter S 40 to GL 45
1	6.1624.000	Adapter SGJ 14 for 6.1619.xxx Adsorber tube
1	6.1805.120	FEP tubing / M6 / 100 cm
1	6.1829.020	FEP aspiration tube M6, 0.5 m
1	6.1831.030	PEEK capillary 0.75 mm ID, 3 m
1	6.2744.070	Pressure screw short
1	6.2744.080	M6 thread / UNF 10/32 adapter
1	6.2057.210*	Holder for Dosino to IC instruments

** One holder fits two Dosinos. If more than two Dosinos are installed with the setup, at least one additional holder needs to be ordered.*

3. Installation

The following is a detailed description of how to install a Dose-in Gradient system that mixes two solutions into one eluent. If a gradient higher than binary is needed, any additional Dosinos are simply installed like the initial one and added to the 6-port adapter used for junction purposes.

Ultimately, the whole setup will look like this:



Here, a T-connector is drawn, but in reality a 6-port adapter is installed in this place so more than two solutions can be combined.

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

3.1. Installation of the software

All programs must be shut down first. Make sure that no Metrohm instrument is connected to the PC. Install MagIC Net**.** All standard directories proposed by the program should be accepted.

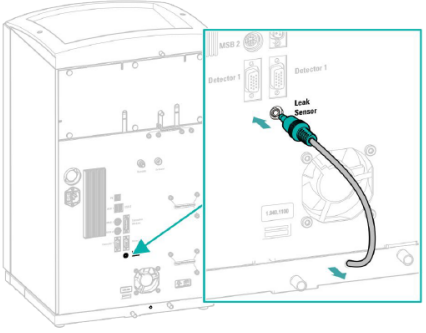
From now on, every new Metrohm instrument connected to the PC will automatically be recognized and its driver will be installed. A window will pop up in MagIC Net, asking you to store this device in your configuration. The names will be checked later in this document, but it is recommended to use the proposed instrument names.

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

Using the Accessory Kit Vario/Flex Basic, which is delivered with the IC instrument, install the 930 Compact IC Flex. Place the detector block in the instrument and connect the detector cable in the back of the instrument. Remove the transport locking screws, connect the leak sensor cable, and connect the drainage tubing.

Plugging in the leak sensor connection cable

The leak sensor connection cable is coiled up in the base tray.



Remove the transport locking screws

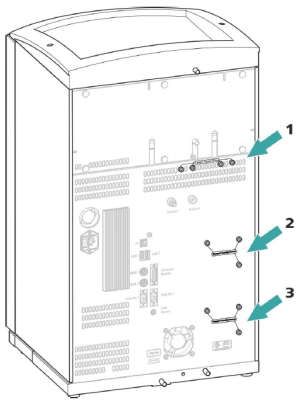
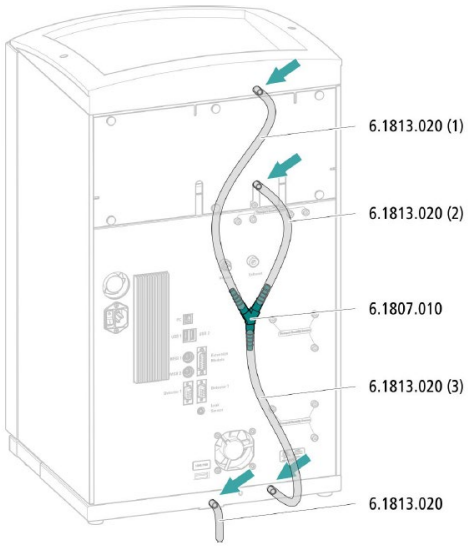


Figure 6 Removing the transport locking screws

1 **Transport locking screws** For the vacuum pump.	**2** **Transport locking screws** For the high-pressure pump.
3 **Transport locking screws** For an additional high-pressure pump in the bottom drawer.	

Connecting the drainage tubing



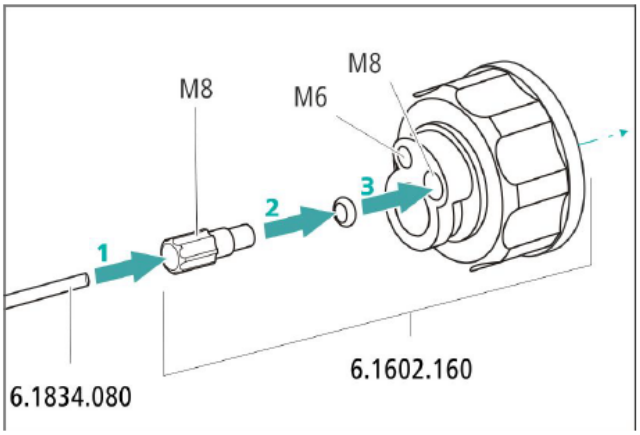
Mount the holder for Dosinos (6.2057.210) on the IC by removing the bottle holder on top of the instrument, placing the Dosino holder on the side of the IC, and remounting the bottle holder on top.

