Metrohm

Application Bulletin 800105020EN

Installation instructions for VoltIC Professional 1

The VoltIC Professional 1 system is used for the fully automated analysis of water samples using voltammetry and ion chromatography. Anions, cations, and heavy metals are determined over a wide range of concentrations within a short analysis time.

Further Metrohm instruments can be incorporated in the existing system at any time and used to determine additional parameters.

Analytical sequence

- The sample is transferred from the 858 Professional Sample Processor to the 940 Professional IC Vario TWO/SeS/PP for the anion and cation analysis.
- Consequently, the sample is transferred into the measuring vessel of the 884 Professional VA, where the voltammetric determination of heavy metals is carried out.

The whole procedure is controlled by the MagIC Net software: The user enters the sample position and sample identification only into MagIC Net. All sample liquid handling is done by MagIC Net. The communication such as starting the voltammetric analysis or transfer of the results from **viva** to MagIC Net is done via RS-232.

All data is summarized in a joint report containing all results by MagIC Net.

Table of contents

1. Scope of delivery

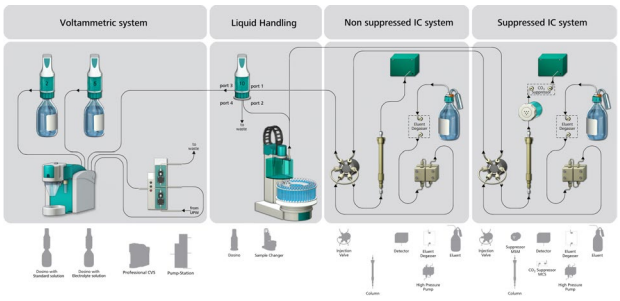
Delivered with VoltIC Professional 1 package:

Nr	Article no.	Article designation
Instruments		
1	2.940.2500	940 Professional IC Vario TWO/SeS/PP
2	2.850.9010	IC Conductivity Detector
1	2.884.1110	884 Professional VA semiautomated for MME
1	2.843.0240	843 Membrane Pump Station for Professional VA/CVS systems
1	2.858.0010	858 Professional Sample Processor
Electrodes and accessories		
1	6.5339.030	Electrode equipment MME for Professional VA
Liquid Handling		
1	6.2041.450	Sample rack 56 x 11 mL + 56 x 50 mL
1	6.2743.050	Sample tubes 11 mL (200 pieces)
1	6.2743.070	Stopper with perforation
4	6.2747.010	Sample vessel 50 mL (25 pieces)
1	6.1831.020	PEEK capillary (0.75 mm ID, 22 cm long) for sample addition VA
1	6.1831.080	PEEK capillary (0.75 mm ID, 110 cm long)
1	2.800.0010	800 Dosino
1	6.1580.210	807 Dosing Unit, glass 10 mL
1	6.2057.210	Holder Dosino for IC Instruments
1	6.1808.280	Adapter Dosino Port 4 / M6
1	6.1618.020	Thread adapter S 40 to GL45
3	6.2744.080	Coupling M6-UNF 10/32 (807 Dosing Unit – IC screw)
1	6.2744.010	Pressure screw made of PEEK, 5x
1	6.2833.020	Needle holder 1/8 in. With tubing connection M6
1	6.1808.010	Coupling piece 2 x M6 outer threads
1	6.1808.060	T Connector M6
1	6.1805.530	FEP tubing/ M6 / 200 cm
1	6.1805.020	FEP tubing/ M6 / 52 cm
Software		
1	6.6059.4x2	MagIC Net 4.x Professional
1	6.6065.202	**viva** 2.0 Full
Cables		
1	6.2134.040	Connecting cable RS-232 DB9 to IBM-PC (DB9)
2	6.2148.050	USB/RS-232 Converter for 900 Touch Control
1	6.2141.300	Remote cable 843 Pump Station to Sample Processor
Optional depending on analysis		
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 2 L
1	6.5330.130	IC Equipment: LQH Station
1	6.5339.500	Equipment with 2 807 Dosing Units for VA and CVS measurements
2	2.800.0010	Dosino (for 6.5339.500)

2. Installation

The following is a detailed description of how to install the VoltIC Professional 1.

The complete setup will ultimately look like this:



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

2.1.1. Software

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

As soon as both programs used in the setup are installed, the RS-232 cable with the USB/RS-232 converters can be connected to two USB plugs on the computer. It is recommended to always use two USB ports on the computer and not those included on the Metrohm instruments, as these ports are faster and more reliable. Wait until the drivers for the converters have been installed.

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 and/or **viva**, asking if you would like to store this device in your configuration. The names will be checked later in this installation instruction, but it is recommended to use the proposed instrument names for method conformity.

2.2. Accessory kits: Vario/Flex Basic (6.5000.000)

Using the two accessory kits Vario/Flex Basic, install the 940 Professional IC Vario. Place the detector blocks in the instrument and connect the detector cables to the back of the instrument. Remove the transport locking screws, connect the leak sensor cable, and connect the drainage tubing.

Remove the transport locking screws

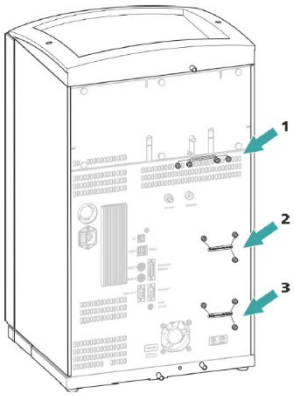
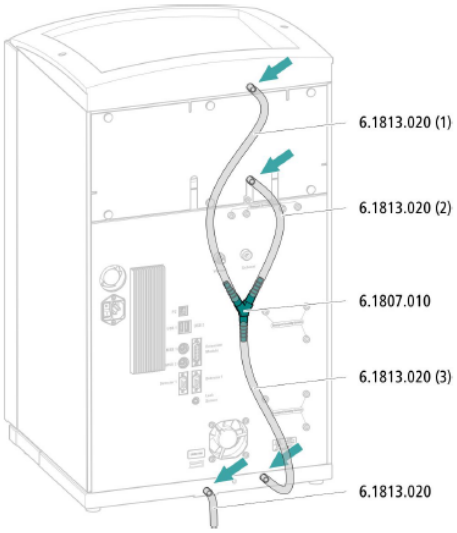


Figure 6 Removing the transport locking screws

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



1 Cut a piece of silicone tubing into three pieces using scissors: 2 × approx. 40 cm and 1 × 20 cm

2 Attach one end of the 40 cm long piece to the drainage tubing connection on the bottle holder.

3 Attach one end of the 20 cm long piece to the drainage tubing connection on the detector chamber.

4 Attach each of the loose ends of both pieces of silicone tubing to one end of the Y connector.

5 Attach one end of the second 40 cm long piece to the third end of the Y connector.

Attach the loose end to the right-side drainage tubing connection on the base tray.

6 Attach one end of the second piece of silicone tubing to the left-side drainage tubing connection on the base tray.

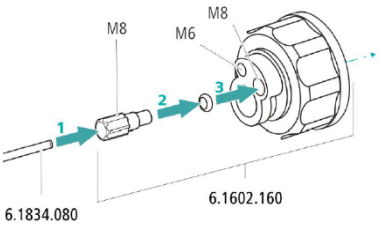
Next, mount the holder for Dosinos (6.2057.210) on the IC by removing the bottle holder on top of the instrument, placing the Dosino holder on the side of the IC, and remounting the bottle holder on top.

Now set up the waste collector by assembling the cap and screwing it onto the vessel. Then hang the waste collector on one of the Dosino holder holes. Make sure you have an unobstructed view of the collector so you can later observe the droplets coming out of the capillaries connected to it. Attach the waste tube to the vessel and lead it to the waste canister. If the tube is too long it must be shortened, because it is important to have a steep path from the vessel to the waste canister in order to reliably drain the waste collector.

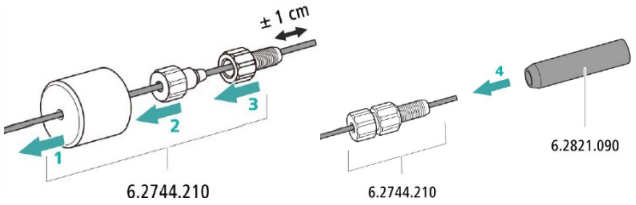
The power cable and USB cable for connection of the IC to the PC (6.2151.020) are plugged into the rear of the Professional IC Vario. Do not switch on the instrument yet. This step will follow after the completed installation.

