Metrohm

Application Bulletin 390

Installation Instruction for ProfIC Vario 14 Cation

ProfIC Vario 14 Cation is a Professional IC Vario system for the fully automatic determination of Cations using the **M**etrohm **i**ntelligent **P**ick-**u**p **T**echnique (MiPuT). MiPuT is the perfect choice when the sample volume is limited. An injection of 5 µL out of a vial filled with only 100 µL sample is precisely carried out and 95 µL remain in the vial for further use. It offers variable injection volumes in the range of 4 - 60 µL without compromise in accuracy.

In combination with the effective Liquid Handling Station, the «Pick-up Technique» allows the achievement of a mg/L to µg/L measuring range with virtually no carry-over (<0.0001%). The method complements the selected sample volume with transfer solution to 60 µL in order to have a constant sample/eluent ratio. Results of this are identical retention time, peak shape and the possibility for automatic calibration. «ProfIC Vario 14» safely masters all routine tasks in ion chromatography. It is simple to use and extremely reliable.

1. Delivery Package

Delivered with ProfIC Vario 14 Cation package:

No.	Article no.	Article designation
IC		
1	2.940.1100	940 Professional IC Vario ONE
Detector		
1	2.850.9010	IC Conductivity Detector
Accessories		
1	2.800.0010	800 Dosino
1	6.5330.170	IC Equipment: MiPuT
Sample processor		
1	2.858.0020	858 Professional Sample Processor - Pump
Optional		
1	6.6059.312	MagIC Net 3.1 Professional CD - 1 License
1	6.6059.313	MagIC Net 3.1 Multi - 3 licenses
1	6.2041.480	Sample rack 159 x 2 mL + 3 x 300 mL
1	6.1050.420	Metrosep C 4 - 150/4.0
1	6.1050.500	Metrosep C 4 Guard/4.0
1	2.941.0010	941 Eluent Production Module
1	6.2769.110	Sensor empty 2 L

2. Installation

Following is a detailed description of how to install ProfIC Vario 14 Cation.

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

2.1. Installation of the software

All programs must be shut down first. Make sure no Metrohm instrument is connected to the PC. Install MagIC Net with the help of the MagIC Net - CD. The Microsoft Installation Wizard is accepted and executed. All the standard directories proposed by the program should be accepted. Restart windows.

Now as soon as you connect a new Metrohm instrument via USB connection to the PC, the driver is installed and a window will pop up in MagIC Net, asking you if you'd like to store this device in your configuration. If you do, please answer with yes. The names will be checked later in this installation instruction.

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

Using the Accessory Kit Vario/Flex Basic, install the Professional IC Vario. Remove handle, place the detector block in the instrument and connect the detector cable, remove the transport locking screws, connect the leak sensor cable and connect the drainage tubing.

Then please 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.

In continuation, set up the waste collector by assembling the cap and screwing it onto the vessel. Then hang the waste collector holder on the side of the IC, so you can observe the droplets coming out of the capillaries later connected to the collector. Attach the waste tube to the vessel and lead it to the waste canister. When the tube is too long, please shorten it, because it is important to have a high level difference for the liquid to drain.

The power cable and USB cable (6.2151.020) are plugged into the rear of the Professional IC Vario. Please don't switch on the instrument yet. This step will follow after the completed installation.

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

In the box with the Accessory Kits ONE, you will find all the accessories for setting up the eluent bottle. Please lead the aspiration tube for the eluent through the M8 stopper, the O-ring 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 detailed description.

2.4. 858 Professional Sample Processor

Please place the 858 Professional Sample Processor **to the right of the IC** instrument. To optimize death volume, the capillaries are too short for an installation on the left side of the IC. 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 (6.9920.231) and the pick-up needle

(6.2624.100) with the help of the Needle/tube holder (6.2833.010). All those parts can be found in the IC equipment: MiPuT (6.5330.170) set. Also the safety shield has to be mounted (for detailed instructions please refer to the 858 Professional Sample Processor manual).

For the pick-up technique, low dead volume between the sample needle and the injection valve is essential. Therefore this setup includes a special ceramic/PTFE coated steel needle designed to puncture the closure of e.g. Eppendorf tubes.

