LiLiChro Ltd.
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LiLiCHRO

miniLiLi liquid-liquid (LLC) column

User manual

Version: 5.0
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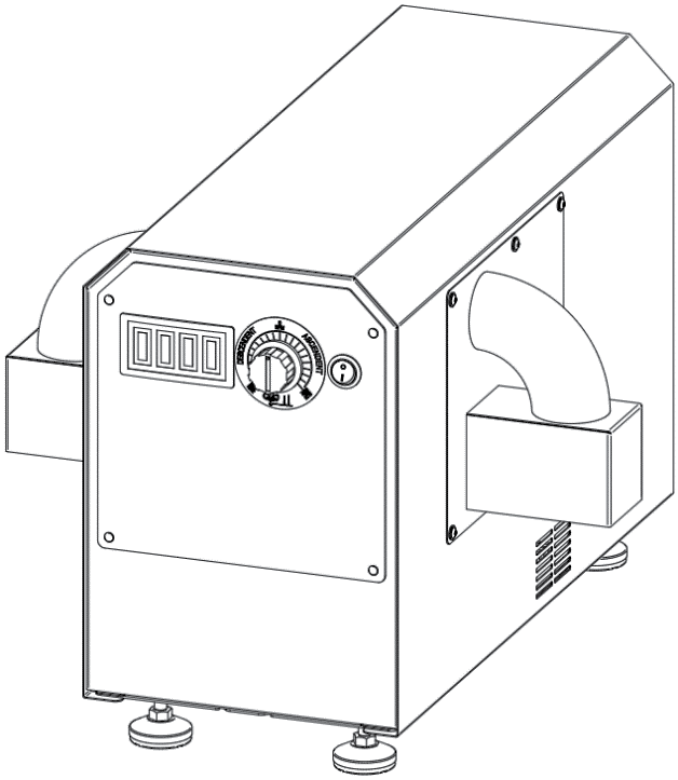
P/N: LC00090-F



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Welcome to the users of the miniLiLi LLC column!

miniLiLi is a laboratory-sized column, based on liquid-liquid Chromatography, which can be used in an HPLC system, installed in place of the column.

Before turning on your device, please read the manual!

Failure to follow these instructions may void your warranty and/or cause an accident. The device is operating at a speed of 1000-2000 rpm and under a voltage of 230 V, ignoring any safety regulations can cause a serious accident.

Please, check the installation location to see if it meets the parameters specified in the miniLiLi Column Site Requirements documentation (space, load capacity, energy, network voltage, power, peripherals, and data connection)! Study the documentation to find out what operations you can do without losing the warranty and when you should call the service center!

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MiniLiLi's controls

Front View

Mains switch

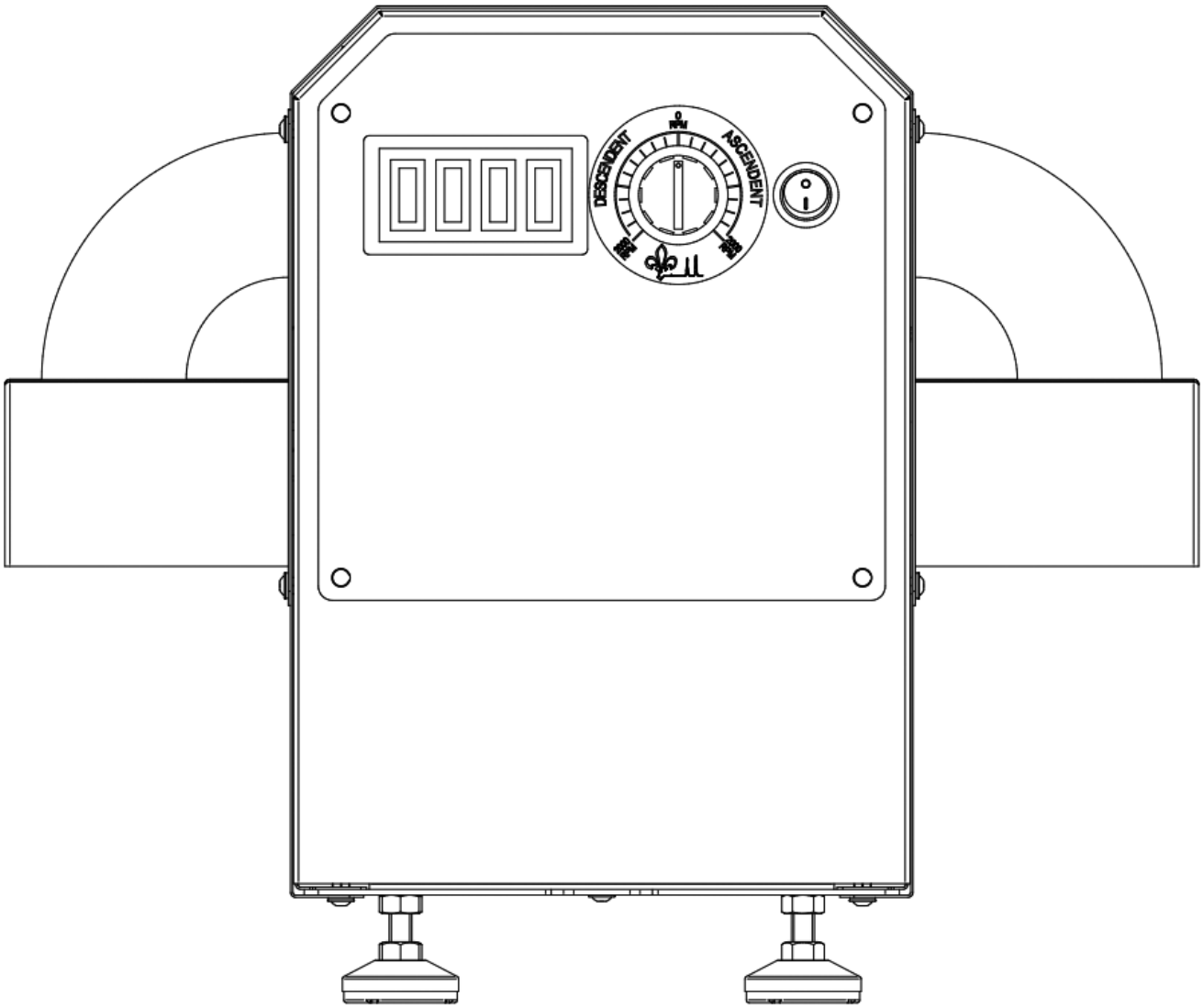
Network indicator (ON)