2.3. Accessory kits: 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 bottles. Lead the aspiration tube for the eluent through the M8 stopper, the O-ring, and the eluent cap.



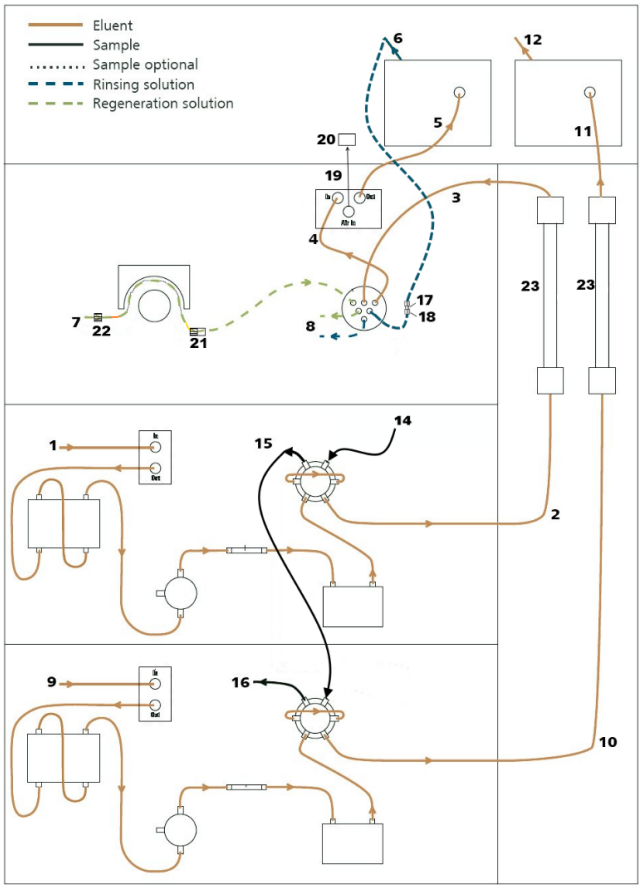
Then fix the white weight (6.2744.210), the adapter (6.2744.210), and the aspiration filter (6.2821.090) on the eluent aspiration tubes, all the while being careful not to touch the filters and its connections with bare hands in order to avoid cross-contamination.



Also fix the filled adsorber tubes on the eluent caps. Please refer to the 940 Professional IC Vario manual for a detailed description.

2.4. 940 Professional IC Vario

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



1. Connection to the eluent bottle for anion determination
2. Capillary to anion separation column
3. MSM inlet capillary – labeled with *In*
4. MSM outlet capillary – labeled with *Out,* which can be connected to the MCS with a long PEEK pressure screw (6.2477.090) or directly to the detector (5) using a coupling (6.2744.040).
5. Capillary from MCS to anion detector, a long PEEK pressure screw (6.2744.090) is used for the MCS connection
6. Detector outlet capillary connected to the MSM inlet capillary – labeled with *rinsing solution* – with the help of a coupling (6.2744.040)
7. Regeneration solution aspiration capillary – PTFE, 0.5 mm ID, connected to the bottle with 100 mmol/L sulfuric acid and the orange/yellow pump tubing. This pump tubing is, on the other side, connected 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
14. Sample aspiration capillary (PTFE, 0.5 mm ID), connected to the anion valve (position 1) and coming from the T-connector at the needle of the Sample Processor using a M6/UNF coupling on the T-connector.
15. Capillary connection (PTFE, 0.5 mm ID) between anion valve (position 2) and cation valve (position 1)
16. Capillary to port 1 of the sample handling 807 Dosing Unit
17. Coupling 2 x UNF 10/32 PEEK (6.2744.040)
18. PEEK pressure screw short (6.2744.070)
19. MCS air aspiration capillary – connected to the CO_2-adsorber cartridge
20. Luer coupling – connect with short PEEK pressure screw to CO_2-adsorber cartridge
21. Pump tubing connector with security lock and filter (6.2744.180)
22. Coupling nozzle - UNF 10/32 (6.2744.034)
23. The UNF 10/32 couplings (6.2744.040) are installed instead of the columns to rinse the system with eluent. After rinsing the columns are installed.

Make sure that all outlet capillaries are put into the waste collector and prepare the appropriate eluents for your separation columns. Pour them into the respective 2 L eluent bottles. 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.

For further installation instructions for the ion chromatograph, please consult the Application Bulletin: Installation Instructions for ProfIC Vario 1 AnCat (800105005EN).

2.5. 858 Professional Sample Processor

It is recommended to place the 858 Professional Sample Processor between the 940 Professional IC Vario and the 884 Professional VA to keep the transfer tubes of the sample as short as possible.

2.5.1. Cable connections

In the back of the 858 Professional Sample Processor, 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. If **viva** is running, make sure the 858 Professional Sample Processor is ignored and only configured in MagIC Net.

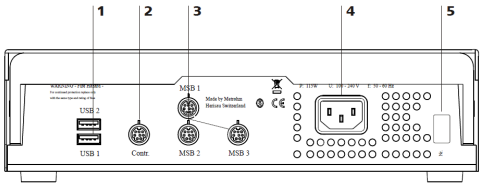


Figure 3 Connector strip 858 Professional Sample Processor

1	**USB connectors**	2	**Controller connector** For the connection to the PC
3	**MSB connectors** For dosing devices, stirrers, etc.	4	**Mains connection**
5	**Type plate**		

2.5.2. Hardware installations

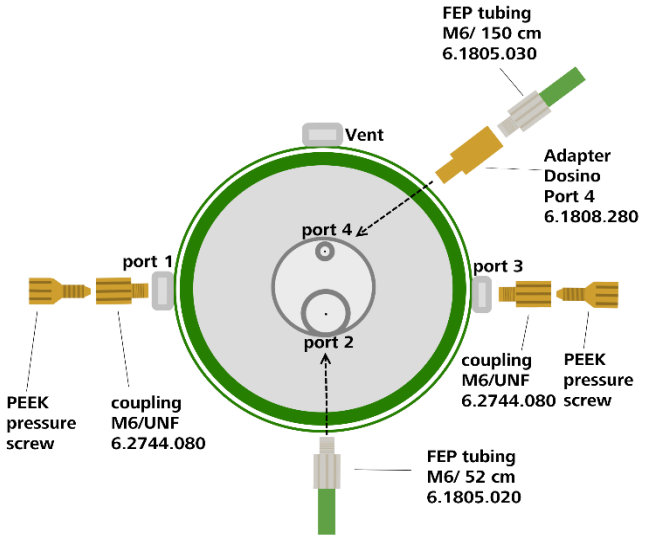
For a detailed installation description, please refer to the 858 Professional Sample Processor manual. In general, as soon as the IC is powered on and recognized by the software, the 858 Professional Sample Processor initializes and lifts its Swing Head.

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. The needle holder with M6 connector (6.2833.020) is used, which is supplied with the accessories of the VoltIC Professional 1. Screw the coupling piece 2 x M6 outer threads (6.1808.010) on the needle holder and screw the T-connector M6 (6.1808.060) on top.

Now, mount the retaining plate and the safety shield.

2.5.3. Liquid handling

Secure the 10 mL 807 Dosing Unit with the Dosino on top of the Dosino Holder of the IC. For this purpose, place the 807 Dosing Unit over the free hole of the Dosino holder and secure 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 position on the 858 Professional Sample Processor.



Port 1 of the 807 Dosing Unit is connected to the port 2 of the cation injection valve (number 16 in chapter 2.4) with the help of a PEEK capillary, 0.5 mm ID, two PEEK pressure screws, and a coupling M6/UNF (6.2744.250) for the connection at the 807 Dosing Unit port.

Port 2 of the 807 Dosing Unit is directly linked to the T-connector using a 52 cm length of FEP tubing (6.1805.020).

Port 3 of the 807 Dosing Unit is connected to the sample port of the 884 Professional VA using the PEEK capillary of 0.75 mm ID and 110 cm length (6.1831.080).

Finally, port 4 of the 807 Dosing Unit needs to be equipped with a 150 cm long FEP tubing (6.1805.030) leading to the waste. It can be installed using the Port 4 adapter (6.1808.280).