The recommended sample rack 6.2041.480 comes with 159 positions for 2 mL sample tubes from Eppendorf.

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

2.4.1. Liquid Handling Station

The Liquid Handling Station consists of two function units: The rinsing unit is equipped with a two-pipe system and is used for rinsing the sample tube from the inside and outside or for fresh ultrapure water pick-up. The second unit is used for dilution of samples and standards. In the present case, the dilution unit won't be used.

The Liquid Handling Station is mounted onto the left side of the Sample Processor. For installation instructions, please refer to the Liquid Handling Station manual. All parts are included in the IC Equipment: MiPuT (6.5330.170). Please make sure that the Liquid Handling Station (LQH Station) is aligned properly before continuing the setup. All angle settings will be configured in a later step.

2.4.1.1. Rinsing unit

For this application, ultrapure water for rinsing will be supplied by the Peristaltic Pump on the 858 Sample Processor. The installation of the Peristaltic Pump tubing will be covered in one of the following paragraphs.

The waste tubing (6.1801.120) is fixed at the bottom of the rinsing station (3) to drain the waste water.

to waste
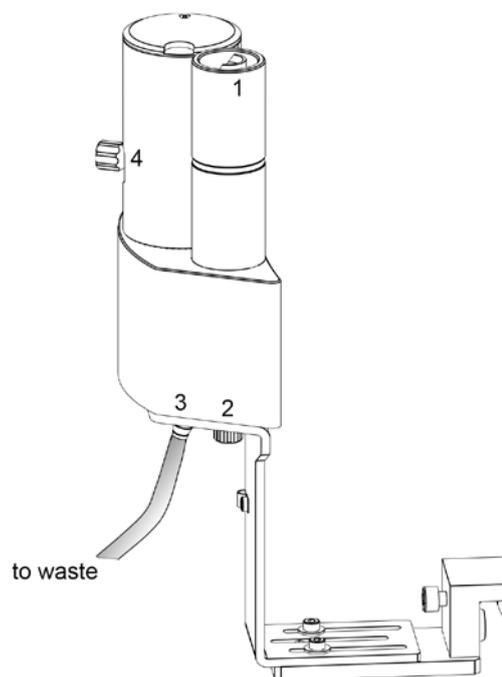
2.4.2. Ultrapure water

Ultrapure water is filled into a 2 L eluent bottle to serve as rinsing solution for the whole sample pathway. To make sure that there are no more air bubbles in the solution, degas the water before using it. The eluent bottle cap (6.1602.160) is equipped with the adsorber tube (6.1619.000), delivered with the Dosing Units, using the adapter for adsorber tube (6.1624.000). Furthermore, the FEP aspiration tubing to canister (6.1819.110) is passed through the M6 hole in the cap and cut to the appropriate length for it to reach the bottom of the water 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 = bigger hole, M6 = smaller hole)

2.4.3. Peristaltic Pump

Please equip the w/w Peristaltic Pump tubing (6.1826.360) out of the 858 accessories set with the coupling nozzle - UNF 10/32 (6.2744.034) and the pump tubing connector with security lock and filter (6.2744.180). Using part of the PTFE capillary (i.D. 0.5 mm, 6.1803.030) of the 858 Sample Processor equipment, connect the inlet of the Peristaltic Pump tubing to the ultrapure water bottle, by passing the capillary through the thread adapter of the M8 hole and the O-ring until it is well inserted in the water. The outlet of the tubing is led to the Liquid Handling Station (2). In between the Peristaltic Pump tubing and the LQH Station, an I-Trap (6.1014.200) is installed in order to cleanse the water going

to the Liquid Handling Station. For all these connections, the PTFE tubing (6.1803.030) out of the 858 Sample Processor equipment is used. For more instructions, please consult the Manual for the IC Equipment: MiPuT.

2.4.4. 800 Dosino

Please fix the Dosino with the Dosing Unit on the Dosino holder on the IC and plug it into the MSB1 at the rear side of the 858 Professional Sample Processor. The 858 must be switched off!