Next, set up the waste collector by assembling the cap and screwing it onto the vessel. Hang the waste collector with its holder on one of the sides of the IC. Make sure there is an unobstructed view to the collector so you can later observe the droplets coming out of the capillaries connected to the collector. Attach the waste tube to the vessel and lead it to the waste canister. If the tube is too long, please shorten it because it is important to have a high-level difference for the liquid to drain properly.

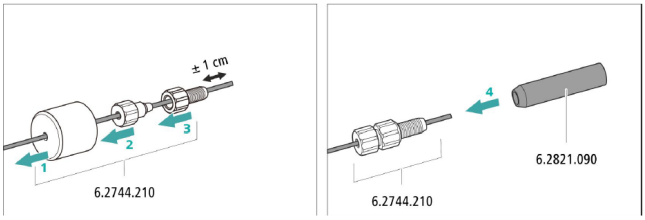
The power cable and USB cable for connection of the IC to the PC (6.2151.020) are plugged into the rear of the Compact IC Flex. Do not turn on the instrument yet. This step will follow after the completed installation.

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

All the accessories for setting up the Eluent bottle are found in the box with the Accessory Kit ONE. To begin, lead the aspiration tube for the Eluent through the M8 stopper, the O-ring, and the eluent cap as shown below.



Then fix the white weight (6.2744.210), the adapter (6.2744.210), and the aspiration filter (6.2821.090) on the eluent aspiration tube, all the while being careful not to touch the filters 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 930 Compact IC Flex manual for a detailed description.

3.4. 807 Dosing Unit for Gradient

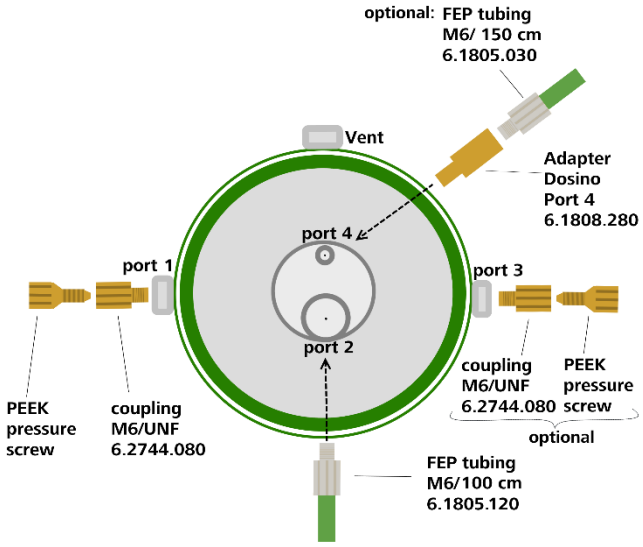
Fix the 807 Dosing Unit with the Dosino on top of the Dosino Holder of the IC. For this, place the 807 Dosing Unit over the free hole of the Dosino holder and fix it from the bottom with the thread adapter (6.1618.020). Then, place the Dosino on top and lock the 807 Dosing Unit to its motor. The Dosino must be plugged into the MSB1 connection of the 930 Compact IC Flex.

Port 1 of the 807 Dosing Unit will be connected to the 6-port adapter with the help of a PEEK capillary, 0.75 mm ID, two PEEK pressure screws, and a coupling M6/UNF for the connection at the 807 Dosing Unit port.

Port 2 of the 807 Dosing Unit is directly linked to the 2 L bottle, containing Eluent B with the FEP tubing 6.1805.120.

Port 3 of the 807 Dosing Unit can be connected to the waste collector (optional).