2.6. 843 Membrane Pump Station

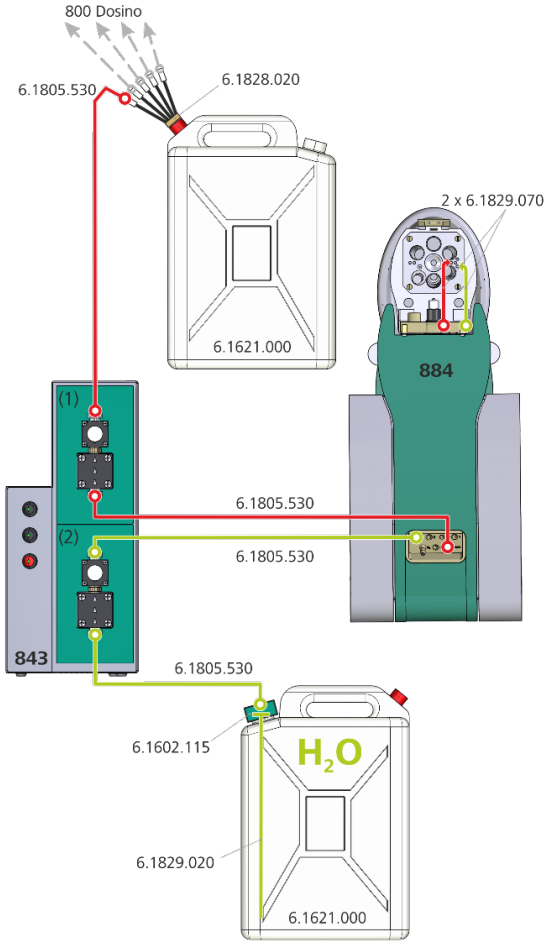
The rinsing and draining of the VA measuring vessel is done with an 843 Membrane Pump Station, which is installed near the 884 Professional VA. The 843 Membrane Pump Station is connected to the 858 Professional Sample Processor with the remote cable 6.2141.300. The individual pump cables are connected to the rear of the 858 Professional Sample Processor according to the labels on the cable.

Important: The 9-pin D-sub plug is connected to the «Remote 2» socket of the 843 Membrane Pump Station.

In the vicinity of the VoltIC Professional 1 system, the two canisters delivered with the 843 Membrane Pump Station are placed on the floor. One of the two 10 L canisters will be used as a waste canister and the other one as a reservoir of ultrapure water used for rinsing of the measuring vessel of the 884 Professional VA.

The 843 Membrane Pump Station is controlled via the 858 Professional Sample Processor in MagIC Net.

The tubing connections are installed as shown in the following picture.

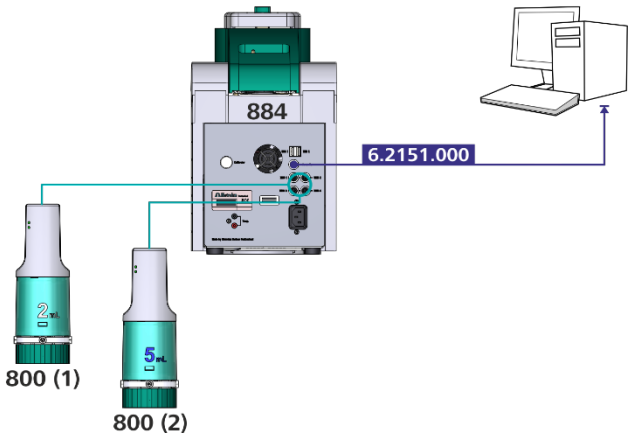


2.7. 884 Professional VA

2.7.1. Electrical connections

The installation of the semiautomated 884 Professional VA with an MME measuring head is done according to the following figures.

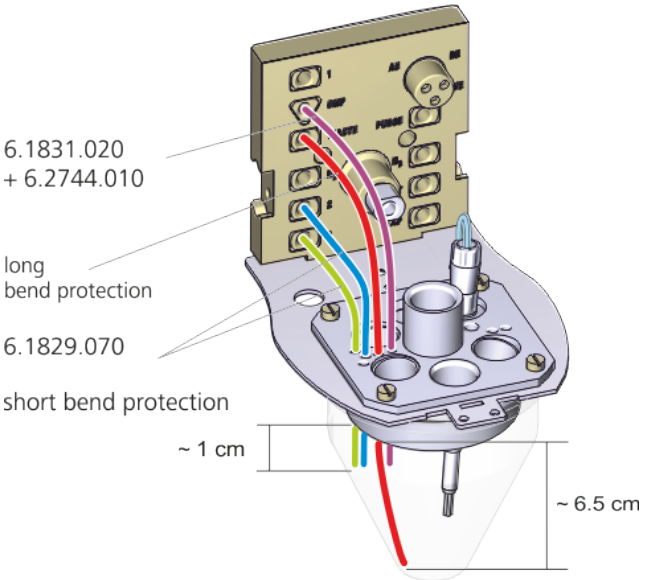
First, the electrical connections are installed as depicted below.

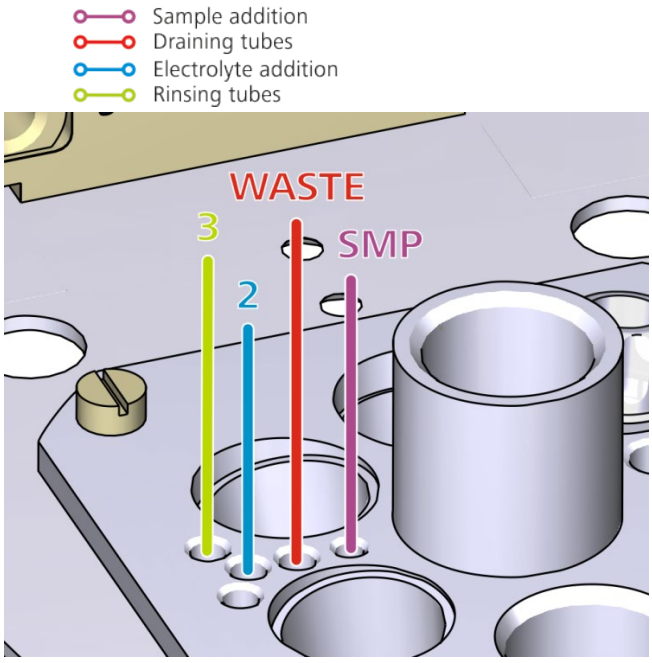


The 884 Professional VA is connected to the computer with a controller cable (6.2151.000). The 800 Dosinos are connected to the MSB ports on the rear of the 884 Professional VA. It is not important which MSB port is used for which Dosino. Note that the 858 Professional Sample Processor and the 843 Membrane Pump Station are not described here since they are both controlled by MagIC Net. For further information read chapters 2.5 and 2.6.

2.7.2. Measuring head tubing connections

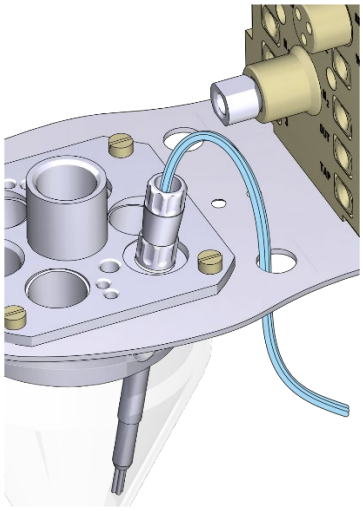
The MME measuring head is connected to the 884 Professional VA and equipped with the respective tubings in the following diagrams.