At port 1 of the 2 mL Dosing Unit, please connect the 2 mL Cation transfer capillary (6.1841.000) with the help of the M6 to UNF adapter (6.2744.080). Port 2 is used for ultrapure water supply; connect the Port 2 of the Dosino with the M6 hole in the bottle cap of the 2 L water bottle using the FEP tube connection (6.1805.120).
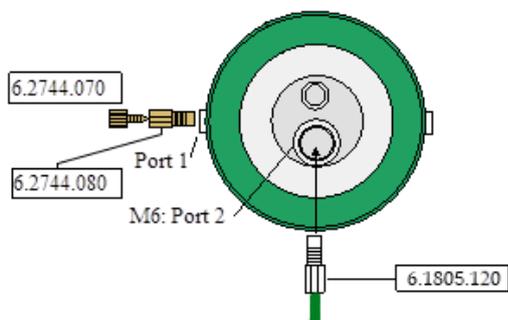
2.5. Interconnection of devices

The whole setup of Sample Processor, Dosino and the IC device is depicted in the following scheme.
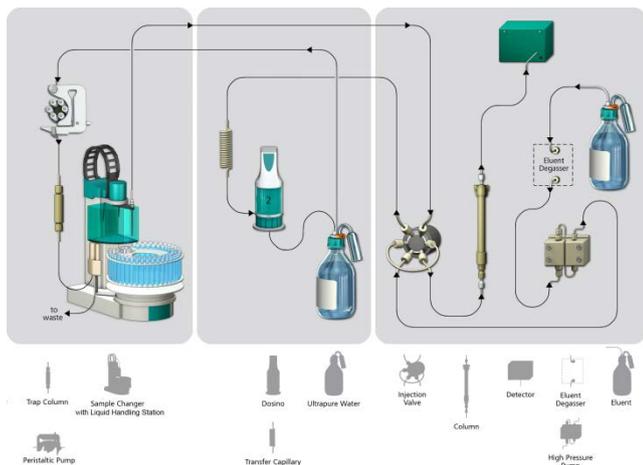
Prepare and degas an eluent suitable for the column you want to use (see column manual). For Metrosep C 4 - 150/4.0 a solution of 1.7 mmol/L HNO3 / 0.7 mmol/L dipicolinic acid (DPA, 2,6-Pyridinedicarboxylic acid) is adequate.

2.6. 940 Professional IC Vario

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

1. Connection to the eluent bottle for Cation determination
2. Capillary to Cation separation column
3. Detector inlet capillary
4. Detector outlet capillary, connected to the waste collector
5. 100 cm PEEK capillary (i.D. 0.25 mm, 6.1831.100) connection from the pick-up needle of the sample processor to the valve position 1 of the injection valve
6. 250 µL loop (6.1825.290) connected to the valve positions 3 and 6
7. 2 mL transfer capillary (6.1841.000) connection from the Cation valve position 2 to port 1 of the Dosino
8. The UNF 10/32 coupling (6.2744.040) is installed instead of the column to rinse the system with eluent. After rinsing the column will be installed.
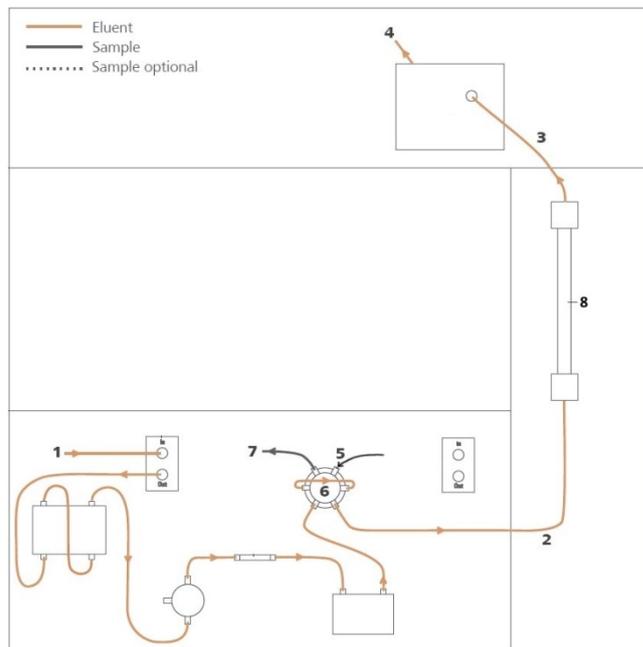
3. MagIC Net

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 and columns are stored in the configuration. The devices are predefined as "940 Professional IC Vario 1" and

"858 Professional Sample Processor 1". Name them accordingly, if other names appear in your configuration (e.g. due to changed setting on your computer). The Dosino connected to the 858 Professional Sample Processor should be labeled as "LH Dosino" (at MSB1).