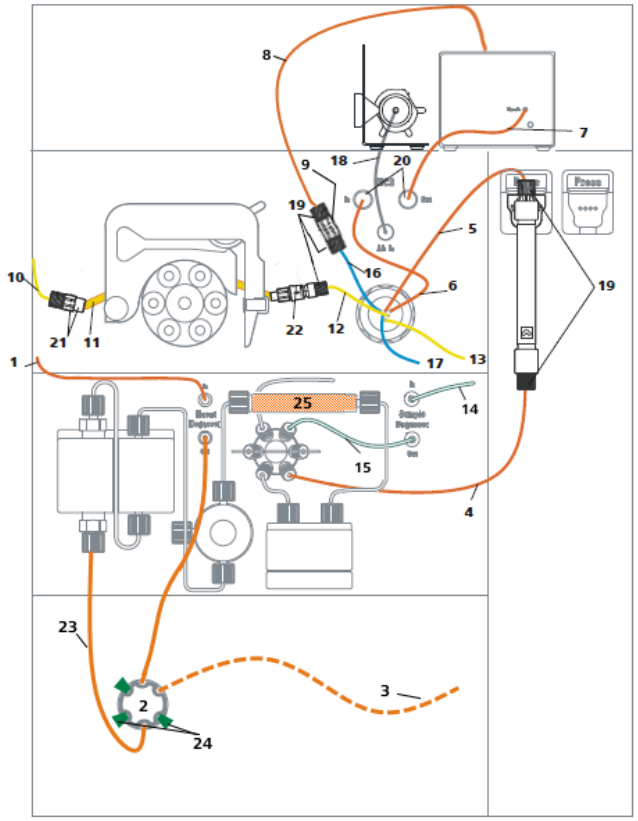
Finally, port 4 of the 807 Dosing Unit can be connected to a bottle filled with UPW or 20% MeOH (methanol) for additional rinsing (optional).



3.5. 930 Compact IC Flex

Capillaries are connected according to the following list and diagram. In this case, the suppressor is regenerated via peristaltic pumps. If the Dosino Regeneration is applied, please refer to Application Bulletin 800105018EN.

1. Connection from the eluent bottle A
2. 6-port adapter (6.2744.340)
3. Connecting capillary to 6-Port-Distributor (use part of 6.1831.030) with 807 Dosing Unit Port 1 (use the adapter for M6 thread / UNF (6.2744.080))
4. Connecting Capillary from Injection Valve to Separation Column
5. Connecting capillary from Separation Column to Suppressor
6. MSM outlet capillary – labeled with *Out,* which has to be connected to the MCS with a long PEEK pressure screw (6.2477.090)
7. Capillary from MCS to anion detector, a long PEEK pressure screw (6.2744.090) is used for the MCS connection
8. Detector outlet capillary connected to the MSM inlet capillary (16) – labeled with *rinsing solution*
9. Coupling (6.2744.040) Capillary from Regenerant Solution
10. Regeneration solution aspiration capillary – PTFE, 0.5 mm ID, connected to the bottle with 100 mmol/L sulfuric acid
11. Peristaltic pump tubing with orange/yellow stoppers (6.1803.030)
12. MSM regeneration inlet capillary – labeled with ‘regenerant’
13. MSM regeneration solution outlet capillary – labeled with ‘waste reg.’ to be connected to the waste collector
14. Connecting capillary (6.1831.160) from Sample Processor peristaltic pump to Sample Degasser (installation of Sample Degasser is optional and not recommended!)
15. Connecting Capillary from Sample Degasser to Injection Valve (installation of Sample Degasser is optional and not recommended!)
16. MSM rinsing solution inlet capillary – labeled with ‘rinsing solution’
17. MSM rinsing solution outlet capillary – labeled with ‘waste rins.’ to be connected to the waste collector
18. Connecting Capillary from Cartridge to MCS
19. High Pressure PEEK Screws
20. MCS and Sample Degasser In-/Outlet
21. Tubing Olives
22. Tubing Olives with inline filter
23. Connecting eluent tubing (6.1834.090) from 6-port adapter (6.2744.340) to high pressure pump
24. Stopper (green, PEEK)
25. Trap column (for mixing purposes)



Make sure that all outlet capillaries are put into the waste collector, then prepare the appropriate eluents for your column and gradient choice. Pour them into the respective 2 L eluent bottles. The eluent bottle cap (6.1602.160) for eluent B is equipped with the adsorber tube (6.1619.000), delivered with the 807 Dosing Unit, using the adapter for adsorber tube (6.1624.000). Furthermore, the FEP aspiration tubing to the canister (6.1819.110) is passed through the M6 hole in the cap and installed such as to reach the bottom of the eluent B bottle. The O-ring, which is not used in this hole, can be inserted into the M8 hole, and the Thread adapter M8 outer / M6 inner (6.1808.090) is screwed on the M8 hole for further use (M8 = larger hole, M6 = smaller hole).

Likewise, prepare the regeneration solution for the MSM and pour it into the 1 L bottle to be aspirated via the peristaltic pump tubing to the suppressor.