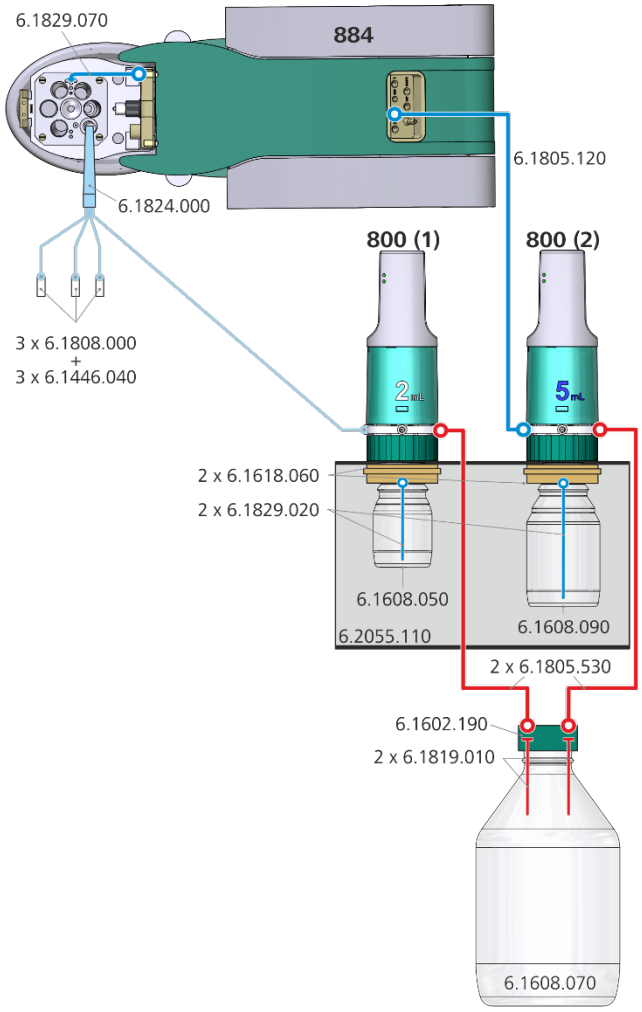
Care must be taken that the tubings of the tubing set 6.1829.070 are connected as described in the diagrams. One tubing with long bending protection is used for draining the measuring vessel, and two tubings with short bending protection are used for rinsing and for the addition of electrolyte. The sample is added via the «SMP» port using the PEEK capillary (6.1831.020) and a pressure screw (6.2744.010). All aforementioned tubings with exception of the draining tube are cut to approximately 1 cm below the PTFE insert of the measuring vessel using a capillary cutter. It is recommended to put the draining tube through the rightmost of the four 2 mm holes to be able to reach the lowest position of the measuring vessel. The length of the drain tube must be adjusted to reach the lowest point of the measuring vessel (6.1456.210). This is important, since the mercury will only be drained if the tubing is adjusted correctly. In addition, it is crucial that for draining the «WASTE» port is used to trap the Hg in the glass vessel to the left. Otherwise, the Hg will be transported into the waste container, or even worse—directly into the sewage system.

The four-fold micro dosing tip is directed through the hole to the right of the measuring head.



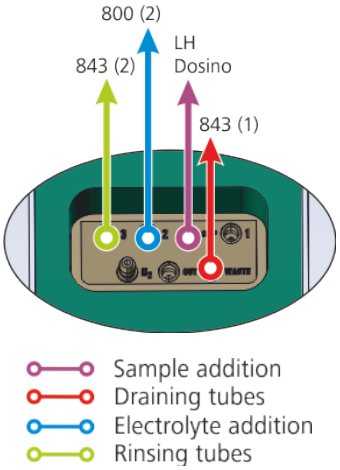
2.7.3. Tubing connections of the 807 Dosing Units

The tubing connections for the two 807 Dosing Units are installed as illustrated in the schematic below.



2.7.4. Rear of the 884 Professional VA

The tubings are installed on the rear of the 884 Professional VA as described in the following diagram.



Attach the bottles with electrolyte and standard solution to the respective 807 Dosing Units, go to the **viva** manual control, and prepare each 807 Dosing Unit twice.

3. MagIC Net and viva configuration

The instruments are connected to the computer via controller cables and their power is turned on. The drivers of the instruments are automatically installed.

After the drivers have been installed, first only MagIC Net is started. It is important that the 858 Professional Sample Processor is added to the devices in MagIC Net and not in **viva**, as only then the methods can work correctly.

3.1. MagIC Net configuration

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. It is recommended to keep the proposed names and call the solution of the 807 Dosing Unit «LH Dosino». The column can be named freely.

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).

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

3.1.1. Solution

The length of tubing and the port assignment for the 807 Dosing Unit need to be defined in the configuration table for solutions:

LH Dosino VoltIC	100 %	10	IDU	858 Professional Sample Processor…

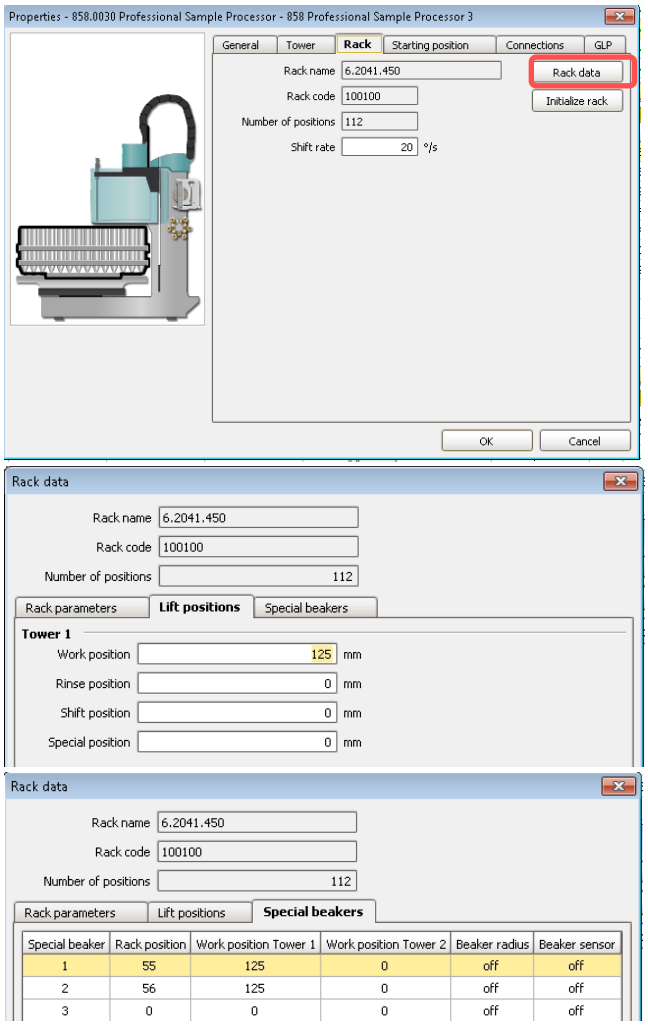
The following details should be entered for «LH Dosino VoltIC»:

	LH Dosino VoltIC		
	Port	Length	Diameter
Dosing Port Prep/Empty	Dosing Port 1	-	-
Dosing Port 1	Port 1	100.0 cm	0.5 mm
Dosing Port 2	Port 3	100.0 cm	0.8 mm
Fill Port	Port 2	53.0 cm	2.0 mm
Special Port	Port 4	150.0 cm	2.0 mm

3.1.2. 858 Professional Sample Processor

The correct installation of the accessories on the Swing Head is described in chapter 2.5.2. Please refer to this paragraph if the configuration has not been completed yet.

For the rack, the work and shift positions need to be defined. For this purpose open the rack data of the Sample Processor and adapt the rack parameters according to the numbers shown below.



3.1.3. RS-232 device

A new RS-232 device must be configured to establish the communication to **viva**. It is recommended to use the same name as is used in the corresponding MagIC Net method.

Name	RS 232 device 1

Communication properties:

COM Port	*Check the Windows device manager
Baud rate	9600
Data bit	8
Parity	None
Stop bit	1
Handshake	None
Timeout	2000 ms
Terminator for send	\0D\0A
Terminator for receive	\0D\0A
Code page	Cp437

3.1.4. 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 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 entire system is purged, reconnect the suppressor «Out» capillary back to the MCS or the detector.

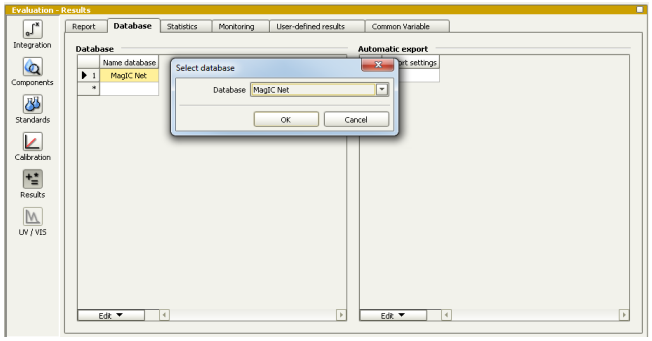
Now insert and rinse the precolumn for 10 minutes by leading the outlet directly into the waste. Afterwards connect the analytical column and flush it likewise for 10 minutes. Only then is the column completely connected to the flow path of the eluent (see the 940 Professional IC Vario manual for more details). 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 «Workplace», load the method, and press «Start HW».

