Sepiatec SFC-660
Operation Manual

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Table of contents

1 About this document

This operation manual is applicable for all variants of the instrument.
Read this operation manual before operating the instrument and follow the instructions to ensure safe and trouble-free operation.
Keep this operation manual for later use and pass it on to any subsequent user or owner.
BÜCHI Labortechnik AG accepts no liability for damage, faults and malfunctions resulting from not following this operation manual.
If you have any questions after reading this operation manual:

▶ Contact BÜCHI Labortechnik AG Customer Service.

https://www.buchi.com/contact

1.1 Mark-ups and symbols

NOTE

This symbol draws attention to useful and important information.

☑ This character draws attention to a requirement that must be met before the instructions below are carried out.

▶ This character indicates an instruction that must be carried out by the user.

⇨ This character indicates the result of a correctly carried out instruction.

Mark-up	Explanation
Window	Software Windows are marked-up like this.
Tab	Tabs are marked-up like this.
Dialog	Dialogs are marked-up like this.
[Button]	Buttons are marked-up like this.
[Field names]	Field names are marked-up like this.
[Menu / Menu item]	Menus or menu items are marked-up like this.
Status	Status is marked-up like this.
Signal	Signals are marked-up like this.

1.2 Trademarks

Product names and registered or unregistered trademarks that are used in this document are used only for identification and remain the property of the owner in each case.

1.3 Connected instruments

In addition to this operation manual, follow the instructions and specifications in the documentation for the connected instruments.

2 Safety

2.1 Proper use

The instrument is designed for super fluid chromatography.
The instrument can be used in laboratories for the following tasks:

- Purification
- Separation of one or more compounds from a mixture

2.2 Use other than that intended

Use of any kind other than that described in Chapter 2.1 "Proper use", page 7 and any application that does not comply with the technical specifications (See Technical data) constitutes use other than intended. In particular, the following applications are not permissible:

- Use of the instrument in areas which require explosion-safe instruments.
- Use of the instrument with solvents containing peroxides.

2.3 Staff qualification

Unqualified persons are unable to identify risks and are therefore exposed to greater dangers.
The instrument may only be operated by suitably qualified laboratory staff.
These operating instructions are aimed at the following target groups:

Users

Users are persons that meet the following criteria:

- They have been instructed in the use of the instrument.
- They are familiar with the contents of these operating instructions and the applicable safety regulations and apply them.
- They are able on the basis of their training or professional experience to assess the risks associated with the use of the instrument.

Operator

The operator (generally the laboratory manager) is responsible for the following aspects:

- The instrument must be correctly installed, commissioned, operated and serviced.
- Only suitably qualified staff may be assigned the task of performing the operations described in these operating instructions.
- The staff must comply with the local applicable requirements and regulations for safe and hazard-conscious working practices.
- Safety-related incidents that occur while using the instrument should be reported to the manufacturer (quality@buchi.com).

BUCHI service technicians

Service technicians authorized by BUCHI have attended special training courses and are authorized by BÜCHI Labortechnik AG to carry out special servicing and repair measures.

2.4 Personal protective equipment

Depending on the application, hazards due to heat and/or corrosive chemicals may arise.

- ▶ Always wear appropriate personal protective equipment such as safety goggles, protective clothing and gloves.
- ▶ Make sure that the personal protective equipment meets the requirements of the safety data sheets for all chemicals used.

2.5 Warning notices in this document

Warning notices warn you of dangers that can occur when handling the instrument. There are four danger levels, each identifiable by the signal word used.

Signal word	Meaning
DANGER	Indicates a danger with a high level of risk which could result in death or serious injury if not prevented.
WARNING	Indicates a danger with a medium level of risk which could result in death or serious injury if not prevented.
CAUTION	Indicates a danger with a low level of risk which could result in minor or medium-severity injury if not prevented.
NOTICE	Indicates a danger that could result in damage to property.

2.6 Warning symbols

The following warning symbols are displayed in this operation manual or on the instrument.

Symbol	Meaning
	General warning
	Instrument damage
	Dangerous electrical voltage
	Hot surface

2.7 Residual risks

The instrument has been developed and manufactured using the latest technological advances. Nevertheless, risks to persons, property or the environment can arise if the instrument is used incorrectly.
Appropriate warnings in this manual serve to alert the user to these residual dangers.

2.7.1 Faults during operation

If an instrument is damaged, sharp edges, glass splinters, moving parts or exposed electrical wires can cause injuries.

- Regularly check instruments for visible damage.
- If faults occur, switch off the instrument immediately, unplug the power cord and inform the operator.
- Do not continue to use instruments that are damaged.

2.7.2 Hot surfaces

The column oven area and the heating elements of the device can become hot. If touched they can cause skin burns.

- Do not touch hot surfaces or else wear suitable protective gloves.

2.7.3 Dangerous vapors

The use of the instrument can produce dangerous vapors that are capable of causing life-threatening toxic effects.

- Do not inhale any vapors produced during processing.
- Ensure that vapors are removed by a suitable fume hood.
- Only use the instrument in well ventilated areas.
- If vapors escape from connections, check the ferrules an fittings concerned and replace them if necessary.
- Do not process any unknown fluids.
- Observe the safety data sheets for all substances used.

2.7.4 Dangerous particles

The use of the instrument can produce dangerous particles that can cause life-threatening toxic effects.

- Do not inhale any particles produced during processing.
- Ensure that particles are removed by a suitable fume hood.
- Only use the instrument in well ventilated areas.
- If particles escape from connections, check the ferrules an fittings concerned and replace them if necessary.
- Do not process any unknown fluids or solvent mixtures.
- Observe the safety data sheets for all substances used.

2.7.5 Glass breakage

Broken glass can cause severe cuts.

Damaged glass components may implode if subjected to high pressure.

Minor damage to the ground joints impairs the sealing effect and may therefore diminish performance.

- Handle the flask and other glass components carefully and do not drop them.
- Always visually inspect glass components for damage every time they are to be used.
- Do not continue to use glass components that are damaged.
- Always wear protective gloves when disposing of broken glass.

2.7.6 Malfunction of a connected instrument (option)

A malfunction on a connected instrument can cause poisoning or death.

- ▶ Make sure that the connected instrument is prepared and maintained according to the user documentation.

2.7.7 Malware infection due to connections with other devices or network

Connections with other devices or a network can cause a malware infection to the instrument.

- ▶ Install antivirus software and firewall on the instrument before connecting it to other devices or network.

2.8 Modifications

Unauthorized modifications can affect safety and lead to accidents.

- ▶ Use only genuine BUCHI accessories, spare parts and consumables.
- ▶ Carry out technical changes only with prior written approval from BUCHI.
- ▶ Only allow changes to be made by BUCHI service technicians.

BUCHI accepts no liability for damage, faults and malfunctions resulting from unauthorized modifications.

2.9 Warning signs

⚠ DANGER

Risk of electric shock

Risk of death by electrocution

- ▶ Never remove the housing.
- ▶ Never operate the instrument with removed housing or without the included pumps.
- ▶ Never use any electric connections or cables that were not supplied with the instrument.

⚠ DANGER

Risk of cold burns

Pressurized gases can generate low temperatures on depressurization.

- ▶ Always wear appropriate personal protective equipment such as safety goggles, protective clothing and gloves.

⚠ DANGER

Hot surface

The column oven can reach temperatures of up to 70°C. The heating can heat up for some time even after switching off power, due to internally stored energy.

- ▶ Open door carefully so that the hot air can vent.
- ▶ Only change columns after complete cool down.
- ▶ Always wear appropriate personal protective equipment such as safety goggles, protective clothing and gloves.
- ▶ Make sure the ventilation slits and the oven heater are not covered.

⚠ WARNING

Pressurized gas

The operating instrument is pressurized. Venting of pressurized gases and solvents is possible. CO_2 is suffocating in high concentrations.

- ▶ Install gas detectors at site.
- ▶ Create sufficient ventilation.

⚠ CAUTION

Risk due to falling objects or material

Personal injury or property damage

- ▶ Never place solvent bottles or other items on the top of the instrument.

NOTE

The pumps switch off automatically when reaching 400 bar.

The detector flow cell is stable up to a pressure of 300 bar. To prevent damage to the flow cell, the back pressure regulator is programmed to switch off at 300 bar.

NOTE

The system my be pressurized after switching off.

Keep in mind, the escape of pressurized gases and solvents are possible.

3 Product description

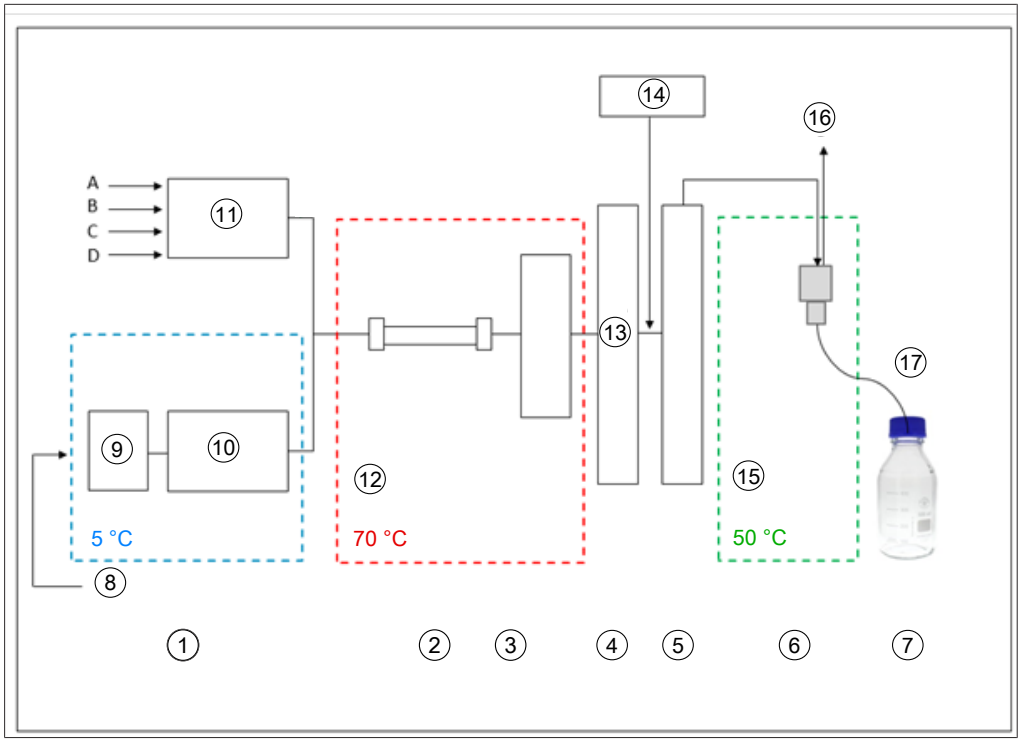
3.1 Description of function

The instrument is for preparative separations on small columns by means of supercritical fluid chromatography (SFC).

