

Application Bulletin 394

Installation instruction for ProfIC Vario 15 AnCat

ProfIC Vario 15 AnCat is a Professional IC Vario system for the fully automatic determination of anions with sequential suppression and cations. The **M**etrohm intelligent **P**artial Loop **T**echnique (MiPT) is applied on both channels.
«Partial Loop Injection» allows samples in mg/L to μ g/L range to be injected without previous dilution as well as multipoint calibrations using just one standard solution.
«ProfIC Vario 15» safely masters all routine tasks in ion chromatography. It is simple to use and extremely reliable.







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Delivery Package

| Delivered with ProfIC Vario 15 AnCat package: | | | | |
|---|----------------|--|--|--|
| Nr | Article no. | Article designation | | |
| IC | | | | |
| 1 | 2.940.2500 | 940 Professional IC Vario TWO/SeS/PP | | |
| Det | ector | | | |
| 2 | 2.850.9010 | IC Conductivity Detector | | |
| Acc | Accessories | | | |
| 1 | 2.800.0010 | 800 Dosino | | |
| 1 | 6.5330.180 | IC Equipment: MiPT | | |
| 1 | 6.1825.290 | PEEK sample loop 250 μL | | |
| 1 | 6.1841.000 | PEEK transfer capillary 2 mL, 5 m | | |
| 1 | 6.2744.080 | M6 thread/UNF 10/32 coupling | | |
| 1 | 6.2744.290 | T connector UNF 10/32 with holder | | |
| San | nple processor | | | |
| | | | | |
| 1 | 2.858.0010 | 858 Professional Sample Processor | | |
| Opt | tional | | | |
| 1 | 6.6059.302 | MagIC Net 3.0 Professional CD - 1 License | | |
| 1 | 6.6059.303 | MagIC Net 3.0 Multi - 3 licenses | | |
| 1 | 6.2041.440 | Sample rack 148 x 11 mL + 3 x 300 mL | | |
| 1 | 6.2041.760 | Sample rack 54 x 11 mL + 1 x 300 mL | | |
| 1 | 6.2743.040 | Sample tubes 2.5 mL | | |
| 1 | 6.2743.050 | Sample tubes 11 mL | | |
| 1 | 6.2743.070 | Stopper with perforation | | |
| 1 | 6.1030.420 | 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 | 941 Eluent Production Module | | |
| 1 | 6.5330.090 | IC Equipment: Additional eluent for 941 | | |
| 1 | 2.800.0010 | 800 Dosino | | |
| 1 | 6.2769.110 | Sensor empty 2 L | | |

2. Installation

Following is a detailed description of how to install ProfIC Vario 15 AnCat.

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 blocks in the instrument and connect the detector cables (anion detector into the "Detector 1" socket, the cation detector to "Detector 2"), 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 two boxes with the Accessory Kits ONE, you will find all the accessories for setting up the eluent bottles. Please lead the aspiration tubes for the eluents through the M8 stopper, the earring and the eluent caps. 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

For a detailed description, please refer to the 858 Professional Sample Processor manual. In general, you will have to do the following: Plug in the Swing Head connection cable and the power supply cable. The controller cable (6.2151.000) is plugged into the plug "Contr." on the 858 and connected to the IC device via USB. The sample processor initializes and lifts its Swing Head, once the IC device it is connected to is recognized by the software. For this, you have to plug the USB cable of the IC into the PC and turn it on. Afterwards, it is possible to mount the retaining plate, the needle and the safety shield (for detailed instructions please refer to the 858 Professional Sample Processor manual).

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

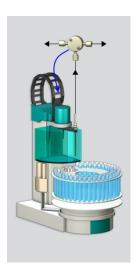
2.4.1. Liquid Handling Station

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: MiPT (6.5330.180). 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.

For this application, a stopper is used to close the outlet of the inner compartment of the rinsing area. The waste tubing is fixed at the bottom of the rinsing station to drain the waste water.

2.4.2. T-connector (6.2744.290)

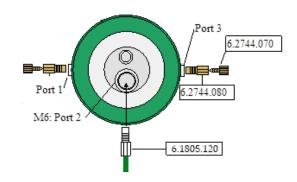
Please remove one element of the power chain, near the swing head with the help of a screwdriver and install the T connector into this hole. Connect the needle and the T connector with a short PEEK capillary (i.d. 0.5 mm).



2.4.3. 800 Dosino

Please fix the Dosino 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!