Speed (RPM, digital)

Speed setting

ASC In/Out, DSC In/Out connections

Emergency STOP



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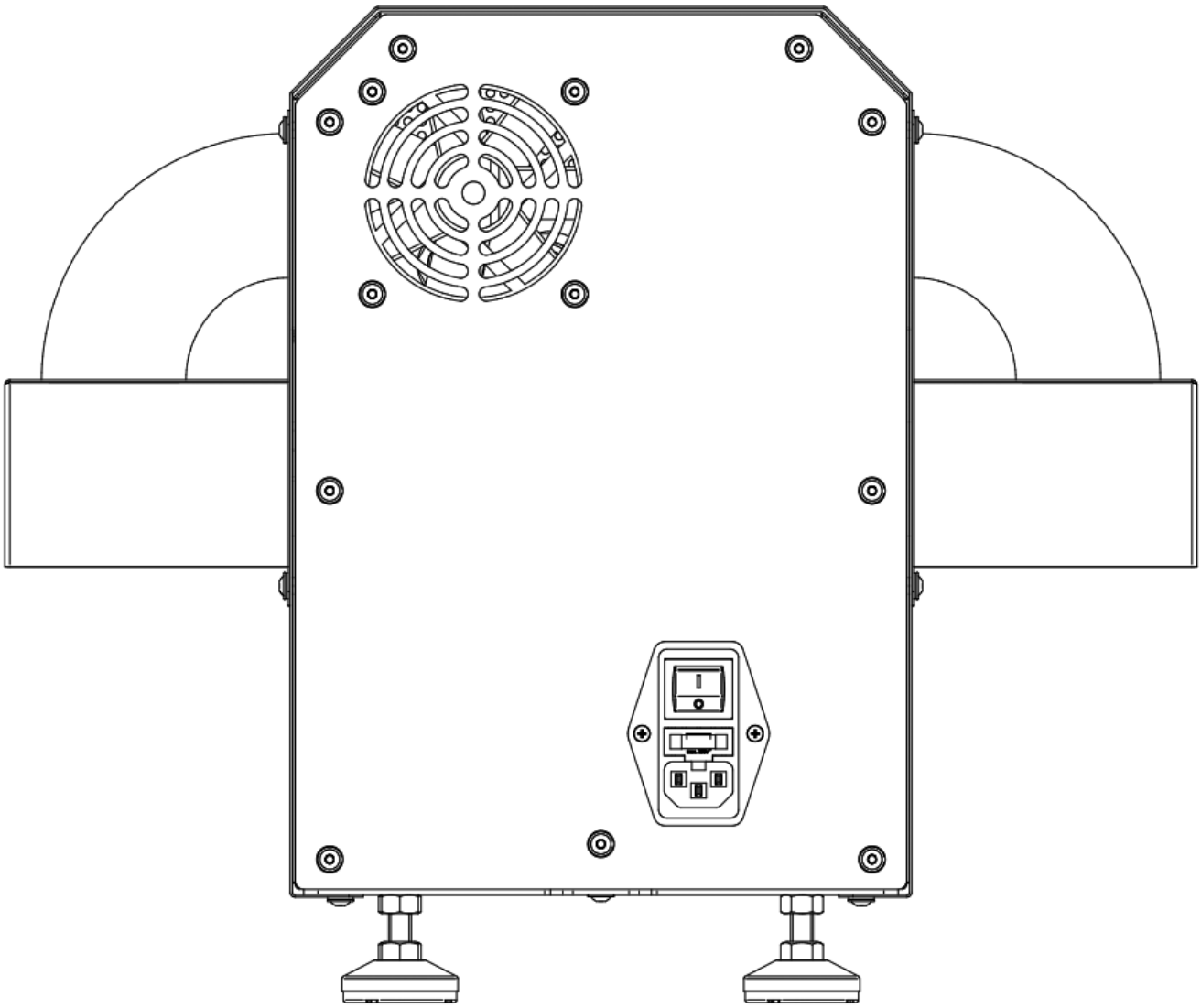
Back View