- Supercritical fluid chromatography has the ability to separate gram size samples.
- The integrated gas-liquid-separators (GLS) allow fast and efficient removal of CO_2 from separated components.

The instrument allows:

- A mix of supercritical CO_2 and one solvent.
  With the optional solvent selection valve, the solvent can be chosen from four different solvents.
- Injection of liquid or solid sample
- Separation on a column
- Detection of the components by using a UV, ELSD or MS detector.
- Collecting the desired fractions



1	Pumps	2	Column oven
3	Detector, flow cell	4	Back pressure regulator
5	Post-heater	6	Gas-liquid-separator
7	Fractions (8 pcs.)	8	Liquid CO_2 60 - 75 bar
9	Pre-cooling	10	CO_2 pump
11	Modifier pump	12	Supercritical CO_2 80 - 250 bar
13	BPR pressure value 80-250 bar	14	Add-on pump
15	Low pressure	16	CO_2 exhaust
17	Depressurized fraction collection		

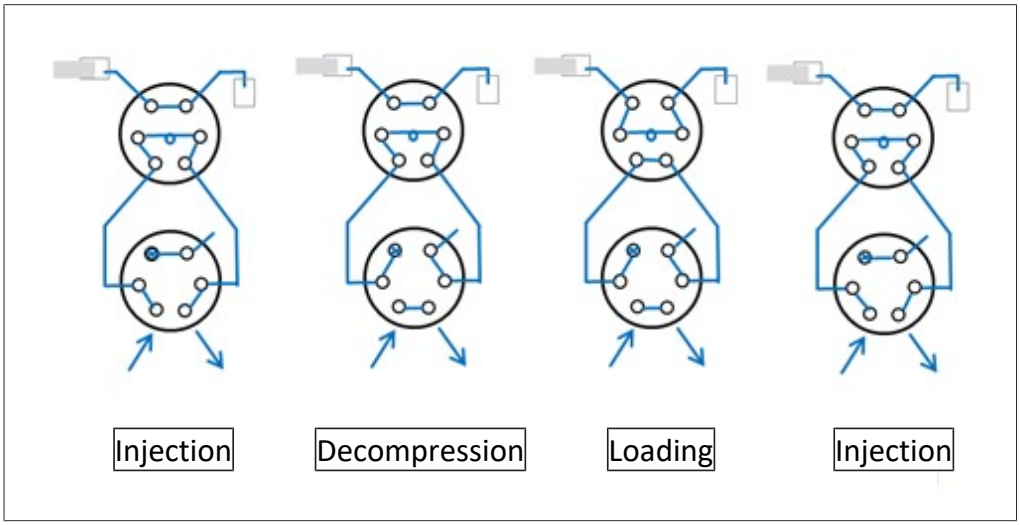
The mobile phase in supercritical fluid chromatography consists of a supercritical fluid and a liquid solvent. This fluid is a condensed gas above the critical temperature and pressure. Its properties are those of a gas and liquid. Carbon dioxide (CO_2) is mostly used.
The CO_2 is transported by the pump in a cooled state. The fluid gets heated above the supercritical temperature in the column oven and mixed with the modifier. The back pressure regulator maintains an elevated pressure to keep the mobile phase above the critical point of CO_2. The sample is then transported over the separation column by the supercritical fluid. A modifier can be added to improve the elution of organic substances.
After detection, the CO_2 is separated from the eluted substances. The Detection and Fraction valve position decides in which GLS the stream is going. The GLS separates most of the CO_2 and removes it from the system by an exhaust pipe and fed into the ventilation system.

The collected fractions contain the dissolved substances in the modifier and a small amount of residual CO_2 and optional in the organic from the add-on pump. The fractions are available in highly concentrated form for further analysis.

NOTE
If the instrument is operated with the optional CO_2 recycling module, the collection of fractions occurs under pressure.

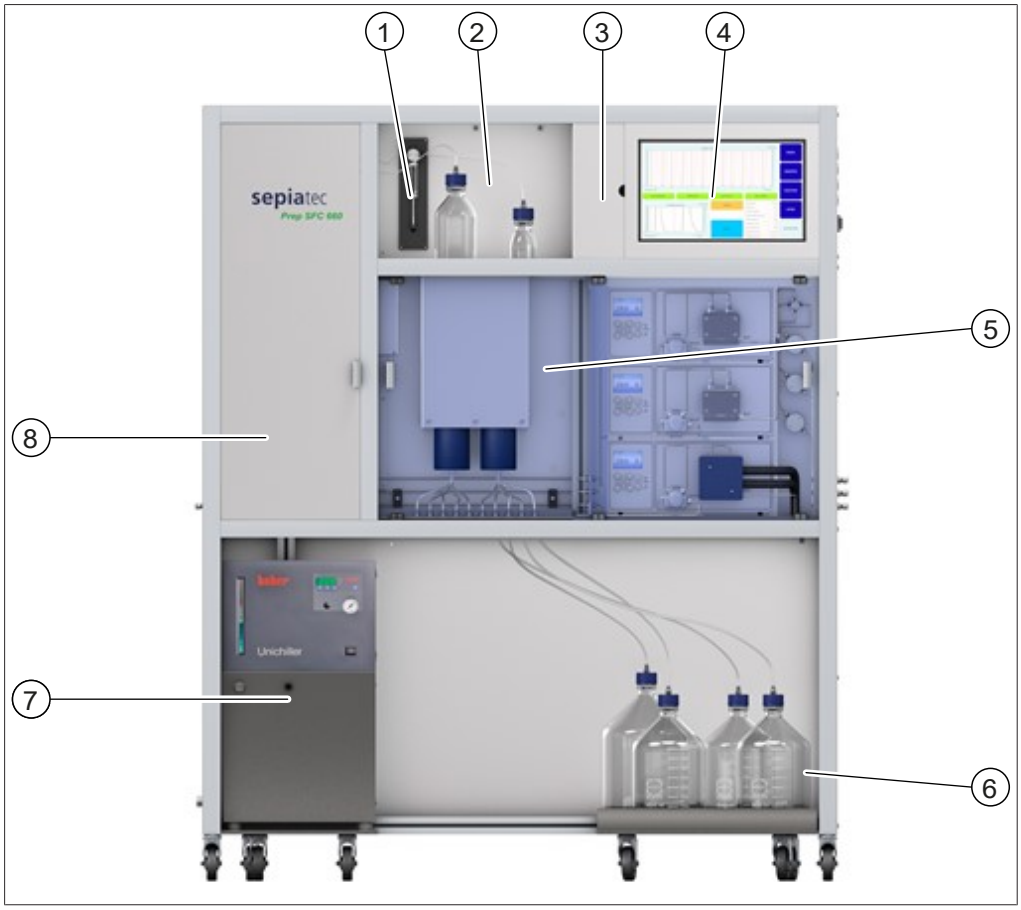
3.1.1 Injection procedure

The pressure in the sample loop can be released into a waste bottle in a controlled manner before loading and the pressure surge into the receiving bottle can be avoided.