In the window configuration, the 940 Professional IC Vario, the 858 Professional Sample Processor, the 800 Dosino, and the column Metrosep C 4 - 150/4.0 are visible. Add and define the eluent.

The settings for the rack require a "work position" fitting to the respective rack (e.g. 125 mm for rack 6.2041.760).
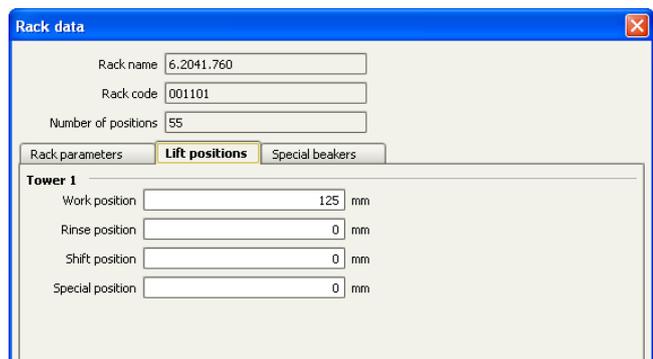
In order to configure the settings of the Liquid Handling Station, two external needle positions have to be defined for the tower of the Sample Processor in the window configuration. At the "external position 1" the needle should be in the inner compartment of the rinsing part which corresponds to an angle of approximately 106° and a work position of 125 mm. The exact angle depends on your installation. In this position, the needle is rinsed from the in- and outside.

For the outer compartment, an external position 2 is defined with an angle of ~109.5° and a work position of 80 mm. The external position 2 is used as drain position (e.g. to expel sample directly to the waste).

Be aware that the angles written above are only guidelines, for each installation the best position has to be determined with the help of the manual control window. Be sure to save the correct angles in the configuration.

In the sub window "Solutions" of the configuration, adjust the settings of the Dosino solution "LH Dosino" to 100% Ultrapure Water and in the Dosing unit subfolder adjust the parameters according to the following image:
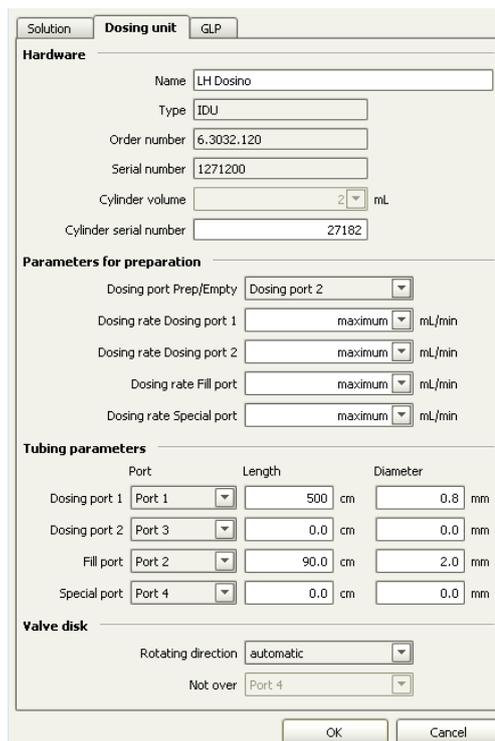
3.2. Method adjustment

The method for this configuration can be found in the example method folder on your MagIC Net - CD. In the window method manager, import the method for ProfIC Vario 14 Cation from the installation CD: Go to File → method manager and choose your method group. Afterwards, click on edit → import and choose the pathway on the installation CD:

(MagICnet\examples\methods\ ProfIC Vario \ProfIC Vario_1 4_Cation.imet).

In the window method, open the ProfIC Vario 14 Cation method and assign the following equipment: Define the rack of the connected sample processor, assign the eluent to the IC pump, define the Dosino solution (LH Dosino) and assign the column Metrosep C 4 - 150/4.0 to the analysis. Adjust the temperature of the column oven to 30°C.