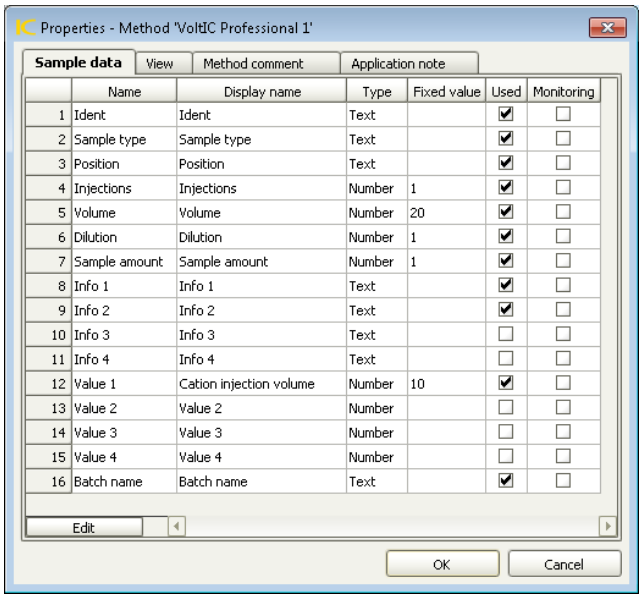
Equilibrate the system until the baseline is stable.

3.1.5. Modifications of the method

In the method window, under Evaluation, enter the ions and the concentrations of the required standards. The required database can be chosen under Evaluation → Results → Database if a different database other than «MagIC Net» is used.

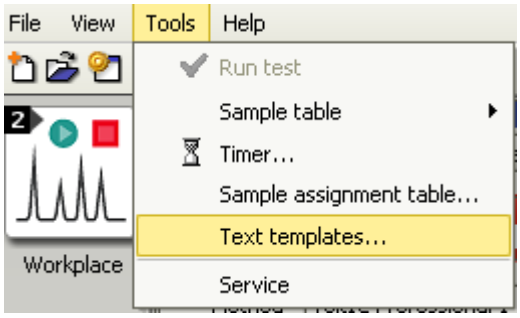


In the method properties, all fixed values can be adapted to the current setup:

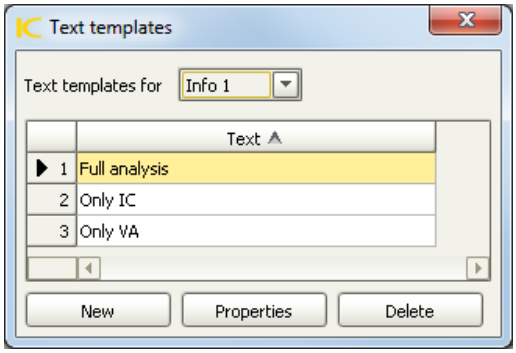


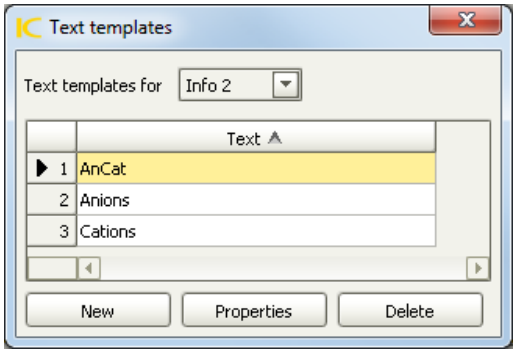
3.1.6. Workplace setup

In order to utilize the full flexibility of the VoltIC Professional 1 method, add some text templates in the workplace of MagIC Net. This will result in drop-down options in the determination table and easier handling for the user. Mainly, it will reduce the risk of typing errors.

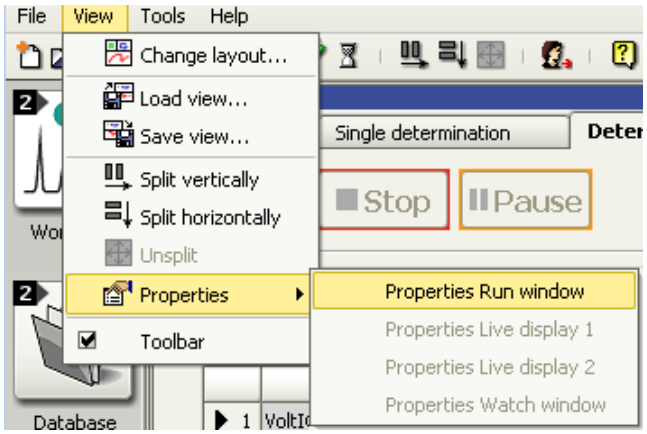


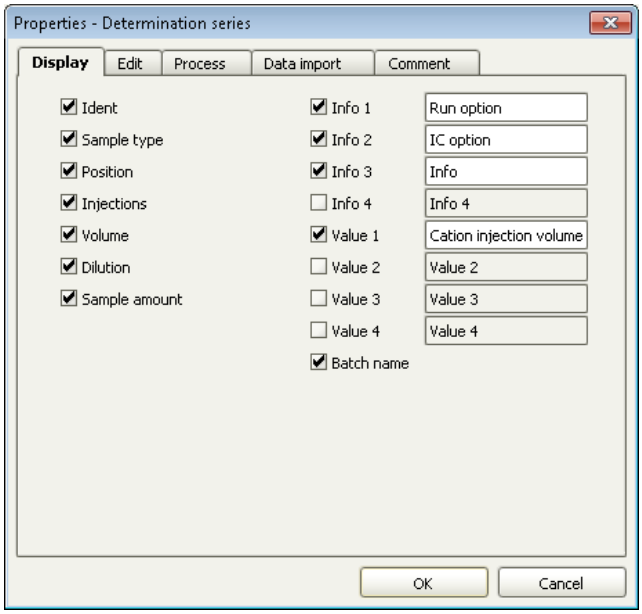
Create three text templates for Info 1 and Info 2 each according to the following figures. Make sure to type the phrases exactly as shown here—if not, the method will not work correctly. These text templates are used to conduct either a full analysis, IC only, or VA only. Additionally for IC, either only anions, only cations, or both anions and cations can be determined. This is described further in chapter 4.1.





Under view → properties, you can also adapt the displayed names for the «Info 1» and «Info 2» boxes to your liking. Similarly, edit the «Value 1» name to be «Cation injection volume». This volume can be fixed in the properties of the method and only altered there.



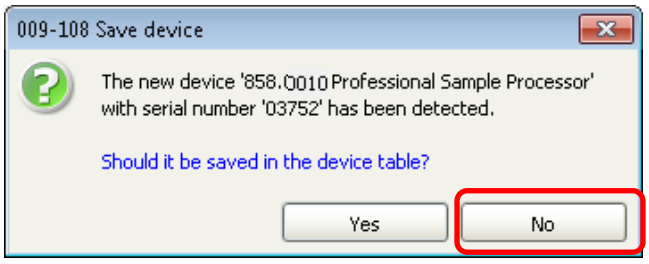


3.2. viva configuration

The 884 Professional VA is automatically recognized by **viva** after it has been connected. The corresponding dialog will pop up automatically.

Device name	884_1

The 858 Professional Sample Processor is not recognized by **viva** if it has already been claimed by MagIC Net. If **viva** has been opened first, please ignore the sample changer by denying the request to save the Sample Processor in the device table.



3.2.1. 807 Dosing Units

The 807 Dosing Units are automatically recognized after they have been connected to an 800 Dosino which is connected to the 884 Professional VA. The 807 Dosing Units should be configured with the parameters given below.