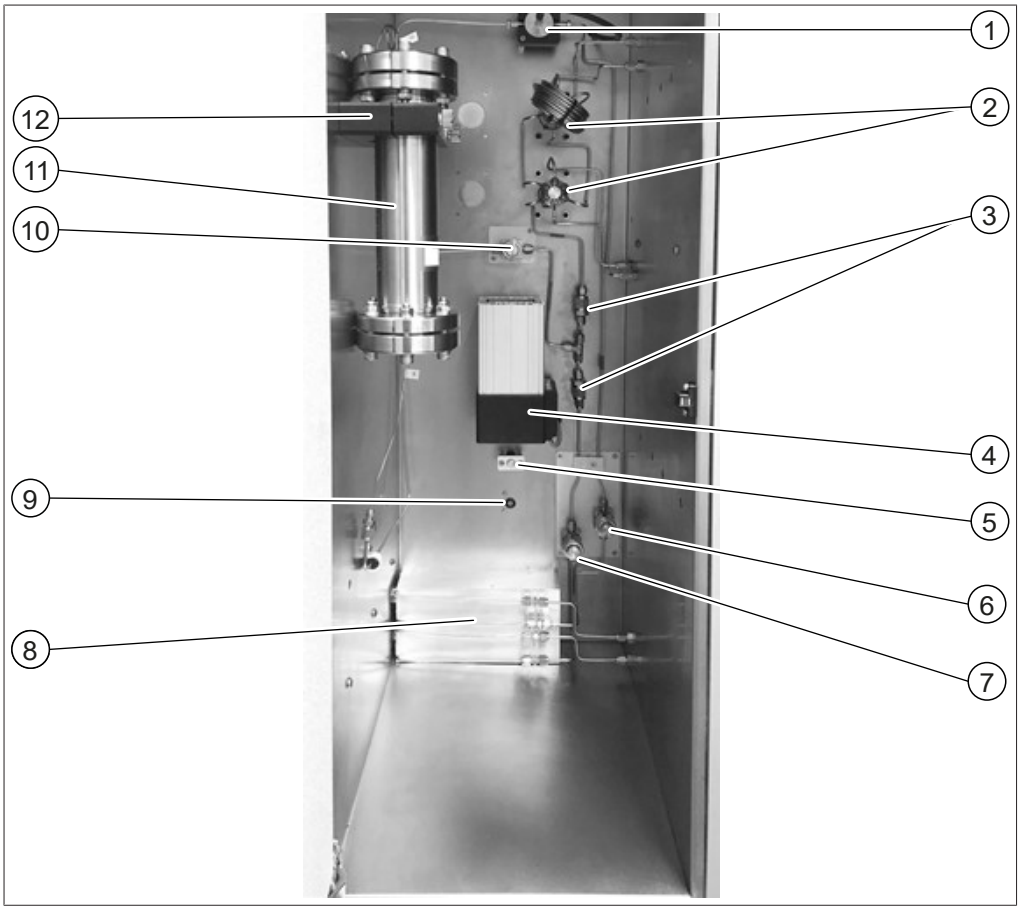
3.2 Configuration

3.2.1 Front view



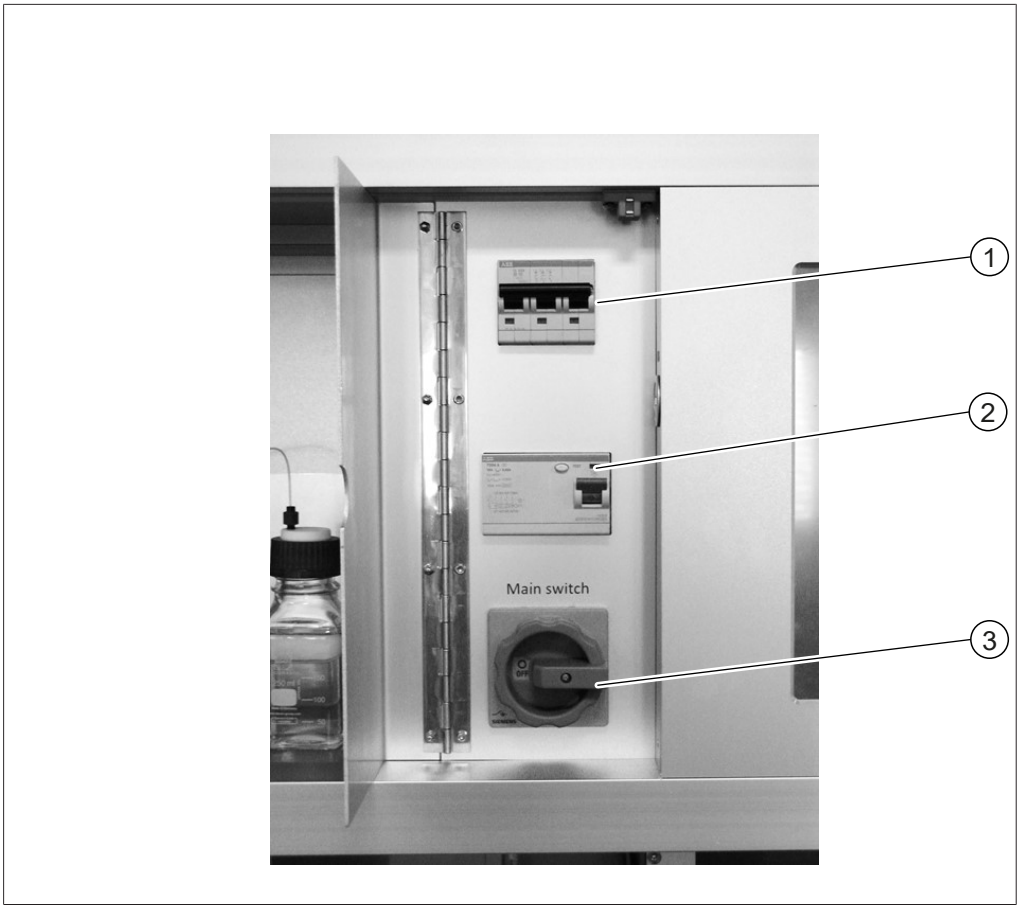
1	Injection syringe	2	Detector flap
3	Control panel cover	4	Touchscreen
5	CO_2 and modifier pump	6	Fraction bottles
7	Chiller	8	Column oven

Column oven

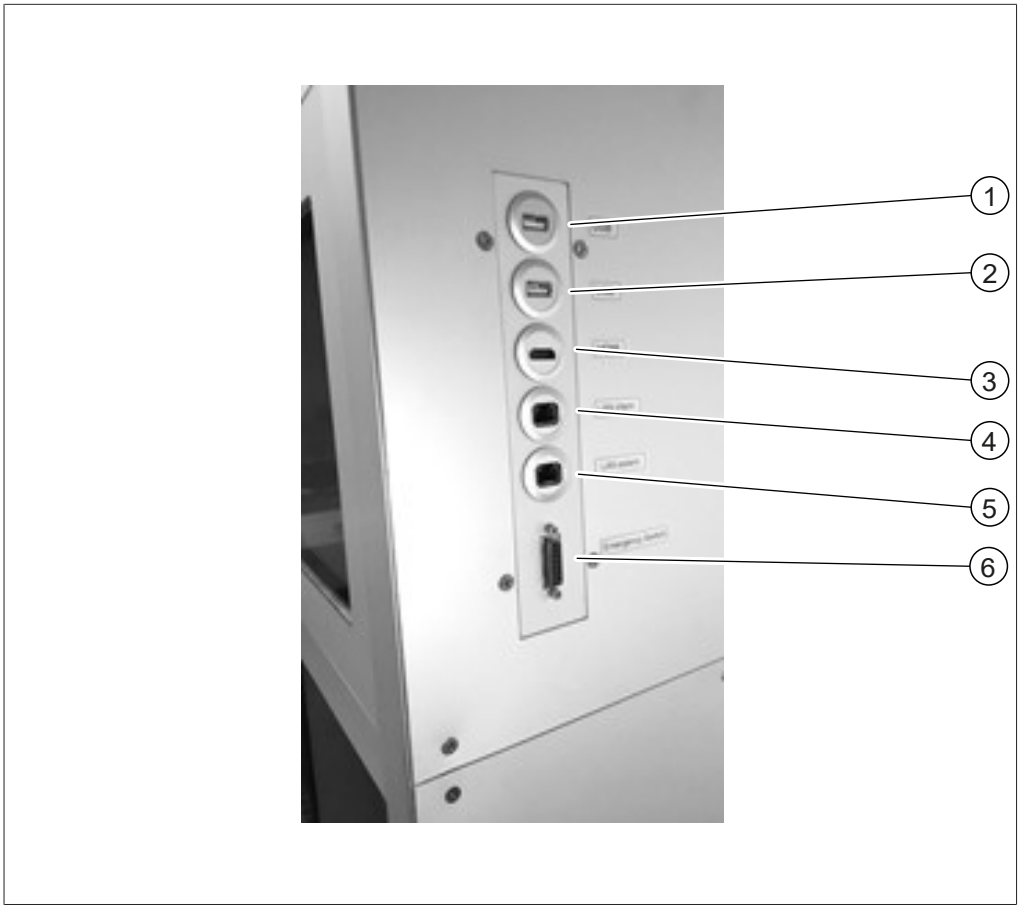


1	Flow cell with optic fibre cable	2	Injection valve
3	Check valves	4	Heating fan
5	Temperature sensor	6	Modifier filter
7	CO_2 filter	8	Heating module
9	Leak sensor	10	Mixed stream filter
11	Separation column	12	Column holder

3.2.2 Electric and electronic connections

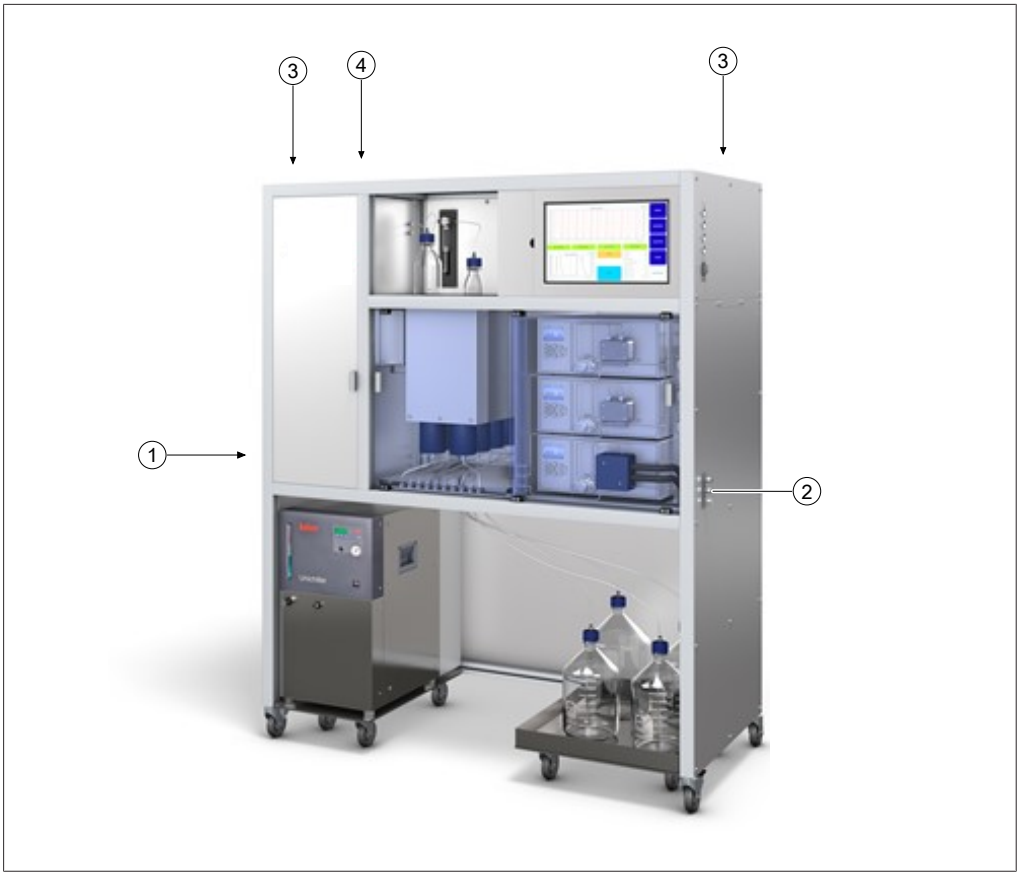


1 Fuses
2 FI-circuit breaker
3 Main switch



1	USB connection	2	USB connection
3	HDMI connection	4	LAN internal connection
5	LAN external connection	6	Emergency switch connection

3.2.3 CO_2- and coolant connections



1	Connection for CO_2 supply	2	Connections for modifier supply
3	Ventilation openings	4	Connection for power supply

3.2.4 Exhaust connections

There are three exhaust connections on this instrument:

- On the left side is the exhaust of the fractionation compartment.
- On the right side is the outlet of the CO_2 safety exhaust valve.
- On the rear side is the GLS CO_2 exhaust.