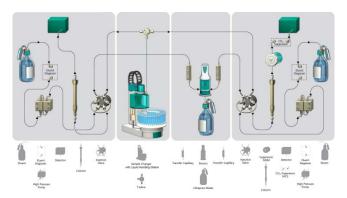
At port 1 of the 2 mL Dosing Unit, please connect the 2 mL anion transfer capillary (6.1841.000) and on port 3, please connect the 2 mL cation transfer capillary (6.1841.000), both with the help of the two M6 threads (6.2744.080). Port 2 is used for ultrapure water supply; therefore please insert the FEP aspiration tubing (6.1819.110) through the M6 hole in the bottle cap (6.1602.160) of the 2 L bottle included in the IC Equipment: MiPT set, following this step, 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). Please also make sure to close the M8 hole with its closer and to fix the adsorber tube (6.1619.000) and its adapter (6.1624.000) in the right place.



2.5. Interconnection of devices

The whole setup of Sample Processor, Dosino and the IC device is depicted in the following scheme. Anion and cation determination can be accomplished with one Professional IC Vario AnCat - MCS or with two IC devices. In both cases, the sample pathway is split after the Sample Processor by a T-connector.





Prepare and degas two eluents suitable for the anion and cation columns you want to use (see column manual). For Metrosep A Supp 15 - 150/4.0 a solution of 5.0 mmol/L Na $_2$ CO $_3$ / 0.3 mmol/L NaOH is recommended. For Metrosep C 4 - 150/4.0 a solution of 1.7 mmol/L HNO $_3$ / 0.7 mmol/L dipicolinic acid (DPA, 2,6-Pyridinedicarboxylic acid) is adequate.

Provide the regeneration solution for the MSM Suppressor (100 mmol/L sulfuric acid).

Ultrapure water is filled into the 2 L bottle at the Dosino to serve as rinsing solution for the whole sample pathway.

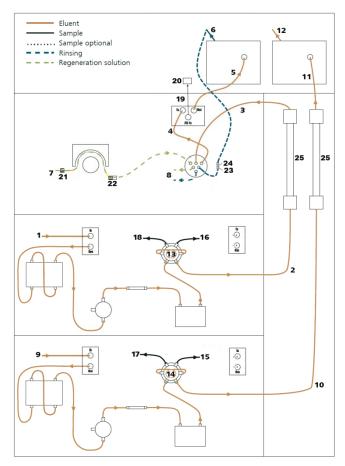
2.6. 940 Professional IC Vario

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

- 1. Connection to the eluent bottle for 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 or directly to the detector using a coupling (6.2744.040).
- Capillary from MCS to anion detector, long PEEK pressure screws (6.2744.090) are used for the MCS connection
- 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, relied to the regeneration solution capillary of the MSM labeled with *regenerant* with a security lock and filter (22)
- 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
- 13. 250 μ L loop (6.1825.290) connected to the valve positions 3 and 6, anion system
- 14. 250 μ L loop (6.1825.290) connected to the valve positions 3 and 6, cation system
- 15. 150 cm PEEK capillary (i.D. 0.5 mm) connection from the T connector on the sample processor to the valve position 1, cation system
- 16. 150 cm PEEK capillary (i.D. 0.5 mm) connection from the T connector on the sample processor to the valve position 1, anion system
- 17. 2 mL transfer capillary (6.1841.000) connection from the cation valve position 2 to port 3 of the Dosino
- 18. 2 mL transfer capillary (6.1841.000) connection from the anion valve position 2 to port 1 of the Dosino
- 19. MCS air aspiration capillary connected to the CO₂ adsorber cartridge
- 20. Luer coupling connect with short PEEK pressure screw to CO₂ adsorber cartridge
- 21. Coupling nozzle UNF 10/32 (6.2744.034)
- 22. Pump tubing connector with security lock and filter (6.2744.180)
- 23. Coupling 2xUNF 10/32 PEEK (6.2744.040)
- 24. PEEK pressure screw short (6.2744.070)
- 25. 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.





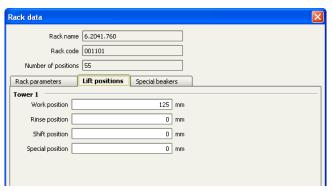
3. MagIC Net

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 columns Metrosep A Supp 15 - 150/4.0 and Metrosep C 4 - 150/4.0 are visible. Add and define the eluents and the suppressor solution.

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



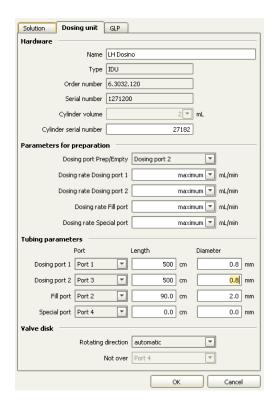
In order to configure the settings of the Liquid Handling Station, two external needle positions have to be defined for the tower of the Sample Processor in the window configuration. At the "external position 1" the needle should be in the inner compartment which corresponds to an angle of approximately 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 50 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

To be able to minimize the carry-over effects from the eluents, this AnCat setup is a combination of a separate anion and a separate cation channel, with only the T-connector and sample processor to share.