After the completed installation, the 6-port adapter can be fixed on the high-pressure pump with the mounting ribbon (6.2053.030) which must be cut to the optimal dimensions to not block access to the screws of the high-pressure pump.

3.6. 858 Professional Sample Processor

It is recommended to work with a Sample Processor when operating an instrument with a Gradient. It is of uttermost importance that the injection of the sample always happens at exactly the same moment of the gradient profile. This can only be guaranteed when using the appropriate sample transfer.

3.6.1. Cable connections

In the back of the 858 Professional Sample Processor, please plug in the controller cable (6.2151.000, Cable USB A – mini-DIN 8-pin) into the corresponding Contr. plug. This cable is then plugged into a USB port on the back of the IC.

Then, plug in the Swing Head connection cable on the tower, connect the power cable, and turn on the IC.

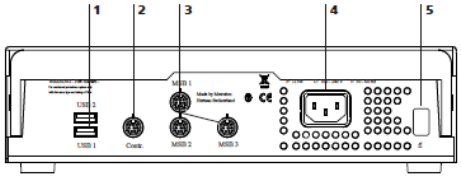


Figure 4 Connector strip

1 **USB connectors**	**2** **Controller connector** For the connection to the PC or Touch Control.
3 **MSB connector** For stirrers, dosing devices, Remote Box.	**4** **Mains connection**
5 **Type plate** Contains specifications concerning mains voltage and serial number.	

3.6.2. Hardware installations

For a detailed installation description, please refer to the 858 Professional Sample Processor manual. In general, as soon as the IC is powered on and recognized by the software, the sample processor initializes and lifts its Swing Head.

Afterwards, the sample needle (6.2846.010) is installed together with the needle holder (6.2833.030) on the swing head of the 858 Professional Sample Processor.

Finally, mount the retaining plate and the safety shield.

3.6.3. Liquid Handling

Insert the pump tubing (white/white 6.1826.360) into the peristaltic pump of the 858 and connect the aspiration side to the needle via a PEEK capillary (6.1831.160) (read the 858 Professional Sample Processor manual for more information).

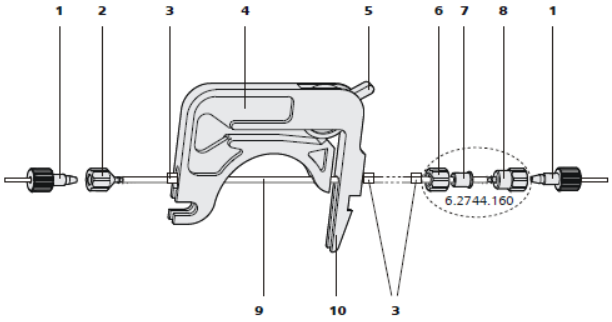


Figure 31 Installing the pump tubing

1	PEEK pressure screws, short (6.2744.070)	2	Tubing olive (6.2744.030)
3	Stopper The colors of the stopper indicate the inner diameter of the pump tubing.	4	Tubing cartridge (6.2755.000)
5	Contact pressure lever	6	Union nut
7	Adapter	8	Tubing olive
9	Pump tubing	10	Snap-action lever

4. MagIC Net configuration

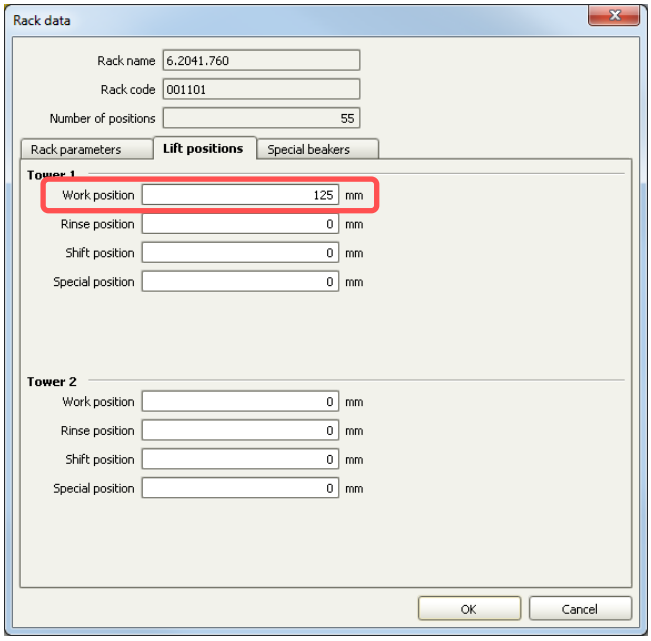
The IC is connected to the computer via its controller cable and the power is turned on. The drivers of all the instruments are automatically installed.