3.3 Scope of delivery

NOTE

The scope of delivery depends on the configuration of the purchase order.

Accessories are delivered as per the purchase order, order confirmation, and delivery note.

3.4 Technical data

3.4.1 Sepiatec SFC-660

Dimensions (W × D × H)	1,500 mm × 680 mm × 1,780 mm
Weight	320 kg
Connection voltage	Europe: 230 ± 10% VAC
	Asia: 220 ± 10% VAC
	USA: 120 ± 10% VAC
Frequency	50 / 60 Hz
Power consumption	Max. 7,500 W
Fuse	15 A
Over voltage category	II
IP Code	IP 20
Pollution degree	2
Minimum clearance on all sides	200 mm
Controller	15.6” touchscreen, 16:9 format
System	Integrated Windows 10
Control software	Prep SFC control software
Operating temperature range	15 – 25 °C
Operating pressure	Max. 300 bar
Injection valve	Electrically operated 6-port / 2-ways valve
Column selection valve	2 electrically operated 11-port / 10-ways valves (optional)
Back pressure regulator valve	Electromagnetic check valve
Fractionation valve	Electrically operated 11-port / 10-ways valve
Tubing	Stainless steel capillary tubes OD: 1/16‘‘ ID: 1.0 mm OD: 1/8” ID: 1/16” OD: 1/8” ID: 2.1 mm OD: 1/4” ID: 4.6 mm
Pump head temperature control	Chiller
Method development	On analytical columns (ID: 4 – 4.6 mm)
Column dimension	ID: 4 – 16 mm

Column length	Max. 250 mm
Column oven temperature	Ambient to 70 °C
Standard column capacity	2
Max column capacity	8 (optional)
Fraction collection number	1 – 8 pcs.
Volumes	unlimited
Pressure-less collection	Standard
Syringe size	5.0 ml Other volumes on request
Loop	2.5 ml
Stack injection	Standard
Certificate	CSA / CE

3.4.2 Ambient conditions

For indoor use only.

Max. altitude above sea level	2,000 m
Ambient and storage temperature	15 – 25 °C
Maximum relative humidity	45 – 75% not condensing

3.4.3 Solvents

NOTE
Only place solvent containers in the tray provided under the appliance. Lay supply lines from solvent cabinets or central supply lines firmly and avoid tripping hazards.

NOTE
Even low concentrations of strongly acidic or strongly basic additives can irreversibly damage sensitive stationary phases, which is why the compatibility must be clarified with the column manufacturer.

The solvent can be supplied via containers that are placed in the tray provided under the unit. Supply from containers from solvent cabinets or from supply lines is possible. In passive systems, care must be taken to ensure that the difference in height between the pump head and a lower suction point for the solvent is less than approx. 1m in order to ensure reliable suction.
If the suction point is elevated in relation to the pump head or if the supply is actively carried out via a pressure line, attention must be paid to the maximum inlet pressure of the pump (please see technical specifications of the pump).
Check that the following solvents are replenished in sufficient quantity for the desired operating time:

- Modifiers
- Solvents for flushing the pumps

Intended use	Solvent

Modifiers	• Methanol • Ethanol • 2-Propanol • Acetonitril
	• Formic acid • Acetic acid • Trifluoroacetic acid (TFA) • Ammonia solution (aqueous/alcoholic) • Diethylamine • Water
Flushing the pumps	• Methanol • Ethanol • 2-Propanol

Strongly acidic or basic additives should not exceed a concentration of 0.5% by mass in the modifier. In the case of water, a concentration of 5-10% in the modifier may be useful for very polar samples.

CO_2	Liquid
Organic modifier solvents	4 pcs. Only with the optional solvent selector valve.

3.4.4 Detectors

Wavelengths range	190 − 720 nm
Light source	Deuterium lamp, Wolfram lamp
Linearity	> 2,0 AU
Wavelength accuracy	0.5 nm
Number of diodes	256
Flow cell (standard)	3 mm / 2 µl / 300 bar (light path/ volume/ max. pressure)
Flow cell (optional)	10 mm / 10 µl / 300 bar
Flow cell (optional)	0.5 mm / 3 µl / 300 bar

3.4.5 Pumps

Number of pumps	3
Pump heads	Modifier pump: 150 ml Modifier/Add-on pump: 250 ml CO_2 pump: 500 ml
Flow rate range	0.1 − 150 ml/min
Flow rate accuracy	± 2% at 2 − 50% of the flow range with methanol (water 80/20 v/v)
Pressure	Max. 400 bar

Reproducibility	0.1%
Operating temperature	5 – 50 °C

3.4.6 Chiller

Dimensions (W x D x H)	420 mm x 490 mm x 580 mm
Weight	60 kg
Operating temperature range	-20 – 40°C Prep SFC 660 system: 0 – 5°C
Refrigeration machine	Air-cooled, CFC- and HCFC-free
Temperature stability at -10 °C	0,5 K
Circulation pump type	Immersion pump
Circulation pump max. delivery	29 l/min
Min. ambient temperature	5°C
Max. ambient temperature	40°C
Power supply	The power is supplied via the instrument. For further details on the power supply, please refer to the manufacturer's manual.

4 Transport and storage

4.1 Transport

NOTICE

Risk of breakage due to incorrect transportation

- ▶ Make sure that the instrument is fully dismantled.
- ▶ Pack all instrument components properly to prevent breakage. Use the original packaging whenever possible.
- ▶ Avoid sharp movements during transit.

- ▶ After transporting, check the instrument and all glass components for damage.
- ▶ Damage that has occurred in transit should be reported to the carrier.
- ▶ Keep packaging for future transportation.

4.2 Storage

- ▶ Make sure that the ambient conditions are complied with (see Chapter 3.4 "Technical data", page 20).
- ▶ Wherever possible, store the device in its original packaging.
- ▶ After storage, check the device, all glass components, seals and tubing for damage and replace if necessary.

4.3 Lifting the instrument

⚠ WARNING

Danger due to incorrect transportation

The possible consequences are crushing injuries, cuts and breakages.

- ▶ The instrument should be transported by two persons at the same time.
- ▶ Lift the instrument at the points indicated.

- ▶ Lift the instrument – this requires two persons each lifting at one of the points indicated on the bottom of the instrument.

5 Installation

5.1 Installation site

NOTE
Please never unpack the shipment unless expressly agreed.

NOTE
Make sure that the power supply can be disconnected at any time in an emergency.

A BUCHI service engineer or an authorized representative will unpack and check the consignment carefully to ensure that all modules and accessories are in proper condition. This has to be done prior to installation.

The installation site must meet the following requirements:

- Firm, level surface.
- Take into account the maximum product dimensions and weight. See Chapter 3.4 "Technical data", page 20
- Clearance on each side of the instrument must be at least 200 mm.
- Do not expose the instrument to any external thermal loads, such as direct solar radiation.
- Do not expose the instrument to increased electromagnetic emissions. Electromagnetic fields in the frequency range between 200 to 300 MHz can cause the instrument to operate incorrectly.
- Make sure that the installation site meets the requirements of the safety data sheets for all solvents and samples used.
- It is recommended to install the instrument in a fume hood that remove solvent vapours and gases directly from the proximity (minimum air exchange 200 m^3/ hour).
- The instrument is designed for installation to solid and load-bearing surface. The instrument can be connected to a venting system by the connection nozzle on the left side.
- The Exhaust line inner diameter for the CO_2 gas waste must be at least 10 mm.
- The instrument must be at least 200 mm away from adjacent walls or other instruments. The distance at the reverse side should be at least 100 mm. This ensures adequate ventilation and unrestricted access to the main switch and fuses.
- Please make an external chiller available to keep the CO_2 pump head at a constant temperature of about 10 °C independent of the ambient temperature. Required cooling capacity (Ethanol) 0.26 kW, connection: G1/4 inch or G1/8 inch.
- The power line must be free from electromagnetic interference (EMI) and grounded. The device should not be connected to a power supply system that is vulnerable to sudden changes in power demand. If significant fluctuations in voltage occur, a constant voltage transformer may be required.
- Liquid CO_2 supply, pressure 55 - 75 bar.
- 1/8 inch connector (Swagelok)
- A CO_2 alarm should be set up at the site to warn of high levels of CO_2 in the air.
- The emergency stop switch, which should be installed within the user’s accessibility and outside the fume hood. It is used to switch off the pumps, heating elements and CO_2 supply in the event of danger.
- For the operation of a mass spectrometer nitrogen supply must be available at the workplace (laboratory supply, nitrogen generator or cylinder).
- It is required to cool the pump head of the Prep SFC 660 CO_2 pump with an air-cooled chiller to a constant temperature of 5°C independent of the ambient temperature.

5.2 Before installation

NOTICE

Instrument damage due to switching it on too early.

Switching on the instrument too early after transportation can cause damage.

▶ Climatize the instrument after transportation.

5.3 Establishing electrical connections

NOTICE

Risk of instrument damage because of not suitable power supply cables.

Not suitable power supply cables can cause bad performance or an instrument damage

▶ Use only BUCHI power supply cables.

Precondition:

☑ The electrical installation is as specified on the type plate.

☑ The electrical installation is equipped with a proper grounding system.

☑ The electrical installation is equipped with suitable fuses and electrical safety features.

☑ The installation site is as specified in the technical date. See Chapter 3.4 "Technical data", page 20.

▶ Connect the mains plug to an own mains outlet socket.

6 Software

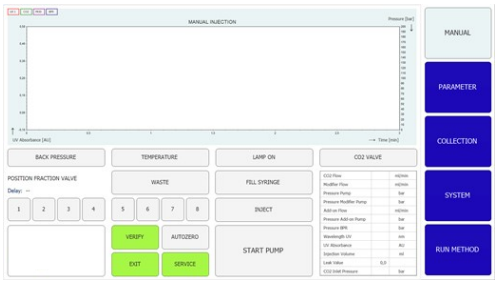
6.1 Introduction

The instrument is controlled by the Prep SFC control software. The software is operated by the integrated touchscreen. All the information needed for a correct separation run (separation parameters, system settings, fractionation conditions) is selected by means of this software.
All parameters are entered by touching the relevant display fields. This opens a dialog box to enter and edit the data. To confirm the data, tap the *[OK]* button. The dialog box closed automatically and the data is visible in the display field.
All display fields are shown in this operation manual. Depending on the configuration the display fields are shown in grey. This grey display fields are inactive or not included.