2 mL Standard:

Name	2 mL Standard

Parameters for preparation

Dosing port Prep/Empty	Dosing port 2
Dosing rate Dosing port 1	**2.0** mL/min
Dosing rate Dosing port 2	maximum mL/min
Dosing rate Fill port	maximum mL/min
Dosing rate Special port	maximum mL/min

Tubing parameters

	Port	Length	Diameter
Dosing port 1	Port 1	80 cm	0.3 mm
Dosing port 2	Port 3	0 cm	2 mm
Fill port	Port 2	12 cm*	2 mm
Special port	Port 4	0 cm	2 mm

Valve disk

Rotating direction	automatic

* to be measured

5 mL Electrolyte:

Name	5 mL Electrolyte

Parameters for preparation

Dosing port Prep/Empty	Dosing port 2	
Dosing rate Dosing port 1	maximum	mL/min
Dosing rate Dosing port 2	maximum	mL/min
Dosing rate Fill port	maximum	mL/min
Dosing rate Special port	maximum	mL/min

Tubing parameters

	Port	Length	Diameter
Dosing port 1	Port 1	138 cm	2 mm
Dosing port 2	Port 3	0 cm	2 mm
Fill port	Port 2	15 cm*	2 mm
Special port	Port 4	0 cm	2 mm

Valve disk

Rotating direction	automatic

* to be measured

3.2.2. Solutions

Solutions that should automatically be dosed have to be defined in the **viva** «Configuration» and need to be assigned to the respective 807 Dosing Unit.

The following table shows the solution names and assigned 807 Dosing Units as used in the **viva** method templates.

Solution name	***807 Dosing Unit***
Standard	2 mL Standard
Electrolyte	5 mL Electrolyte

Solution type for «Standard» has to be Standard solution, for «Electrolyte» Auxiliary solution is used.

3.2.3. RS-232 device

A new RS-232 device must be configured to establish the communication to MagIC Net. It is recommended to use the same name as used in the corresponding **viva** method.

Name	RS 232 device_1

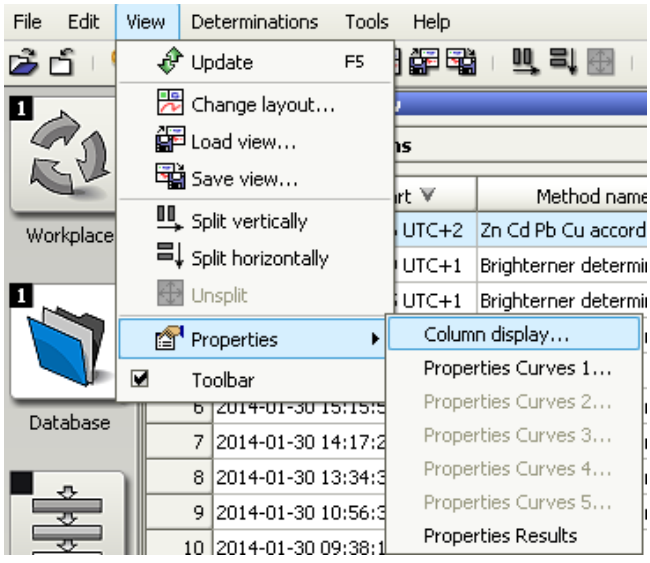
Communication properties:

COM Port	*Check the Windows device manager
Baud rate	9600
Data bit	8
Parity	None
Stop bit	1
Handshake	None
Timeout	2000 ms
Terminator for send	\0D\0A
Terminator for receive	\0D\0A
Code page	Cp437

3.2.4. Database

In the method, some identification parameters are transferred from MagIC Net to **viva**. In order to make finding the corresponding data set in **viva** easier, it is recommended to adapt the database view in **viva**.

Go to View → Properties → Column display:



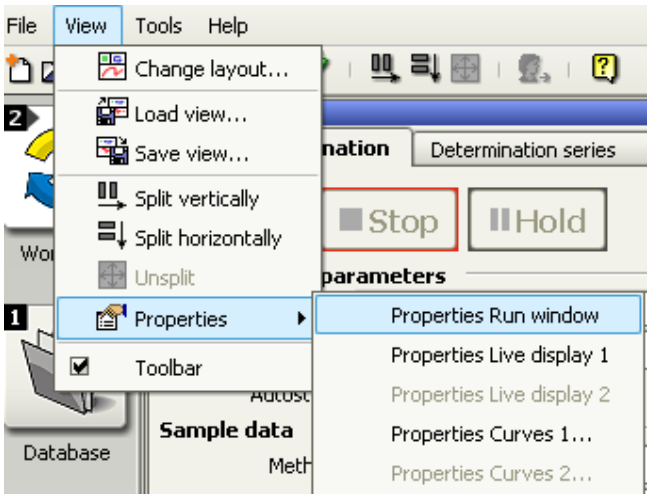
There, add the RS05.Value, RS06.Value, RS07.Value, and the results 01–04 Value and Unit to be displayed, and change the displayed names:

Columns displayed

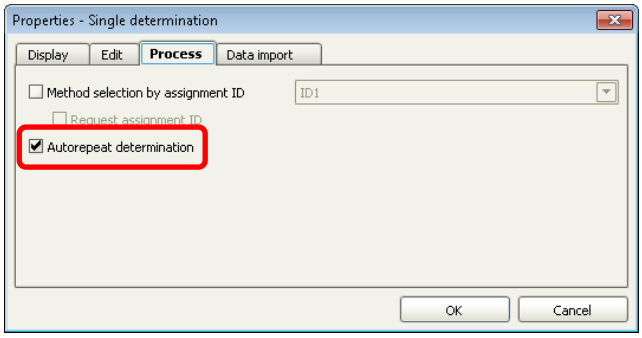
	Default name	Displayed name
1	Determination start	Determination start
2	RS05.Value	Ident
3	Method name	Method name
4	RS06.Value	Position
5	RS07.Value	Info 1
6	ID3.Value	ID3.Value
7	RS01.Value	Zn
8	RS01.Unit	Zn Unit
9	RS02.Value	Cd
10	RS02.Unit	Cd Unit
11	RS03.Value	Pb
12	RS03.Unit	Pb Unit
13	RS04.Value	Cu
14	RS04.Unit	Cu Unit

3.2.5. Workplace

In the workplace, go to View → Properties → Properties Run window:



In the process subtab, activate the “Autorepeat determination” option:



4. Methods

The MagIC Net and **viva** methods are described in brief.

4.1. General

There are several different paths through the methods, depending on the user's choice. Three main paths can be chosen using «Info 1», where the techniques to be applied must be selected.

Info 1	
«Full analysis»	Both techniques are carried out.
«Only IC»	The sample is only analyzed via IC (anions and cations).
«Only VA»	The sample is only analyzed with the VA part of the system for transition metals.

If IC analysis is chosen, the user can select if either anions or cations shall run independently or if both should be determined in parallel. The preferred option can be chosen in the «Info 2» field using a drop-down menu.

Info 2	
«AnCat»	Anions and cations are determined.
«Anions»	Only anions are determined.
«Cations»	Only cations are determined.

The **viva** method is automatically repeated and waits for inputs from MagIC Net.

4.2. MagIC Net method

4.2.1. Full analysis

1. MagIC Net checks if **viva** is running and the method has been started. If this is not the case, the user is asked to start the autorepeat determination via pop-up window.
2. MagIC Net sends all necessary sample information (IDENT, SAMPLE POSITION, and INFO 1) to **viva** for better traceability.
3. 1 mL of sample is aspirated into the PTFE tubing connected to Port 2 of the LH 807 Dosing Unit in order to avoid contamination from previous voltammetric determinations during the sample transfer to the IC.
4. Sample is aspirated through both of the IC loops and into the 807 Dosing Unit through Port 1.
5. The sample is injected into both IC channels and the determinations are started.
6. The 807 Dosing Unit and its connected capillaries are rinsed twice with the sample for the voltammetric determination.
7. The measuring vessel of the 884 Professional VA is rinsed thoroughly and prepared for sample delivery.
8. Sample is transferred to the measuring vessel. During the transfer the sample is already deaerated by a constant N_2 flow.
9. After the addition of 10 mL sample and 1 mL electrolyte the voltammetric determination is started. The quantification is done with two standard additions.
10. After the sample has been determined, the results are sent to MagIC Net which stores them as user-defined results.
11. The VA measuring vessel is emptied and rinsed in order to be prepared for the next measurement. The electrodes are stored in ultrapure water between the determinations.
12. The needle is rinsed by dipping it first in ultrapure water of the first rinsing vessel and aspirating a small volume of ultrapure water out of a second rinsing vessel.

4.2.2. Only IC

This main path is especially useful for calibration purposes.