Connected USB devices are automatically recognized when MagIC Net is started. After confirmation of the pop-up windows, the devices and columns are stored in the configuration. The devices in most methods are predefined as “930 Compact IC Flex 1” and “858 Professional Sample Processor 1”. If other names appear in your configuration (e.g., due to changed settings on your computer), rename them accordingly. The column and 807 Dosing Unit solution can be named freely.

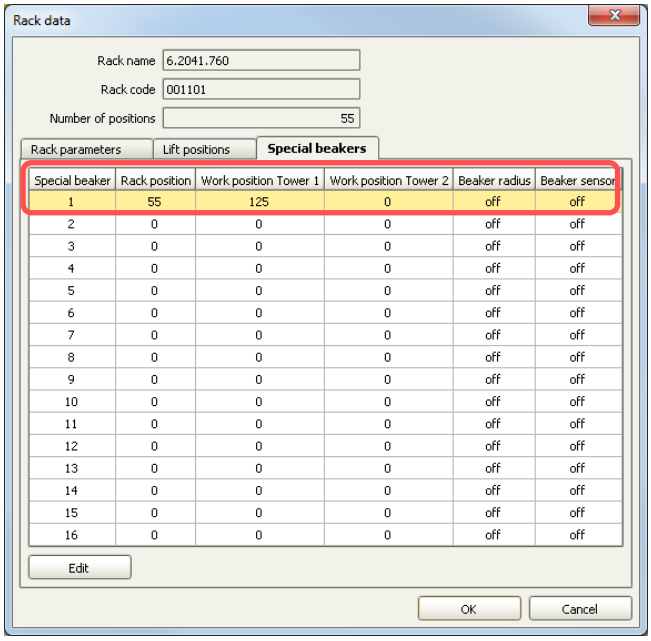
Add and define the eluent and the suppressor solution in the configuration window and register the rotor with its serial number.

4.1. 858 Professional Sample Processor

The work position needs to be defined for the rack. For this purpose, open the rack data of the sample processor and adapt the rack parameters depending on the rack (e.g., 125 mm for rack 6.2041.760).

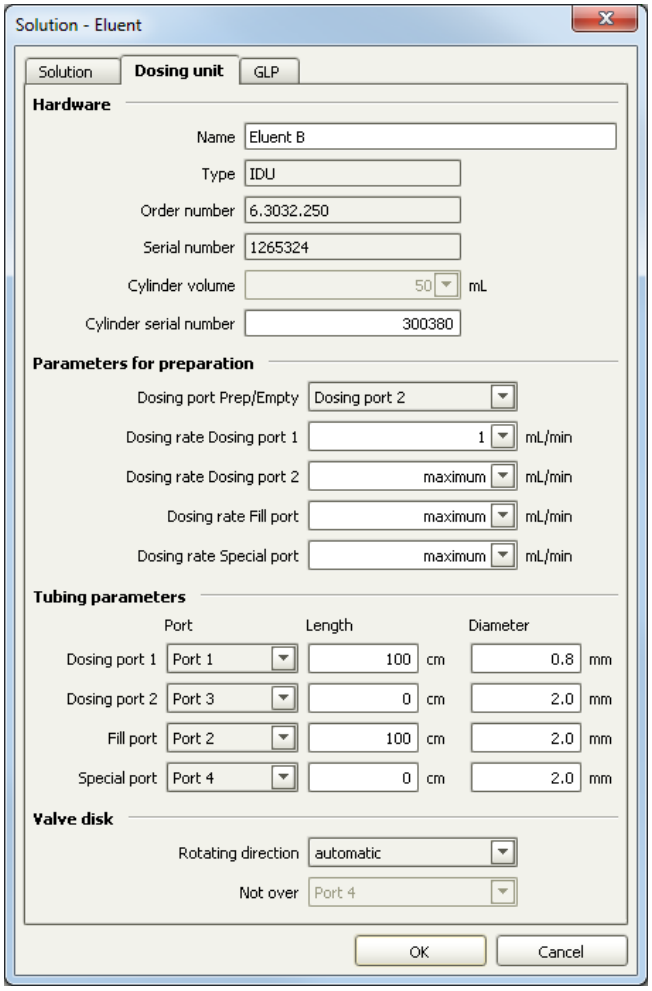


Also, define one or more special beakers for rinsing the needle. Fill the appropriate beaker(s) with ultrapure water and place it/them at the defined position(s) on the rack.