6.1.1 Starting the software

Precondition:
☑ Instrument is switched on.

▶ Tap the *[Prep SFC]* symbol on the touchscreen to start the software.
⇨ Start window opens.



▶ Tap the *[VERIFY]* button.
⇨ The system checks if all the functional elements are responding properly.

6.1.2 Entering values

Enter numbers

▶ Tap on an entry field.
⇨ The display shows a dialog box with a numeric input box.

▶ Enter the value.

▶ Tap the *[OK]* button to confirm.

⇨ The value is saved.

⇨ The dialog box closes.

▶ Tap the *[CANCEL]* button to leave the dialog box without changing the values.

⇨ The dialog box closes.

Enter names

▶ Tap on an entry field.

⇨ The display shows a dialog with an alphanumeric input box.

▶ Enter the value.

▶ Tap the *[OK]* button to confirm.

⇨ The value is saved.

⇨ The dialog box closes.

6.1.3 Exiting the software

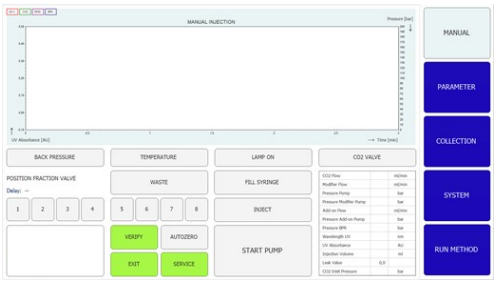
Precondition:

☑ Run is finished.

☑ Pumps are switched off.

▶ Tap the *[EXIT]* button to exit the software.

⇨ Prep SFC control software closes.



6.2 Layout

No.	Name	Description
1	*Chromatogram* display	To display the measured values.

No.	Name	Description
2	*MANUAL* tab	To start and stop a run manually. See MANUAL tab.
3	*PARAMETER* tab	To set, load and edit parameter. See PARAMETER tab.
4	*COLLECTION* tab	To configurate fractionation conditions, number of peaks to be collected and the fractionation method. See COLLECTION tab.
5	*SYSTEM* tab	To configurate system settings. The system parameters are set in this window. They do not depend on the method which has been loaded and can be adjusted individually for each run. See SYSTEM tab.
6	*RUN METHOD* tab	To start a loaded method and view online. This window is used to start the current run, which can be monitored online. See RUN METHOD tab.
7	*[VERIFY]* button	To check if all the functional elements are responding properly.
8	*[EXIT]* button	To exit the control software.
9	*[SERVICE]* button	To open the service settings of the system.
10	*[Windows]* button	To exit the control software. Opens the windows home screen.

6.3 MANUAL tab

No.	Name	Description
1	*MANUAL* tab	To start and stop a run manually.
2	*[FILL SYRINGE]* button	To rinse the tubing to the syringe.

No.	Name	Description
3	*[SERVICE]* button	To open the service settings of the system. Parameters, sensor signals and valve positions of the recycling module are displayed.
4	*[AUTO ZERO]* button	To set the detector signal to zero.
5	*[START PUMP]* button	To start the pump.
	[STOP PUMP] button	To stop the pump.
6	*[INJECTION]* button	To start the injection after starting the pump.
	[END INJECTION] button	To end injection.
7	CO_2 VALVE (grey)	Valve is closed
	CO_2 VALVE (green)	Valve is open
	CO_2 VALVE (red)	The CO_2 inlet pressure is too high, the system cannot be started.
	CO_2 VALVE (yellow/orange blink)	The CO_2 inlet pressure is too low. The pumps can be manual started for a flush. Starting an automated run is not possible
8	*[LAMP ON]* button	See RUN METHOD tab.
9	*[TEMPERATURE]* button	
10	*[BACK PRESSURE]* button	

6.3.1 Rinsing the tubing and syringe

Navigation path:

➔ *MANUAL*

▶ Tap the *[FILL SYRINGE]* button to rinse the tubing to the syringe.

⇨ The display shows a dialog box with a numeric input box.

▶ Enter the value.

▶ Tap the *[OK]* button to confirm.

⇨ The value is saved.

⇨ The dialog box closes.

6.3.2 Starting the pump

NOTE

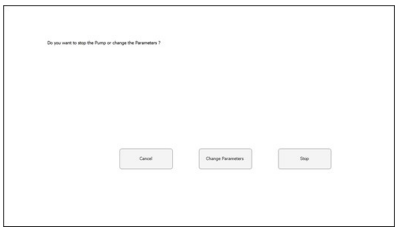
Before or during an isocratic method run in after starting the manual mode. By tapping the *[STOP PUMP]* button a new window opens and allows you to change the parameters or to stop the pump.

Navigation path:

➔ *MANUAL*

▶ Tap the *[START PUMP]* button.

⇨ A new window opens.



▶ Tap the *[CHANGE PARAMETERS]* button.

⇨ A new window opens to change the flow and the modifier percentage.

▶ Enter the value.

▶ Tap the *[OK]* button to confirm.

⇨ The value is saved.

⇨ The dialog box closes.

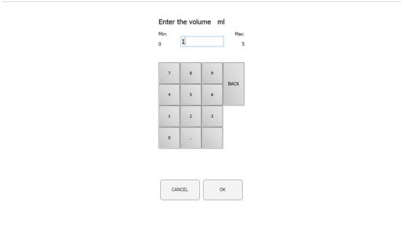
▶ Tap the *[INJECTION]* button.

▶ Enter the value.

▶ Tap the *[OK]* button to confirm.

⇨ The value is saved.

⇨ The dialog box closes.



▶ Tap the *[END INJECTION]* button to stop the injection.

▶ Tap the *[STOP PUMP]* to stop the pump.

NOTICE! An automatic run can be started, while the pump is still running but the injection has to be finished.

The chromatogram can then be called up in the *COLLECTION* menu. The fractionation parameters can be adjusted for automated further separations.

6.3.3 Setting detector signal to zero

Navigation path:

➔ *MANUAL* ➔ *[SERVICE]*

▶ Tap the *[AUTO ZERO]* button.

⇨ The detector signal is set to zero.

6.3.4 Changing CO_2 recycling module

Navigation path:

➔ *MANUAL* ➔ *[SERVICE]*

NOTE
Activated buttons have a green color.

▶ Tap the *[SERVICE]* button.

⇨ A new window opens. Parameters, sensor signals and valve positions of the recycling module are displayed.

▶ View parameter table.

▶ Tap the button to activate or deactivate.

▶ Tap the *[BACK TO MAIN VIEW]* button to close the service view window.

⇨ The window closes automatically.

6.4 PARAMETER tab

The parameter menu has two isocratic modes depending on the configuration:

- UV detector
- DAD detector (optional)

No.	Name	Description
1	*PARAMETER* tab	To set, load and edit parameter.
2	Equilibration	Column equilibration time in minutes.
3	Run Time	Run time for the separation in minutes.

No.	Name	Description
4	Flow	Flow rate in ml/min.
5	Modifier	Modifier concentration in percentage.
6	Add-On Flow	Flow rate in ml/min of the Add-On pump
7	Use Add-On	Determines if the third pump is used as Add-On pump (On) or works together with the modifier pump (Off).
8	Use GLS tandem mode	Combines two GLS for higher flow rates
9	Wavelength	Wavelength at which detection takes place in nm.
10	ELSD Gain	Number of ELSD gain.
11	Injection	Injection volume in ml.
12	No. of Injections	Number of injections to be carried out one after the other.
13	Modifier	Selection of pump inlet at the modifier pump.

6.4.1 Setting new methods

Navigation path:

➔ *PARAMETER*

▶ Tap the *[METHOD NAME]* button.

⇨ The display shows a dialog with an alphanumeric input box.

▶ Enter the value.

▶ Tap the *[OK]* button to confirm.

⇨ The value is saved.

⇨ The dialog box closes.

▶ Tap the COLUMN *[...]* button to enter a column designation.

⇨ A new window opens to choose a column from a list or add a new one.

▶ Tap the appropriate *[A]*, *[B]*, *[C]*, *[D]* button to select the solvent connection.

▶ Tap the MODIFIER *[...]* button to enter solvent designation.

⇨ A new window opens to choose a solvent from a list or add a new one.

▶ Set the parameters on the left part of the window.

Optional detector

▶ Tap the WAVELENGTH *[...]* button to set the number of wavelengths.

6.4.2 Setting gradient mode

Navigation path:

➔ *PARAMETER*

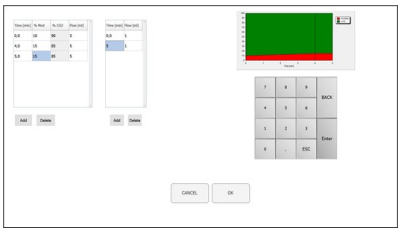
▶ Tap the *[GRADIENT]* button.

⇨ A new window opens displaying the gradient table.

▶ Tap into the gradient table.

⇨ A new window opens to edit the gradient table.

▶ Tap on the field on the control panel with a blue or white background to enter time, modifier and flow values.



▶ Enter the value.

▶ Tap the *[OK]* button to confirm.

⇨ The value is saved.

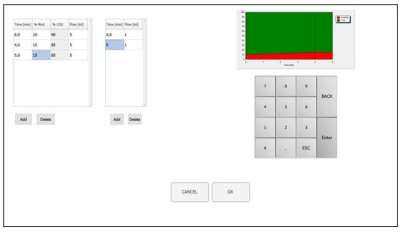
⇨ The dialog box closes.

NOTICE! The necessary volume of CO_2 is calculated automatically.

▶ Tap the *[ADD]* button to insert a row at the bottom of the table.

▶ Select a row and tap the *[DELETE]* button to remove a row.

NOTICE! The course of the gradient is shown in the upper left part of the gradient window.