Network plug

Fuse

(Attention: before replacing the fuse, eliminate the error causing the fuse, then replace the blown fuse with a new one that is identical to the original one!)



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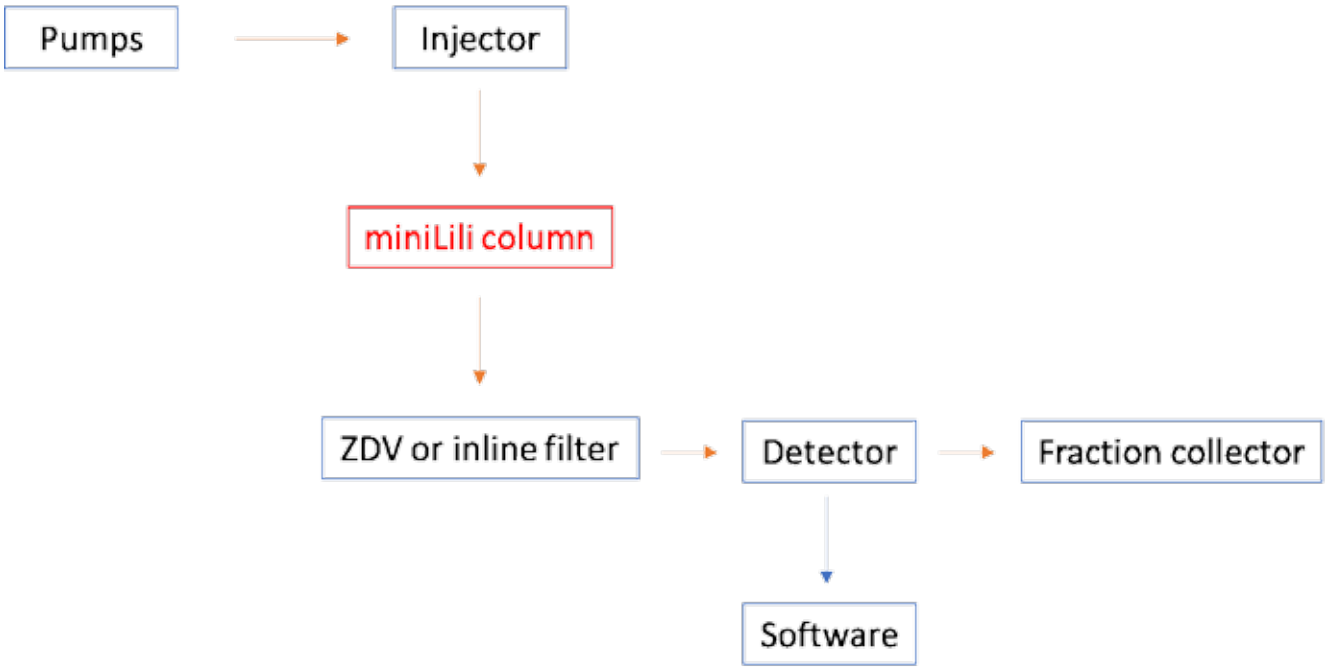
The main parts of the system

1, 80-cell, high-efficiency rotor. Volume 35-38 ml.
2, OD= 1/16' (1.6mm), minimum ID=0.01' (0.25mm) SS HPLC tubes.
3, Rotary union.
4, Drive, electronics

Necessary accessories for the system (miniLiLi not included)