4.2. Dosino solution

The solution for the 807 Dosing Unit needs to be adapted to the following values:

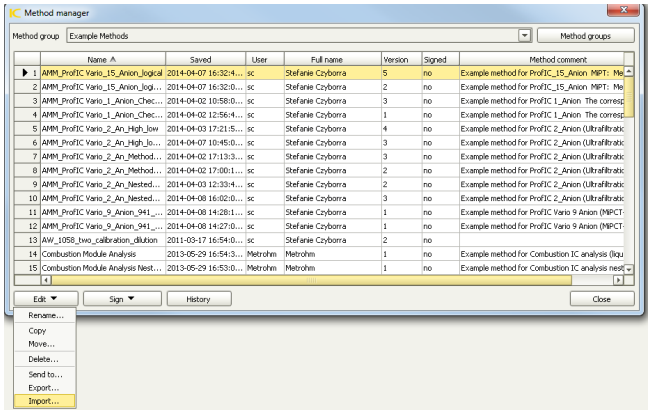


If the optional tubing has been connected to Port 3 and 4, please enter the correct values in the length and diameter fields.

After the values have been adapted, prepare the 807 Dosing Unit in order to have all capillaries and tubing filled with eluent.

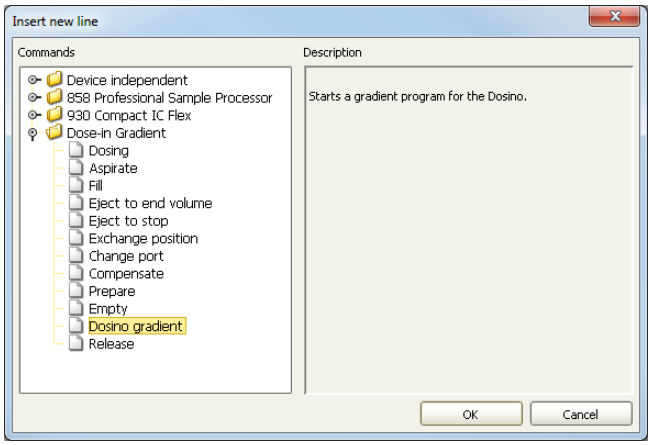
4.3. Import of a Gradient method

Import the method (ask your Metrohm representative for help) into MagIC Net. As the gradient profile depends on which analytes need to be separated, look for the analytes in the literature database and add the search term “gradient”. Transferring a gradient profile from one technique (e.g., Low-Pressure Gradient) to a Dose-in Gradient profile is not as simple as one may think. It is, therefore, recommended to use a Dose-in Gradient profile as a starting point.



Adapt the method to your system by adjusting the instrument type(s) and name(s). Then link the eluent A to the “eluent” field for the high-pressure pump. This eluent will be the eluent that runs during the equilibration phase; therefore, it is recommended to choose the lower concentrated eluent for this function.

The gradient profile itself has to be added in the time program – recommended to start concurrently with the data acquisition. If the profile is started before the liquid handling part is finished, this can lead to retention time shifts, depending on the sample position on the rack.

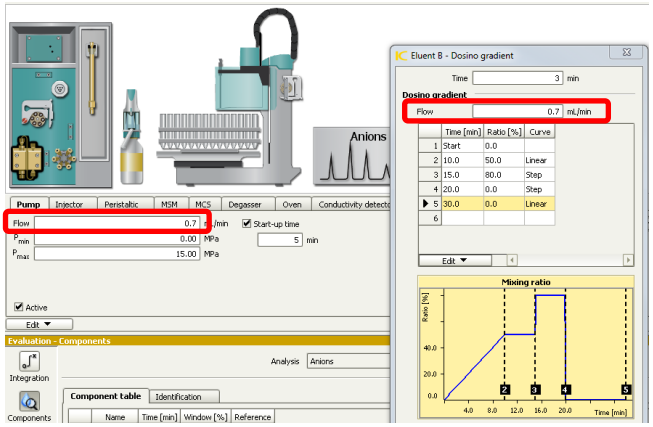


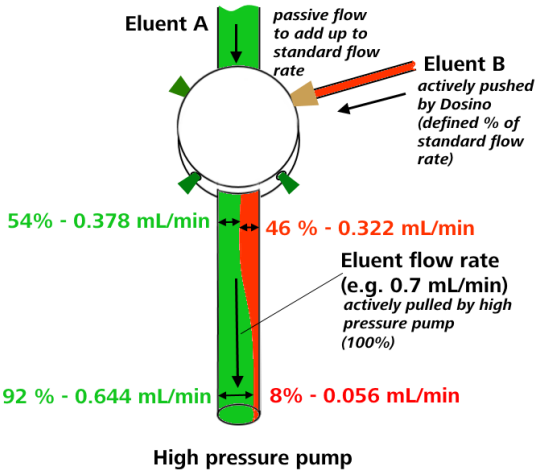
The start parameters of the gradient profile should be the same as the ones defined in the “devices” section of the method. Two different curve types exist: either linear which will change the eluent gradually, or step gradients for abrupt changes in the eluent composition.