▶ Tap the *[OK]* button to confirm and return to parameter menu.

▶ Tap the *[CANCEL]* button to discard the changes and return to parameter menu.

⇨ The programmed gradient is displayed.

NOTICE! In the isocratic mode and gradient mode, the values for equilibration time, wavelength and injection volume and modifier inlet are determined in this window.

▶ Tap the *[SAVE AS]* button to save the parameter in a different name.

⇨ The display shows a dialog box with an alphanumeric input box.

▶ Tap the *[SAVE]* button to set the parameter in the methods.

NOTICE! The number of injections to be carried out in sequence is not saved in the method and must be specified again whenever reloading the method or starting the system.

6.4.3 Loading saved methods

Navigation path:

➔ *PARAMETER*

▶ Tap the *[Load]* button to open saved methods.

⇨ A list of all methods appears.

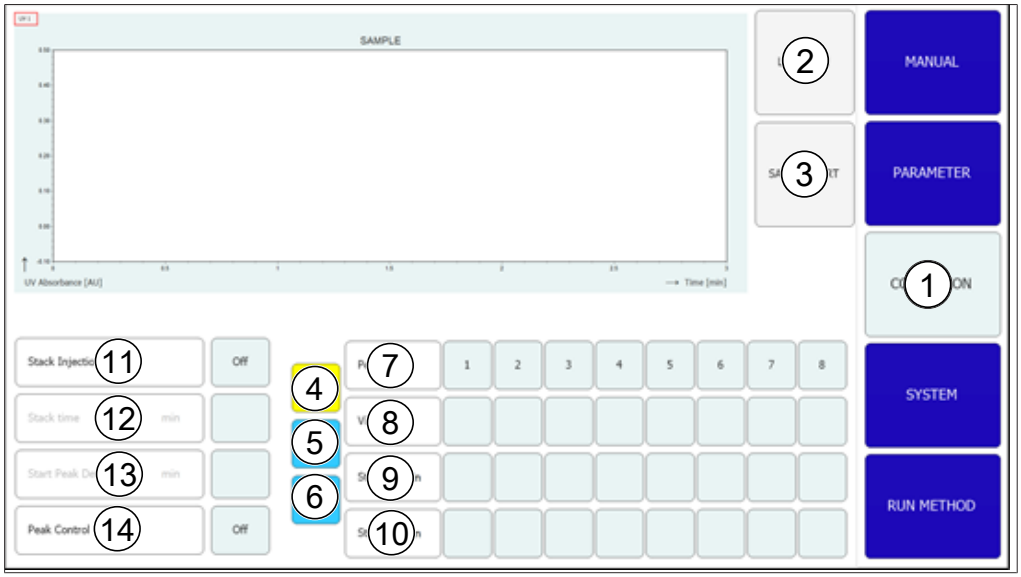
▶ Select the needed method.

▶ Tap the *[Load]* button to open the selected method.

6.4.4 Editing saved methods

A method that has been saved and opened can be processed as described under Chapter 6.4.1 "Setting new methods", page 34. This method must be saved again before starting it.

6.5 COLLECTION tab



No.	Name	Description
1	*COLLECTION* tab	To configurate fractionation conditions, number of peaks to be collected and the fractionation method.
2	*[Load Run]*button	To open completed runs.
3	*[Save Report]*button	Allows the operator to save a report in PDF format.
4	*[TIME]* button	Chooses the time based fractionation.
5	*[PEAK]* button	Chooses the peak based fractionation.
6	*[T/P]* button	Switches on the combined fractionation. In this mode, the peaks are searched inside the programmed intervals.
7	Peak No.	Number of peaks to be collected.
8	Vial	Fractionation bottle in which the fraction is collected.
9	Start min	Start of fractionation.
	Start AU	This value is the threshold for the start of collection.
10	Stop min	End of fractionation.
	Stop AU	End of peak detection.
11	Stack injection	To start and stop stack injection.
12	Stack time	The time between one injection and the next.
13	Start peak detect time	To starts the detection mode and control the peaks.
14	Peak control	To set parameters for multiple injections.

No.	Name	Description
	Trace	Optional detectors Determines which UV wavelength is used for the peak detection. This value is used to determine which chromatogram line is shown in the overview chart on the RUN menu.
	V	Intermediate part between the peaks, the Valley.

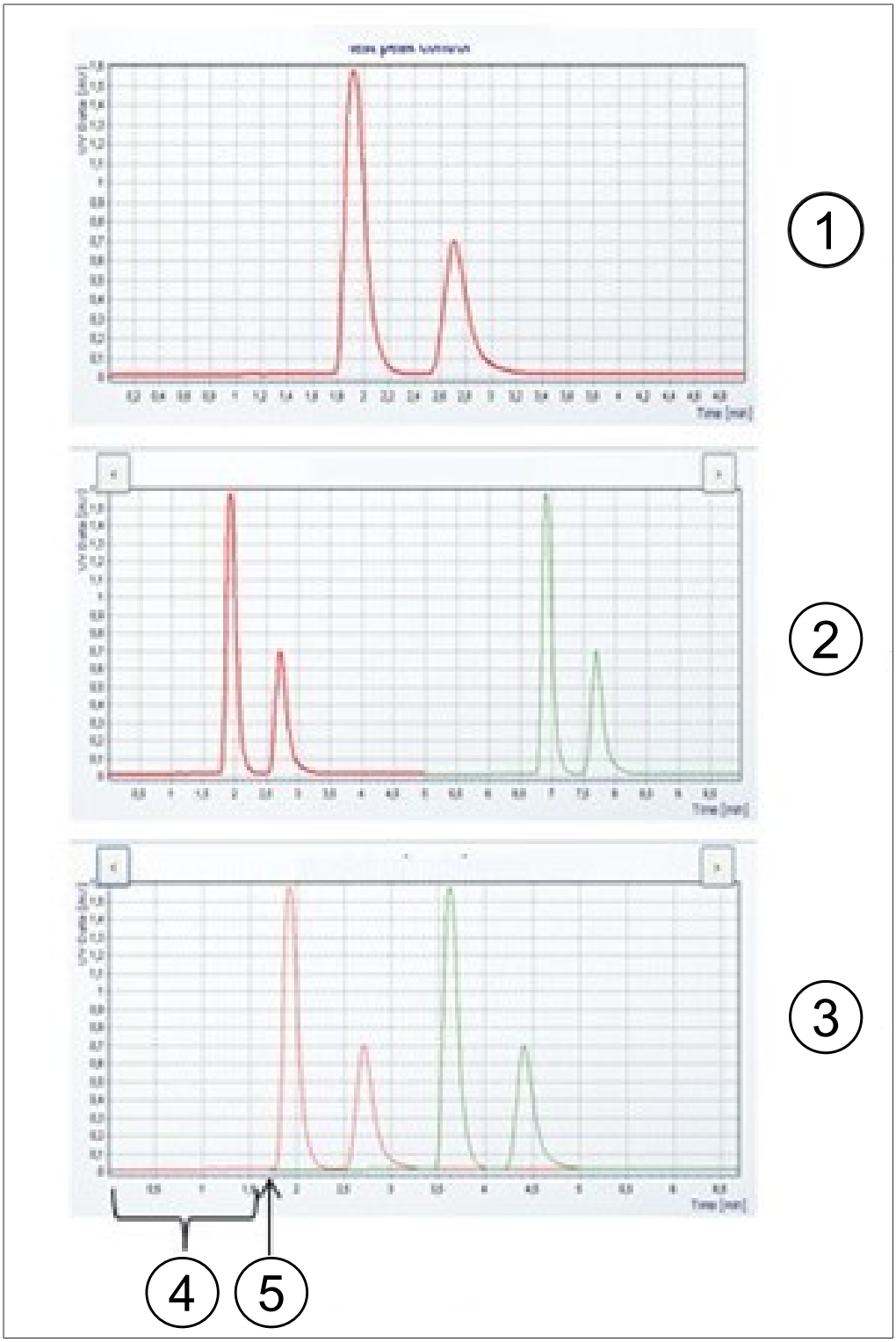
NOTE
Up to eight fractions in total can be collected. All parts of a run that are not to be collected will be captured in the waste container.

Peak control window

No.	Name	Description
1	Use Peak Control	Switch peak control on and off.
2	Controlled Peak	Number of the controlled peak.

No.	Name	Description
3	Automatic Shift ON	The collection window is automatically corrected.
	Automatic Shift OFF	The shift of the peak is just observed to be smaller than max. shift.
4	Max. Shift	The maximum shift difference is in seconds. The maximum allowed shift from the retention time observed during the first injection.
5	Min. Height	The minimum height is in percentage. Compare the minimum height the controlled peak with the fist injection peak. If the peak is smaller, the run is automatically stopped. The minimum height is in percentage. If the peak is smaller, the run is automatically stopped.

Injection Diagram



No.	Name	Description
1	First injection	Chromatogram of the first injection.
2	Multiple injections	Chromatogram of multiple injections.
3	Stack injections	Chromatogram of stack injection.
4	Stack time	The time between one injection and the next.
5	Start second injection	Time when the second injection takes place.

6.5.1 Setting peak control

When using a fractionation mode with peak detection the programmed number of peaks are compared with the actual detected number of peaks at the end of each injection. The run stops automatically if the peak detection is not successful.

The number of peaks to be collected is determined by touching the appropriate peak number. The operator can specify the fractionation start and stop time and the sample bottle in which the fractions are collected for the number selected. The start time of the following peak must be higher than the stop time of the preceding peak. The stop time of the final fraction must be lower than the total separation run time. Tap the *[Peak No.]* button if no peaks are to be collected. This deactivates the input fields.
The peak control feature allows the system to recognize automatically when a peak is outside the collection area and either stop the run or correct the collection window accordingly.
If a mode with peak detection is used, the number of detected peaks has to be the same as the number of programmed peaks, otherwise the run will stop. This test is performed even if the Peak Control is switched off.

Navigation path:

➔ COLLECTION ➔ *[Peak control]*

▶ Tap the *[OFF]* button to deactivate peak control.
▶ Tap the *[ON]* button to activate peak control.
⇨ A new window will open to set the control conditions.
▶ Set the control conditions.

▶ Tap the *[OK]* button to confirm.