The methods in the example method folder on your MagIC Net - CD are designed for two separate systems with two 940 Professional IC Vario instruments. To be able to perform an AnCat analysis you need to modify the methods, so they fit to your instruments. The adjustments to be made are described in the following paragraphs.

In the window method manager, import the methods for ProfIC Vario 15 Anion and ProfIC Vario 15 Cation from the installation CD: Go to File \rightarrow method manager and choose your method group. Afterwards, click on edit \rightarrow import and choose the pathway on the installation CD:

(MaglCnet\examples\methods\ ProfIC Vario \ProfIC Vario_1 $5_Anion.imet$ and

MagICnet\examples\methods\

ProfIC Vario \ProfIC Vario_15_Cation.imet).

In the window method, open the two methods.

In the anion method, please assign the 2.940.2500 you have connected to the PC to the method. In a first step, do not name it correctly, but insert a "-" instead of the name.



Now, your method should look like this:



Please compare the names of all the IC instrument parts (pump, injector, ...) and adjust the names of the 2.940.**1500** parts to fit the ones of the newly added 2.940.2500 in the edit window, for example: Pump => Pump 1. Following this renaming, please remove the 2.940.1500 from the method. Confirm the warning message which is popping up. Now please name the 2.940.2500 "940 Professional IC Vario 1".

Then please untick the "Active" check mark of the Pump 2, Injector 2 and Conductivity detector 2. These elements will be needed in the cation method.

In the analysis "Anions", please assign "Conductivity" of the Conductivity Detector 1 as Data source, the Injection valve 1 and the Pump 1 to their respective field. Also, link the column Metrosep A Supp 15 - 150/4.0 method.

As a further step, please define the Dosino solution (LH Dosino), assign the anion eluent to the IC pump 1, match the suppressor solution to the IC peristaltic pump and switch it on with the rate 1, set the column temperature to 45°C and choose the right number for the rack of the sample processor.

Now you'll have to do the almost same thing for the cation method. In the cation method, add the device, rename the instrument components, remove the old IC, deactivate the Pump 1, Injector 1, Peristaltic Pump, MSM, MCS and Conductivity Detector 1.

Following this, choose the correct components in the analysis cation and add the column Metrosep C 4 - 150/4.0.



Assign the cation eluent to the IC pump 2, define the Dosino solution (LH Dosino), and define the rack of the connected sample processor. Fix the temperature of the column oven to 45° C.

Perform a method test and save the methods.

3.3. 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.

Now, please flush the system for about 10 minutes and get rid of 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 whole system is purged, reconnect the suppressor *Out* capillary back to the MCS or the detector.

Now insert and rinse the precolumns for 10 minutes by leading the outlets directly into the waste. Afterwards connect the analytical columns and flush them likewise for 10 minutes. Only afterward the columns are completely connected to the flow path of the eluent (see also 940 manual). Please move the Dosino 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). 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 work place, load the anion method, and press "Start HW".

Then open a second workplace for the cation method. This, you can do by choosing "File" - "Workplace" - "New...".

Name it as you wish and load the cation method. Press "Start HW" to start the equilibration of the system.

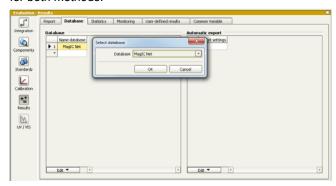
In this setup, anion and cation method are started in two separate workplaces, they share the Dosino and the Sample Processor. In this way you are able to set specific voluminas for each, anion and cation which will be considered in the calculations.

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 methods ProfIC Vario_15_Anion and ProfIC Vario_15_Cation, only one standard is needed for cation calibration and one standard is needed for anion calibration. The standard with

the lowest concentration (standard 1) has to be prepared in these methods (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 both methods.



For trace analysis, it is recommended to prepare the highest standard, as this one is more stable. If a trace setup is wished, the formula in the subprogram Standard, LH Dosino, Dosing needs to be changed from:

'SD.VOLUME' /1000 * 'SD.TYPEVALUE' to 'SD.VOLUME' /1000 / 'SD.TYPEVALUE' in both, anion and cation method.

In the window work place, setup up the two "determination series", describing your samples by ident, vial number, sample type (standard, blank, or sample etc.). The volumes to be injected can vary from 4-200 μL , but for Standards, please always enter the volume of 4 μL (200 μL for trace analysis).

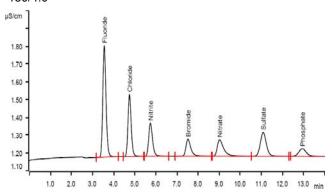
Be aware that with this setup, the risk for contamination by the eluent is too high for analyte concentrations below 5 μ g/L to be recommended. For such applications, please install a preconcentration configuration (ProfIC Vario 5).