It is also recommended to return to using the initial eluent mix for 7–10 minutes before starting the next determination. This helps to avoid retention time shifts due to the full equilibration back to the standard eluent

If more than one eluent is added via a Dosino, multiple Dose-in Gradient profiles need to be created and are best started simultaneously, one for each Dosino/807 Dosing Unit.

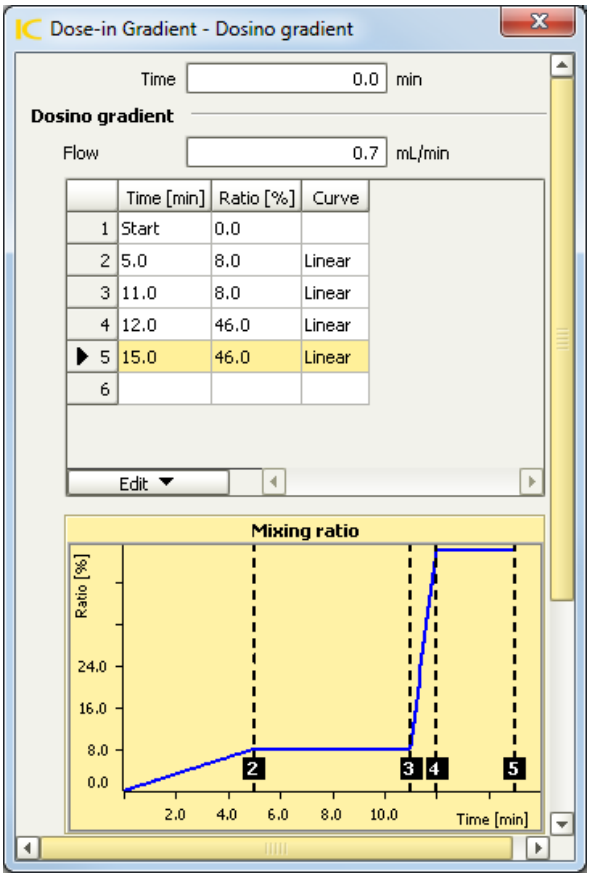
An example profile can be seen in the following picture:





The eluent composition (eluent A+B) is defined by the total flow rate (here, 0.7 mL/min) and the dosing percentage of the Dosino (eluent B). The rest of the 100% comes from the use of eluent A.

For example, when following a gradient profile, the eluent mixture will be composed as shown in the graphic here.



It is possible to run 100% of eluent A, which (by default) happens during the equilibration phase. Nevertheless, it is not recommended to run more than 95% of eluent B, as the Dosino doses at an accurate 0.7 mL/min and if the high-pressure pump is not aspirating at exactly 0.7 mL/min, then a portion of eluent B may enter the eluent A bottle.

For ternary to quintary gradients, the total of the different dosing percentages of the Dosinos must add up to a maximum of 95% of the total flow. The “missing” 5% will always be filled in from eluent A.

4.4. Purge of the system

Before inserting the column, 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.

Flush the system for about 10 minutes and eliminate any air bubbles (by using the purge valve and syringe). During these 10 minutes, step the MSM three times 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 guard column for 10 minutes by leading the outlet directly into the waste. Afterwards, connect the analytical column and flush it likewise for 10 minutes. Only then is the column completely connected to the flow path of the eluent (also see the 930 manual). Start the peristaltic pump and adjust the pressure of the lever on the tubing to see the droplets of suppressor regeneration solution drop into the waste collector.

To start the equilibration, go to the window workplace, load the method, and press “Start HW”.

Equilibrate the system until the baseline is stable.

4.5. Starting a determination series

Please put the analyte solutions on the rack.

In the window workplace in MagIC Net, set up a “determination series” describing the samples by ident, sample position and sample type (standard, blank, or sample, etc.).

When using a gradient with IC, it is recommended to always run a Blank/UPW sample as the first line of a sample series. This run is additionally needed for equilibration purposes.