6.5.2 Setting stack injection

The stack injection in isocratic mode allows time saving fractionation of large samples. The sample portions are injected after the elution of a peak.

Navigation path:

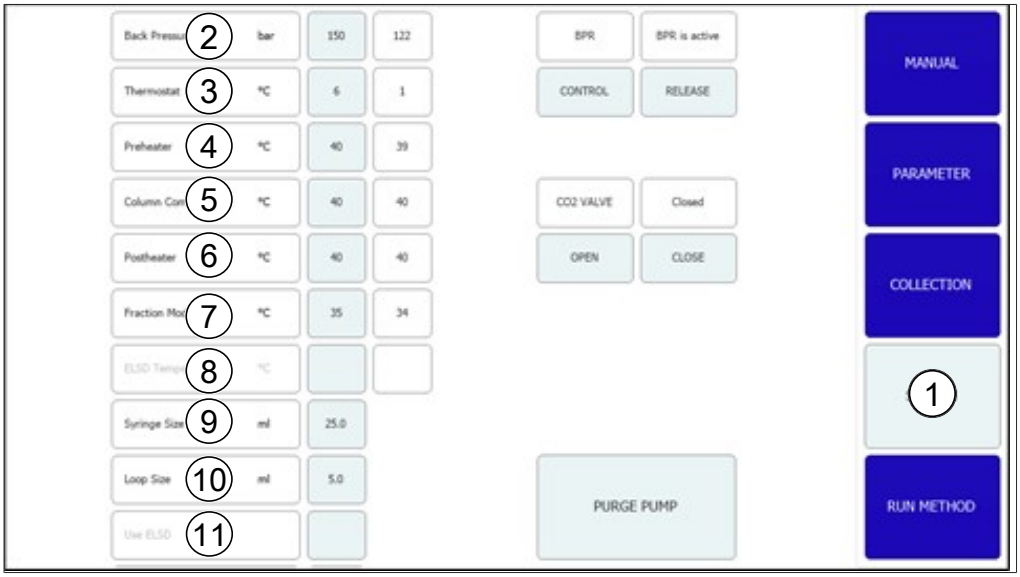
➔ COLLECTION

▶ Tap the *[Stack injection]* button to choose the stack injection option.

▶ Tap the *[Stack time]* button to enter the stack time.

⇨ The stack time determines when the next injection is done. This time has to be shorter than the runtime, but long enough to include all fractionations.

6.6 SYSTEM tab



No.	Name	Description
1	*SYSTEM* tab	To configurate system settings. The system parameters are set in this window. They do not depend on the method which has been loaded and can be adjusted individually for each run.
2	Back Pressure	Indicates the back pressure in the system in bar.
3	Thermostat	Temperature in the heat exchanger in °C.
4	Preheater	Temperature in the preheater in °C.

No.	Name	Description
5	Column Compartment	Temperature in the column oven in °C.
6	Postheater	Temperature at the gas-liquid-separators in °C.
7	Fraction Module	Temperature at the gas-liquid-separators in °C.
8	ELSD Temperature	Temperature for the ELSD in °C.
9	Syringe Size	Syringe volume in ml or µl .
10	Loop Size	Injection loop volume in ml.
11	Use ELSD	Switches the ELSD on or off.

6.6.1 Controlling the back pressure

The button controlling the back pressure regulator is in the top right-hand corner. The white field shows the status of the back pressure regulator.

Navigation path:

➔ *SYSTEM*

Precondition:
☑ Instrument is running. Displayed BPR status: BPR is active.

▶ Tap the *[RELEASE]* button.

⇨ The valve opens and release the pressure in the system. BPR status changes to BPR is open.

Precondition:
☑ Displayed BPR status: BPR is open.

▶ Tap the *[CONTROL]* button.

⇨ The valve close and activates the back pressure regulation. BPR status changes to BPR is active.

6.6.2 Controlling the CO_2 valve

The button controlling the CO_2 valve is located in the middle. The white field shows the status of the CO_2 valve.

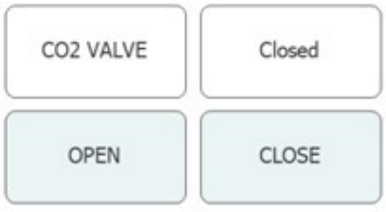
Navigation path:

➔ *SYSTEM*

Precondition:
☑ Displayed CO_2 VALVE status: Closed.

▶ Tap the *[OPEN]* button.

⇨ The valve opens. CO_2 VALVE status changes to Open.



Precondition:

☑ Displayed CO_2 VALVE status: Open.

▶ Tap the *[CLOSE]* button.

⇨ The valve closes. The CO_2 VALVE status changes to Closed.

6.6.3 Controlling the purge pump

Navigation path:

➔ *SYSTEM* ➔ *[Purge Pump]*

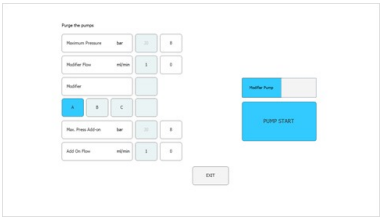
Precondition:

☑ Instrument is running.

☑ Displayed CO_2 VALVE status: Closed.

▶ Tap the *[Purge Modifier Pump]* or *[Purge Add-on Pump]* button.

⇨ A new window opens to set modifier pump.



▶ Tap the *[Open]* button to release the purge valve from the pumps.

NOTICE! The Purge Add-on Pump has just one inlet. The modifier inlet can not be selected.

NOTICE! If the pressure at the pump is higher than the maximum pressure for the purge pump, it will not start to purge.

▶ Select the modifier inlet and the flow rate.

▶ Tap the *[Pump Start]* button to start the process.

▶ Tap the *[Pump Stop]* button to close the purge valve at the pump and exit purge.

6.7 RUN METHOD tab

No.	Name	Description
1	Chromatogram	The current chromatogram is shown in the upper section of the window.
2	Title bar	The method name and sample name are displayed in the title bar. The injection number can be seen in the title bar.
3	Process overview graph	In the process overview graph in the bottom left-hand corner, chromatograms are displayed overlaid in case of multiple injections. In case of stack injection, the collected fractions are displayed overlaid.
4	Parameter status	The status of individual system parameters and method parameters during the run is shown in the table in the bottom right-hand area.
5	*[PEAK CONTROL]* button (grey)	Peak control is off.
	[PEAK CONTROL] button (green)	Peak control is on. The position of the peaks has not changed compared to the first injection.
	[PEAK CONTROL] button (orange)	Peak control is on. The peak being monitored has moved and the collection window was adjusted automatically.
	[PEAK CONTROL] button (red)	Peak control is on. The peak being monitored is outside the collection range and the run was stopped.

No.	Name	Description
6	*[LAMP OFF]* button (grey)	Lamp is off.
	[LAMP HEATING] button (yellow)	Lamp is heating up.
	[LAMP ON] button (green)	Lamp is on.
7	*[TEMPERATURE]* button (red)	The temperatures in all monitored parts (column department, preheating and fractionation module) have not been reached. The run cannot be started.
	[TEMPERATURE] button (yellow)	The column department and the preheating have reached the set temperature. The run can be started.
	[TEMPERATURE] button (green)	The fractionation module as well as the column department and the preheating have reached their temperature intervals. Injections can be done.
8	*[BACK PRESSURE]* button (orange)	The back pressure regulator valve is active. The pumps can be started. A run can also been started, but the equilibration time only starts counting after the back pressure has been reached.
	[BACK PRESSURE] button (green)	The back pressure set in the system has been reached. The run can be started.
	[BACK PRESSURE] button (yellow)	The back pressure set in the system has not been reached within 2 minutes after the start, or the pressure has been 10 bars lower than the set pressure for more than 30 sec during the run, or the pressure has dropped for more than 50 bar during the run. The pumps are switched off as there may be a leak.
9	*[CHANGE]* button	The button allows changing parameters during the run.

6.7.1 Before starting a run

Navigation path:

➔ *RUN METHOD*

▶ Tap the *[Start]* button.

⇨ The display shows a dialog with an alphanumeric input box.

▶ Enter sample name.

▶ Tap the *[OK]* button to confirm.

⇨ The value is saved.

⇨ The dialog box closes.

▶ Start a run. See Chapter 7.6 "Starting a run", page 56.

6.8 Changing during a run

NOTE
The minimum number of injections is the current injection or 1, if the equilibration is still running.

The number of injections can be changed for all injection types.

NOTE
With multiple injection, the injection volume and the run time for isocratic runs can be adjusted.

NOTE
A change of the injection volume is effective on the next not yet started aspiration of sample volume. If the start of the injection process is later than it should be for a new increased volume, the next injection will wait the necessary amount of time. This will not change the run time.

NOTE
The fractionation parameters can be adjusted starting with the second fractionation.

NOTE
When thresholds are changed the effect is simulated on the already recorded and shown in the graph to show the effect. The increasing tail slope (arrows to the right) will move the line to the left and vice versa.

NOTE
If collection intervals are changed the originally used values are shown in a less vibrant colour, the now used values will be shown in the known color.

NOTE
If runs are loaded on the collection menu, the original values for the collection are shown, if only one injection is shown, the values used in this injection are shown.

6.8.1 Change No. of Injections

Navigation path:

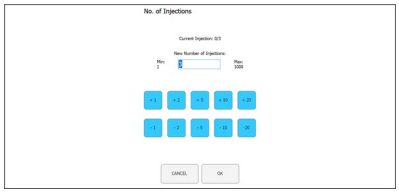
➔ *RUN METHOD* ➔ *[Change]*

NOTICE! This window close automatically after 10 seconds if no selection is made.

▶ Tap the *[No. of Injections]* button to change numbers of injection.

⇨ A new window opens.

▶ Tap a *[negative number]* button to set a minimum number.



▶ Tap the *[CANCEL]* button to close the window without any changes.

▶ Tap the *[OK]* button to update the version of the solvent amount.

⇨ The window closes automatically.