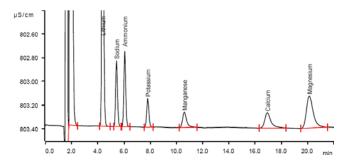
For evaluation and after recording the first chromatogram, check the retention times of your compounds. Since they depend on the performance of your column, you may have to adjust them in your methods.



4. Exemplary measurement

An exemplary measurement was performed, including the ions fluoride, chloride, nitrite, bromide nitrate, sulfate, phosphate, lithium, sodium, ammonium, potassium, manganese, calcium, and magnesium (8 μL injected, 2 mg/L of each component). Anions and cations were measured from separate sample vials with the sample for cation measurement being acidified by 100 μL nitric acid (2 mol/L) per 100 mL. The two following chromatograms were recorded in parallel on an A Supp 15 - 150/4.0 and a C 4 - 150/4.0

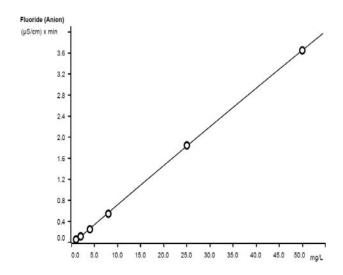




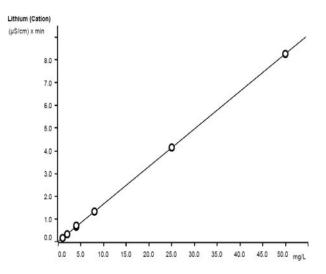
For the calibration, the average peak areas of 2 injections were evaluated. Within the concentration range of 1 - 50 mg/L (6 levels), a quadratic curve type was used for evaluation of anions, and a linear curve type was used for evaluation of cations. Correlation coefficients and standard deviations are listed in the following table. The calibration curves for fluoride and lithium are shown as examples.

| | Correlation coefficient | Percentage standard deviation [%] |
|----------|-------------------------|---|
| Fluoride | 0.999941 | 1.853 |
| Chloride | 0.999944 | 1.87 |
| Nitrite | 0.999964 | 1.499 |
| Bromide | 0.999987 | 0.886 |
| Nitrate | 0.999988 | 0.874 |
| Sulfate | 0.999974 | 1.265 |

| Phosphate | 0.999996 | 0.467 |
|-----------|----------|-------|
| Lithium | 0.999995 | 0.460 |
| Ammonium | 0.999999 | 0.204 |
| Potassium | 0.999999 | 0.212 |
| Manganese | 0.999941 | 1.853 |
| Calcium | 0.999944 | 1.870 |
| Magnesium | 0.999964 | 1.499 |



| 5 A 0.0006414 L | 0.01000070 0.54: |
|------------------------------|-----------------------|
| Function: $A = -0.0396414 +$ | 0.0189307 × Q - 2.547 |
| Relative standard deviation | 1.853% |
| Correlation coefficient | 0.999941 |
| Curve type | Quadratic |
| Weighting | 1 |

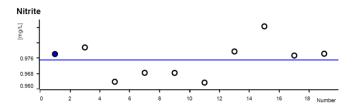


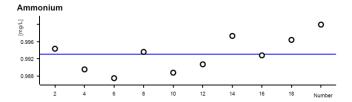


| Function: $A = 6.62584E-3 + 0.0413135 \times Q$ | | |
|---|----------|--|
| Relative standard deviation | 0.460% | |
| Correlation coefficient | 0.999995 | |
| Curve type | Linear | |
| Weighting | 1 | |

The precision of 10 injections of 1 mg/L standard ions was determined. The results are listed in the following table and exemplary trend charts are shown for nitrite and ammonium.

| Ion | Mean value | Standard | deviation |
|-----------|------------|-----------------|--------------------|
| | [mg/L] | relative [%] | absolute [mg/L] |
| Fluoride | 0.974 | 0.616 | 0.006 |
| Chloride | 0.984 | 0.711 | 0.007 |
| Nitrite | 0.975 | 0.923 | 0.009 |
| Bromide | 0.982 | 0.611 | 0.006 |
| Nitrate | 0.978 | 0.716 | 0.007 |
| Sulfate | 0.987 | 0.709 | 0.007 |
| Phosphate | 0.979 | 0.511 | 0.005 |
| Lithium | 0.976 | 0.205 | 0.002 |
| Ammonium | 0.992 | 0.403 | 0.004 |
| Potassium | 0.993 | 0.403 | 0.004 |
| Manganese | 0.990 | 0.606 | 0.006 |
| Calcium | 1.017 | 1.377 | 0.014 |
| Magnesium | 1.035 | 2.415 | 0.025 |







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.

5.2. Alternative MSM rinsing and regeneration methods

For alternative suppressor rinsing and regeneration methods and setups, please refer to the AB-395.