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

5. Optional equipment

5.1 Liquid Handling Station

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

The following kit is needed:

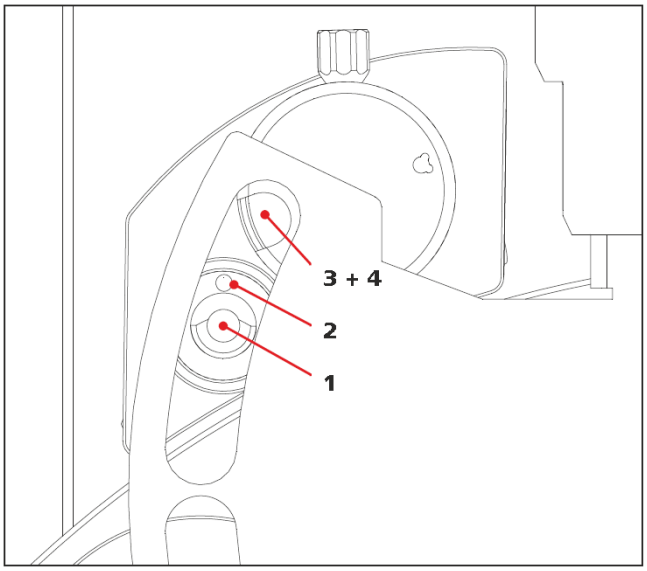
Nr	Article no.	Article designation
1	6.5330.130	IC Equipment: LQH Station

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 807 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 ID 0.5 mm, 3 m
1	6.2744.010	Pressure screw 5×
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 the LQH Station and the rack is 0.5–1 mm.

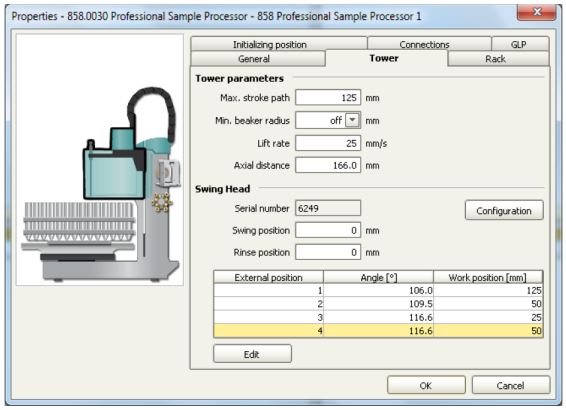
Now, the Liquid Handling Station must be aligned with the retaining plate. For this, loosen the big screw again and move the LQH Station underneath the retaining plate. Looking from above, you should now be able to see the small hole of the rinsing unit and part of the larger hole of the dilution vessel,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

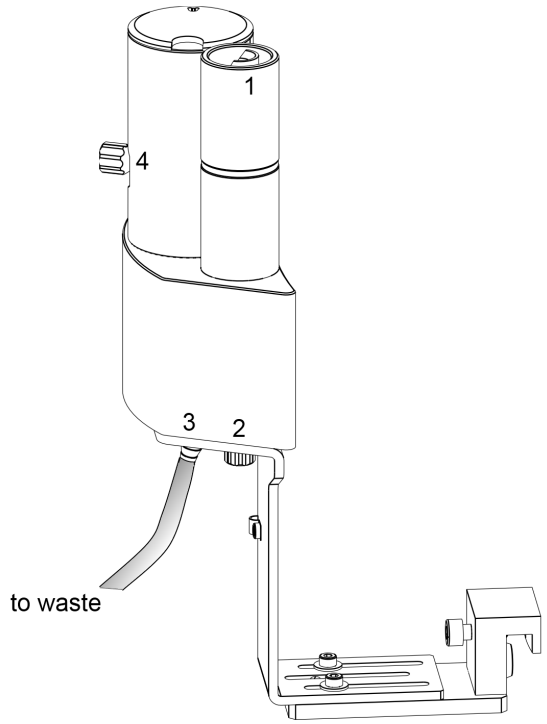


Depending on the available parts, the water supply to the Liquid Handling Station is managed differently. The inner tube of the rinsing unit is meant to be constantly filled with fresh ultrapure water, so the aspiration needle can be thoroughly cleaned from the outside.

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 free peristaltic pump channel, 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 in the following image). 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 described 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 free from any impurities that could have accumulated in the water reservoir.



Number 4 in the picture above shows the attachment point of a capillary in case a dilution feature is integrated, 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 desired angle, go to the work position, and start the Dosino or the peristaltic pump. 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

For alternative suppressor rinsing and regeneration methods and setups, please refer to Application Bulletin 800105018EN.