Perform a method test and save the method.

3.3. Purge of the system

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

Now insert and rinse the precolumn for 10 minutes by leading the outlet directly into the waste. Afterwards connect the analytical column and flush it likewise for 10 minutes. Only afterward the column is completely connected to the flow path of the eluent (see also 940 manual). Please move the needle into the external position 2 for pre-rinsing. The

Dosino and the buffer tube can be rinsed by using the "prepare" button (under manual control).

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

Equilibrate the system until the baseline is stable.

3.4. User defined determination

In the window method, under evaluation, enter the concentrations of the required standards. With the method ProfIC Vario_14_Cation, only one standard is needed for calibration. The standard with the lowest concentration (standard 1) has to be prepared in the method (meant for mg/L analyte concentrations). All other standards have to be a multiple of standard 1, e.g. all components of standard 2 have twice the concentration as those in standard 1. Now please add the correct Database in the Evaluation window (Evaluation - Results - Database) for the method.
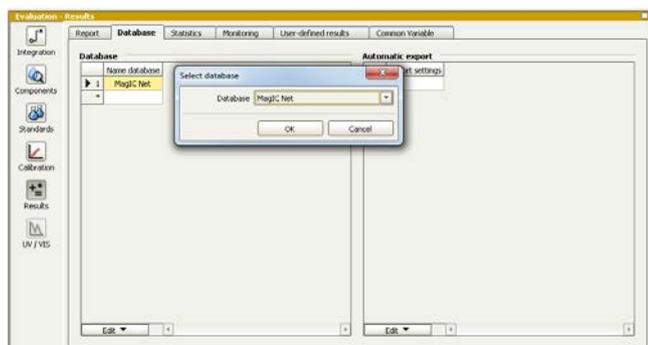
In the window work place, set up the "determination series", describing your samples by ident, vial number, sample type (standard, blank, or sample etc.). The volumes to be injected can vary from 5-50 µL, but for Standards, please always enter the volume of 5 µL.

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

An exemplary measurement was performed, including the cations lithium, sodium, ammonium, potassium, nickel, zinc, magnesium and calcium (5 µL injected). An exemplary chromatogram is shown below. The following chromatogram was recorded on a C 4 - 150/2.0.

Within the concentration difference of 1:10 (5 - 50 µL), a linear curve type was used for evaluation of cations. Correlation coefficients and standard deviations are listed in the following table. The calibration curve for lithium is shown as an example.

	Correlation coefficient	Percentage standard deviation [%]
Lithium	0.99999	0.24
Sodium	0.99998	0.46
Ammonium	0.99996	0.85
Potassium	0.99992	1.22
Nickel	0.99962	2.73
Zinc	0.99961	2.72
Magnesium	0.99998	0.57
Calcium	0.99997	0.79
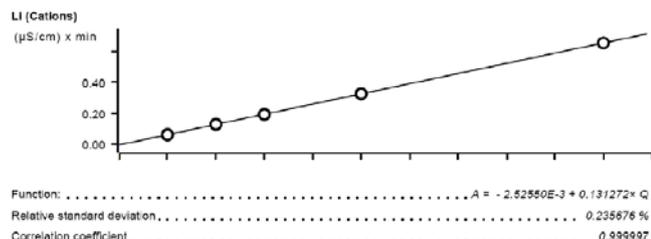
The precision of 6 injections of 10 µL standards was determined. The results are listed in the following table.

Ion	Mean value [mg/L]	Standard deviation absolute [mg/L]	RSD [%]
Lithium	0.10	0.5	99.7
Sodium	0.99	0.6	98.5
Ammonium	0.09	2.1	94.4
Potassium	0.50	0.9	100.7
Nickel	0.49	2.2	97.0
Zinc	0.48	3.9	96.2
Magnesium	4.98	0.5	99.6
Calcium	14.97	0.6	99.8

5. Optional equipment

5.1. 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.

The 941 can be connected to an ELGA flex ultra pure water system so that a continuous water supply can be guaranteed. The ELGA can be further integrated into the system as well for all the ultra pure water supplies.