1. 1 mL of sample is aspirated into the PTFE tubing connected to Port 2 of the LH 807 Dosing Unit in order to avoid contamination from previous voltammetric determinations during the sample transfer to the IC.
2. Sample or standard is aspirated through one or both of the IC loops and into the 807 Dosing Unit through Port 1.
3. The sample or standard is injected into one or both IC channels and the determination(s) are started.
4. The needle is rinsed by dipping it first in ultrapure water of the first rinsing vessel and aspirating a small volume of ultrapure water out of a second rinsing vessel.

4.2.3. Only VA

1. MagIC Net checks if **viva** is running and the method has been started. If this is not the case, the user is asked to start the autorepeat determination via pop-up window.
2. MagIC Net sends all necessary sample information (IDENT, SAMPLE POSITION, and INFO 1) to **viva** for better traceability.

3. The 807 Dosing Unit and its connected capillaries are rinsed twice with the sample for the voltammetric determination.
4. The measuring vessel of the 884 Professional VA is rinsed thoroughly and prepared for sample delivery.
5. Sample is transferred to the measuring vessel. During the transfer the sample is already deaerated by a constant N_2 flow.
6. After the addition of 10 mL sample and 1 mL electrolyte the voltammetric determination is started. The quantification is done with two standard additions.
7. After the sample has been determined, the results are sent to MagIC Net which stores them as user-defined results.
8. The VA measuring vessel is emptied and rinsed in order to be prepared for the next measurement. The electrodes are stored in ultrapure water between the determinations.

4.3. viva method

The method for VoltIC Professional 1 in **viva** is basically semiautomated since all automation except for the addition of electrolyte and standard solution is done by MagIC Net.

1. First, **viva** waits for the request from MagIC Net, if it is ready for operation. Sample data is received and stored.
2. Then **viva** waits for a signal from MagIC Net indicating the start of the sample transfer. Stirring and purging are switched on in order to deaerate the sample already during the transfer time.
3. Electrolyte is added to the sample and the electrode test is carried out. Then the solution is purged again for 3 minutes to remove residual oxygen in the measuring solution.
4. The sample is measured and quantified by two standard additions.
5. At the end of the determination the two 807 Dosing Units are filled again, and the results are individually transferred to MagIC Net.
6. The VA results are stored in the database and the next determination is carried out.

5. Determination

5.1. Starting a determination series

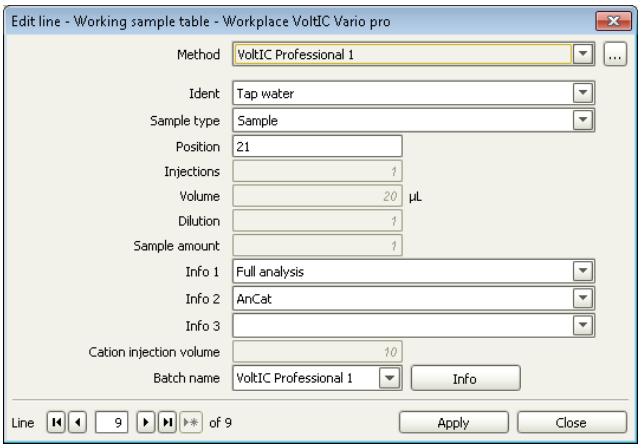
In MagIC Net, click on «Start HW» with your method loaded. Before starting a determination series, both baselines (Anion and Cation) have to be stable.

The analyte solutions are put on the rack.

In **viva**, prepare the two 807 Dosing Units and fill in a single determination. Make sure to choose the correct method. Then click on «Start».

The method is now running in a loop and will always turn back to the first TRANSFER command when one sample run is finished. There, it waits for the input from MagIC Net.

In the workplace window in MagIC Net, set up a «determination series» and describe samples by ident, sample position, sample type (standard, blank, or sample etc.) and choose one option each for «Info 1» and «Info 2».



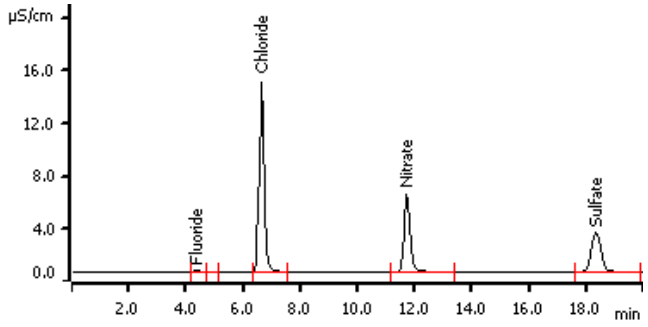
For evaluation and after recording the first chromatogram, check the retention times of the components. Since they depend on the column performance, you may have to adjust them in your method.

6. Example

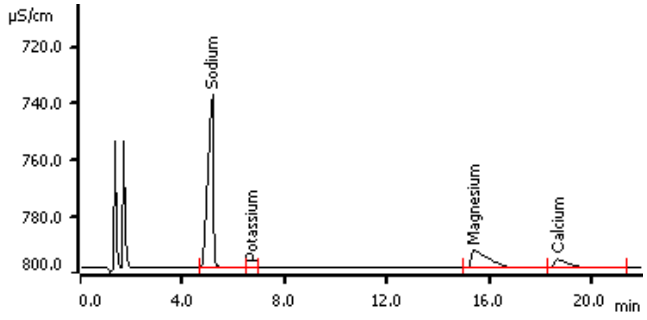
Anions, cations, and heavy metals were measured in a tap water sample. The corresponding Application Work is the AW IC CH6-1285-082016 / AW VA CH4-0554-082016.

6.1. Measuring curves

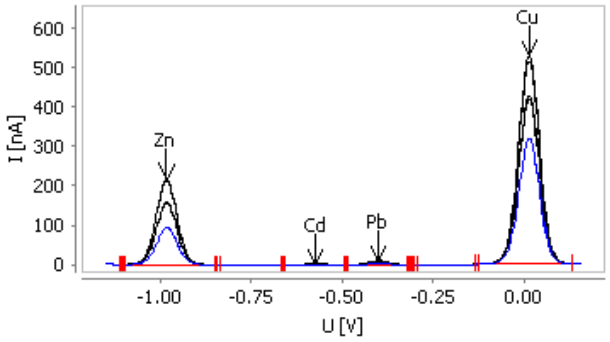
Chromatogram – Anions



Chromatogram – Cations



Voltammogram – Heavy metals



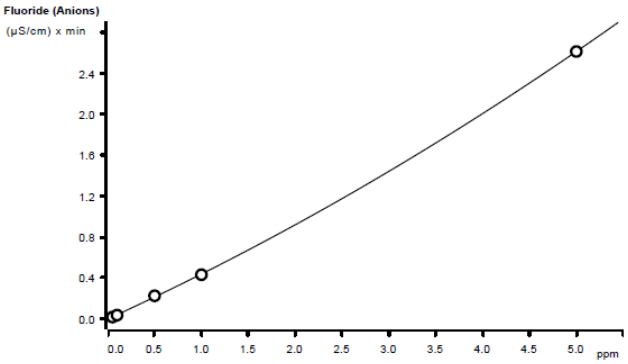
6.2. Results

F	0.04 mg/L
Cl	8.3 mg/L
NO_2	n.d.
Br	n.d.
NO_3	8.1 mg/L
PO_4	n.d.
SO_4	5.0 mg/L
Li	n.d.
Na	53.3 mg/L
NH_4	n.d.
K	0.4 mg/L
Mg	11.0 mg/L
Ca	9.0 mg/L
Zn	19.1 µg/L
Cd	n.d.
Pb	1.9 µg/L
Cu	77.2 µg/L

6.3. IC calibration

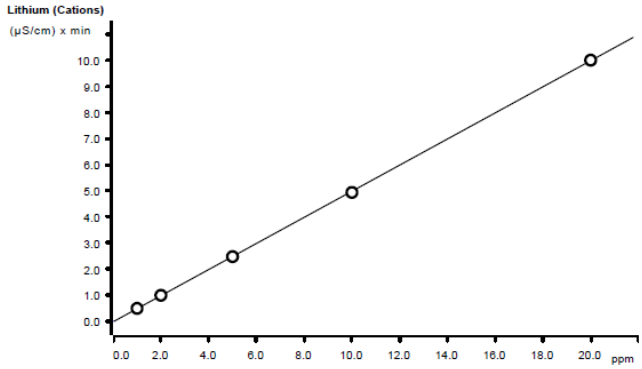
Fluoride is covered by a 1:100 calibration range; chloride, nitrate, and magnesium are calibrated in a range of 1:50; the sodium standards cover a 1:40 range, and nitrite, bromide, phosphate, sulfate, lithium, ammonium, potassium, and calcium are calibrated in a range of 1:20. For anions, a quadratic standard curve type was used for evaluation, while all cation calibration curves are linear. Two representative calibration curves are given here as examples:

	Correlation coefficient	Percentage standard deviation [%]
Fluoride	0.999977	1.60
Chloride	0.999998	0.34
Nitrite	0.999969	1.18
Bromide	0.999972	1.16
Nitrate	0.999947	1.62
Phosphate	0.999977	1.06
Sulfate	0.999970	1.19
Lithium	0.999976	0.82
Sodium	0.999989	0.51
Ammonium	0.999965	0.97
Potassium	0.999790	2.35
Magnesium	0.999990	0.57
Calcium	0.999907	1.51



Function: . . . . . A = - 2.64406E-3 + 0.0209268× Q + 5.22646E-5× Q²
Relative standard deviation . . . . . 1.597240 %
Correlation coefficient 0.999977

Sample type	Index	Conc.	Volume	Dilution	Sample amount	Area	Ident	Date
Standard 1	1	0.050	20.0	1.0	1.0	0.016	Calibration	2016-04-06 09:41:02 UTC+2
Standard 2	1	0.100	20.0	1.0	1.0	0.035	Calibration	2016-04-06 10:06:44 UTC+2
Standard 3	1	0.500	20.0	1.0	1.0	0.225	Calibration	2016-04-06 10:32:23 UTC+2
Standard 4	1	1.000	20.0	1.0	1.0	0.430	Calibration	2016-04-06 10:58:05 UTC+2
Standard 5	1	5.000	20.0	1.0	1.0	2.613	Calibration	2016-04-06 11:23:53 UTC+2



Function: . . . . . A = - 0.0196700 + 0.0250074× Q
Relative standard deviation . . . . . 0.821954 %
Correlation coefficient 0.999976

Sample type	Index	Conc.	Volume	Dilution	Sample amount	Area	Ident	Date
Standard 1	1	1.000	20.0	1.0	1.0	0.494	Calibration	2016-04-06 09:41:02 UTC+2
Standard 2	1	2.000	20.0	1.0	1.0	0.995	Calibration	2016-04-06 10:06:44 UTC+2
Standard 3	1	5.000	20.0	1.0	1.0	2.477	Calibration	2016-04-06 10:32:23 UTC+2
Standard 4	1	10.000	20.0	1.0	1.0	4.937	Calibration	2016-04-06 10:58:05 UTC+2
Standard 5	1	20.000	20.0	1.0	1.0	10.005	Calibration	2016-04-06 11:23:53 UTC+2

6.4. Repeatability

The precision of 6 injections of check standard solutions was determined. The results are shown in the following table.

n = 6	Prepared concentration [mg/L (IC) or µg/L (VA)]	Result [mg/L (IC) or µg/L (VA)]	Relative standard deviation [%]
F	0.5	0.5	3.9
Cl	25	25.2	0.07
NO_2	5	4.9	0.6
Br	5	4.8	0.4
NO_3	25	24.7	0.3
PO_4	5	5.3	0.5
SO_4	5	4.8	0.1
Li	5	4.8	0.03
Na	25	24.7	0.05
NH_4	5	4.9	0.2
K	0.5	0.45	1.5
Mg	10	8.6	0.3
Ca	10	10.4	0.3
Zn	10	10.3	1.9
Cd	0.5	0.49	2.4
Pb	2	2.0	3.7
Cu	20	20.3	1.7

7. Optional equipment

7.1. 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:

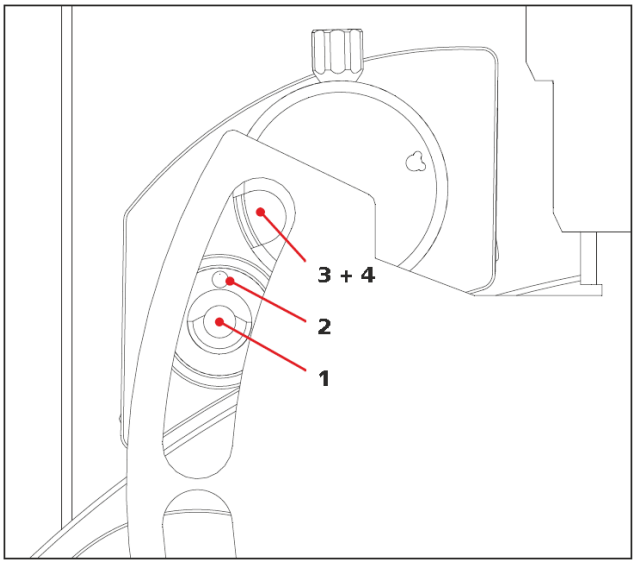
Nr	Article no.	Article designation
1	6.5330.130	IC Equipment: LQH Station

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 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 the LQH Station and the rack is 0.5–1 mm.

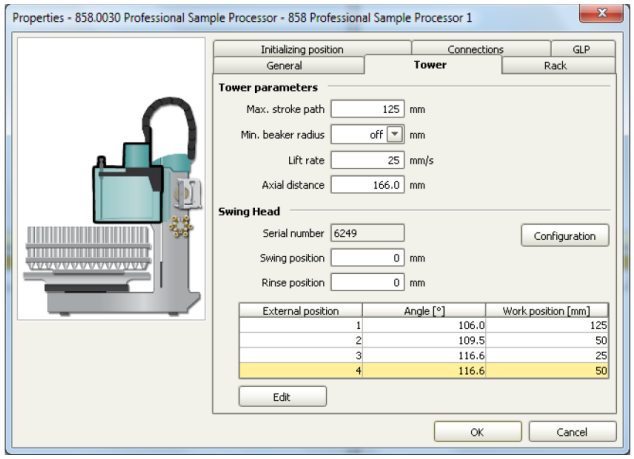
Now the Liquid Handling Station must be aligned with the retaining plate. For this purpose, loosen the big screw again and move the LQH Station underneath the retaining plate. Looking from above, you should now be able to see the small hole of the rinsing unit and part of the larger hole of the dilution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



When you have found the correct position, the screw must be tightened to secure 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.

For this purpose, 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.



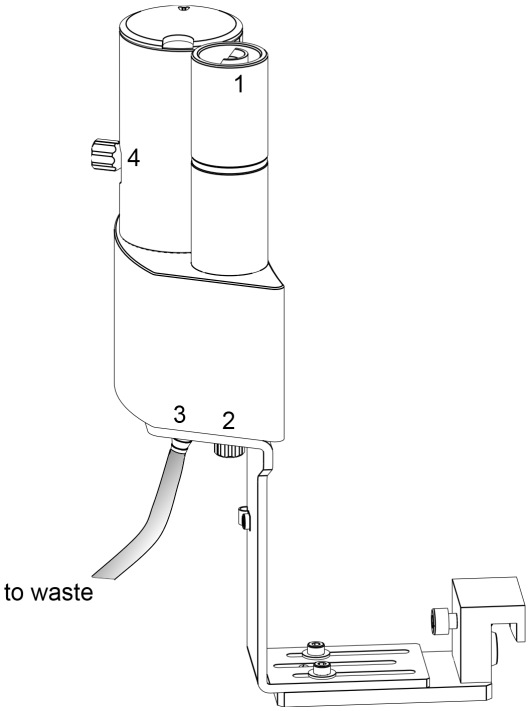
Depending on the available parts, the water supply to the Liquid Handling Station is managed differently. The inner tube of the rinsing unit is meant to be filled constantly with fresh ultrapure water, so the aspiration needle can be thoroughly cleaned from the outside.

All possibilities have the waste tube in common (number 3 in the following diagram). 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 on the following schematic). 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 enters the IC system.

Another possibility arises with a Dosino:

Similar to the setup with the peristaltic pump mentioned earlier, connect one of the Dosino ports with a capillary to the PEEK pressure screw (number 2 in the following schematic) 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 so that the water is cleansed from any impurities that could accumuate in the water reservoir.



Number 4 in 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.

7.2. 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.

7.3. Alternative MSM rinsing and regeneration methods

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