1. HPLC pump (pumps, 2 pumps recommended for different phases).
2. Eluent tanks (stationary and mobile phase).
3. Injector or injector pump.
4. If a detector is connected to the system, an In-line filter or ZDV (zero dead volume unit) be in front of the detector.
5. Detector recommended with preparative cell (optional).
6. HPLC tubes, OD= 1/16' (1.6mm), minimum ID=0.005' (0.13mm) HPLC tubes.
7. Fraction collector (optional)

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Measurement

1. The miniLiLi device must be connected as a column in the user's own HPLC system. Set the maximum pump pressure to 100 bar (1450 psi). The column cannot be used at higher pressures.

   info: As the two phases (moving phase and stationary phase) are immiscible, it is advisable to use phase-dedicated pumps to prevent the pump valves from sticking when changing phases from the heavy to the light phase.
2. Select the LLC system mode you want to use (stationary or mobile phase, operating mode: ascending, ASC/descending, DSC.)
3. Mix and equilibrate the phases and filter it before use. Succinic acid and organic amines are recommended for pH adjustment, the permissible range of pH 2-10. Strong acids and alkalis can damage the rotor!
4. Filter the sample solution.
5. Turn on the device, and connect the HPLC pumps, (ASC IN or DSC IN corresponding to the chosen system), but DO NOT connect the detector and the fraction collector.
6. Connect a 10 ml measuring cylinder to the output (ASC OUT or DSC OUT).
7. Set the device to a low speed of approx. 200-250 rpm. Fill the column using the HPLC pump, flow rate 2-4 ml/min. The filling time is about 20 minutes. If you use descendent mode, first fill the column with water - pump connect to ASC IN inlet - then change the inlet (DSC IN) and fill the column with stationary phase.
8. If the stationary phase flows bubble-free at the output of the module, stop the pump and then connect the pump delivering the mobile phase. Four port connections unit aviable in the service pack. Raise the speed to the nominal speed of the measurement and start dosing the mobile phase at a rate of 1.0-3.0 ml/min (at which you want to perform the measurement). The mobile phase displaces a part of the stationary phase, after which equilibrium is established. Only the mobile phase now flows at the output. The device is ready for measuring. Retention can be calculated by reading the volume of the displaced stationary phase.
9. The outgoing (Out) branch must then be connected to the detector (and fraction collector) modules. If possible, conect the detector after column equlibration process. The mixed phases in tne detector cell give extra peaks.
10. The user can check the baseline, noise, and drift using his chromatography software. If all parameters are within the desired limits, start the measurement, inject the sample, and start data collection and fraction collection.
11. After eluting the components, the next measurement can start with unchanged parameters.

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After measurement

1. Neutralize the system if necessary.
2. If you want to set up a new system, flush the system with the appropriate solvents. Pay attention to the mixing rules and avoid clogging.
3. If you stop LiLiChro miniLiLi for a long time:

In ASC mode, fill and flush the system with a neutral solvent (i-propanol, water)
Do not leave the buffer prone to crystallization in the rotor and capillaries!
The clogged rotor can only be replaced!

Maintenance

- Due to the HPLC tubes used and applicable in the device, no contaminant can enter the rotor that gets stuck in the rotor - OD= 1/16' (1.6mm), ID=0.01' (0.25mm) - the rotor is it did not clog during testing. Normally we use ID 0.02”-0.04” (0,4-1,0 mm) Stainless or Peek tube th inlet side and ID 0.03” (0.8mm) PTFE tube the outlet side. Spare tube inclouded the service pack.
- It is extremely important to monitor the pressure and keep the maximum pressure of the pumps at the correct value. The pressure also depends on the speed used, the viscosity of the eluents, and the flow rate. - In most cases, the ZDV or in-line filter (connected in front of the detector gets clogged first), as the seal wears out during use. In such a case, the ZDV must be washed in countercurrent, or the in-line filter must be replaced. A lower speed reduces seal wear. Recommended speed 1000-1500rpm.
- The inlet tube or injector may be blocked if the sample solution or eluents were not properly filtered. Therefore, it is recommended to choose a smaller cross-section for the inlet pipe than the pipes used inside - OD= 1/16' (1.6mm), ID=0.005' (0.13mm) - the problem can be remedied by replacing the pipe or cleaning the injector.

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Troubleshooting

ERROR	REASON	ACTION TO REQUIRED	SOLUTION
Pressure drop	Leakage	Check connections and seals	Replace the faulty element. Call a LiLiChro' support/service!
Pressure rise	Blockage	Please contact LiLiChro' support / Service.	-
The device does not turn on	Electrical fault	Check the electrical network	Specialist (electrician)
Noisy baseline	Bubbles in the detector	Dilution of the outgoing eluent with ethanol.	
Noisy baseline	Bubbles in the detector	Improper stationary / mobile phase	Modify solvent system components