6.8.2 Change injection and run time parameter

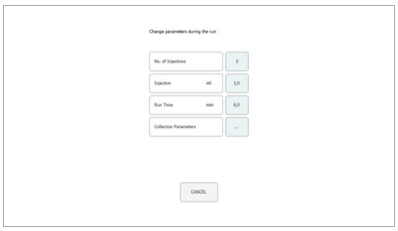
Navigation path:

➔ *RUN METHOD* ➔ *[Change]*

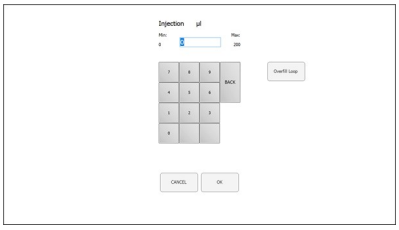
NOTICE! This window close automatically after 10 seconds if no selection is made.

▶ Tap the *[Injection]* or *[Run Time]* button to change parameters.

⇨ The display shows a dialog with a numeric input box.



▶ Enter the value.
▶ Tap the *[OK]* button to confirm.
⇨ The value is saved.
⇨ The dialog box closes.



NOTICE! The used maximum volume is not only determined by the injection loop or the size of the syringe but also by the time between injections.

▶ Tap the *[CANCEL]* button to close the window without any changes.
▶ Tap the *[OK]* button to update the version of the solvent amount.
⇨ The window closes automatically.

6.8.3 Change fraction parameter

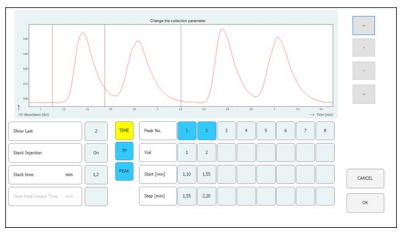
Navigation path:

➔ *RUN METHOD* ➔ *[Change]*

NOTICE! This window close automatically after 10 seconds if no selection is made.

▶ Tap the *[Collection Parameters]* button.
⇨ A new window opens.

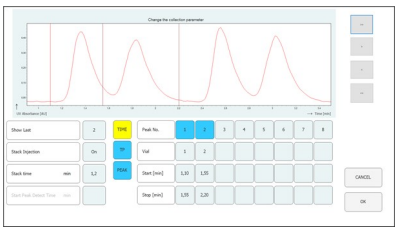
▶ Tap the *[Show Last]* button.
⇨ Allows to show and toggle between the last, the last two or the last three injections (if these are already recorded).



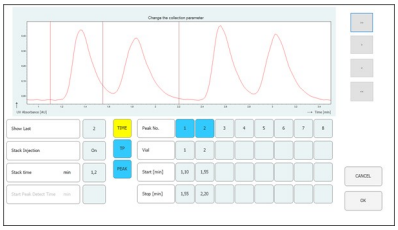
NOTICE! The method for the fractionation (time and/or peak based) as well as the Start peak detect time can not be changed after the run is started. These buttons are just for information.

▶ Tap one of the value button for Start or Stop to change the fraction parameter.

⇨ The value will be shown in blue.



▶ Tap the *[>]* button to change the value + 0.01.
▶ Tap the *[<]* button to change the value - 0.01.
▶ Tap the *[>>]* button to change the value + 0.05.
▶ Tap the *[<<]* button to change the value - 0.05.

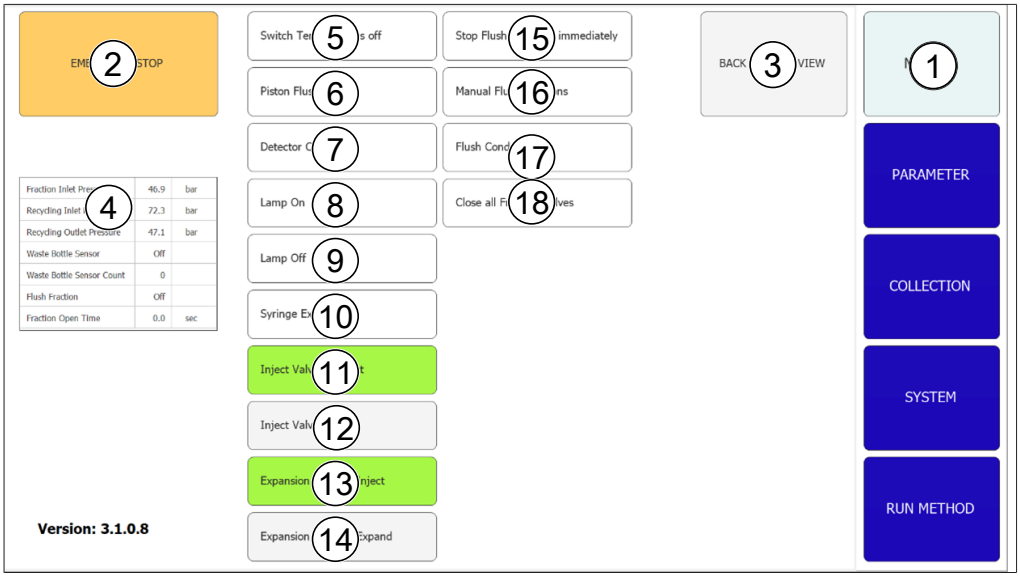


▶ Tap the *[OK]* button to make the changes effective.

6.9 CO_2 recycling module

Navigation path:

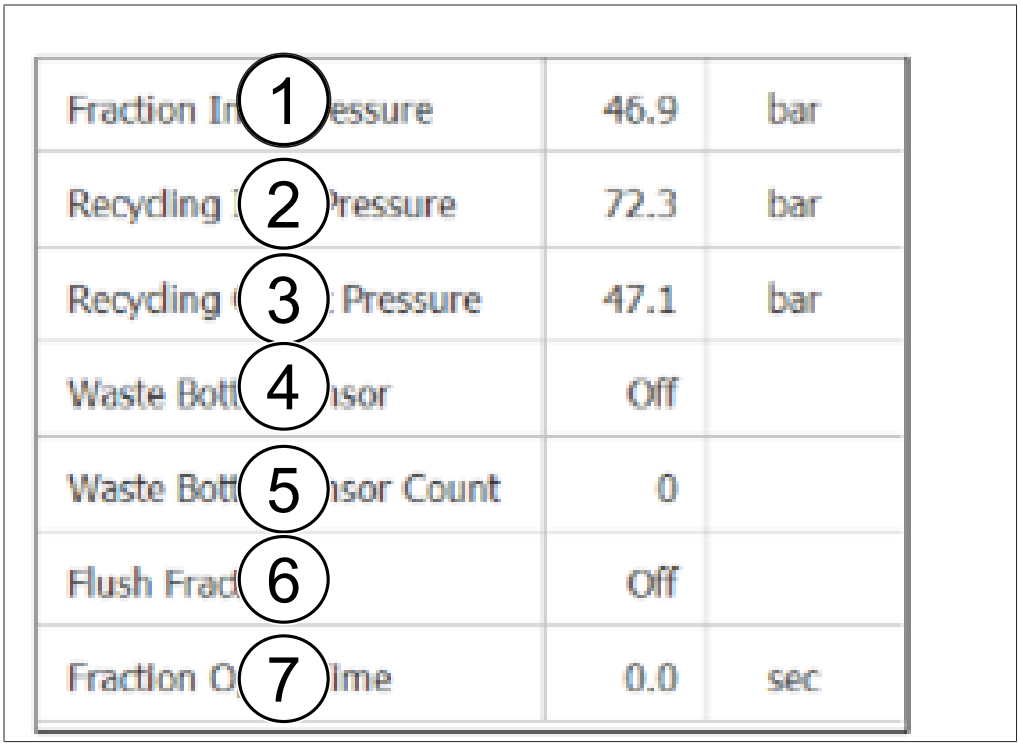
➔ *MANUAL* ➔ *[SERVICE]*



No.	Name	Description
1	*MANUAL* tab	To start and stop a run manually.
2	*[EMERGENCY STOP]* button	To perform the EMERGENCY STOP.
3	*[BACK TO MAIN VIEW]* button	To go back to the MANUAL tab view.

No.	Name	Description
4	Parameter table	To see several parameters about the recycling module.
5	*[Switch Temperature OFF]* button	To switch off all temperature controllers.
6	*[Piston Flush 10 sec]* button	To flush the piston for 10s.
7	*[Detector Calibration]* button	To manually start the calibration of the UV detector.
8	*[Lamp ON]* button	To switch on the lamp.
9	*[Lamp OFF]* button	To switch off the lamp.
10	*[Syringe Exchange]* button	To move the syringe pump into a position that allows the exchange of the glass syringe.
11	*[Inject Valve to Inject]* button	To switch the injection valve into the manual position.
12	*[Inject Valve to Load]* button	To switch the injection valve into the load position.
13	*[Expansion Valve to Inject]* button	To switch the expansion valve into the injection mode.
14	*[Expansion Valve to Expand]* button	To switch the expansion valve into the expansion mode.
15	*[Stop Flush Fraction immediately]* button	This button cancels the fractionation cycles of all 10 GLS channels.
16	*[Manual Flush Fractions]* button	This button starts the fractionation cycle of a GLS channel that can be selected in a new window.
17	*[Flush condensate]* button	This button simulates a liquid detection in the first cylinder, so that any residue condensate is flushed out.
18	*[Close all Fraction Valves]* button	This button closes all fractional valves for a service on the recycling module.
19	Software Version	Displays the current software version.

Parameter table



No.	Name	Description
1	Fraction Inlet Pressure	The pressure at the input of gas-liquid separators
2	Recycling Inlet Pressure	The pressure from the CO_2 supply that goes into the recycling module
3	Recycling Outlet Pressure	The pressure of the recycled CO_2
4	Waste Bottle Sensor	The sensor is set to detect liquid *[ON]* or not *[OFF]*
5	Waste Bottle Sensor Count	This value indicates how often the sensor has detected liquid. This value is an indication how stable the recycling and the gas-liquid separators are working. A low value means stable conditions. A high value means a lot of liquid is entrained. This value is set to zero at every start in the Run Method window.
6	Flush Fraction	This value indicates whether the fraction valve out is closed OFF or open ON.
7	Fraction Open Time	This value indicates that fractionation is in progress and how long the fraction valve in is still open.

Functional description

If the Recycling Inlet Pressure and Recycling Outlet Pressure are approximately equal, the CO_2 pump is supplied with recycled CO_2.

In the first cylinder of the recycling module dissolved modifier can condense. The liquid collects at the bottom of the cylinder, where a valve-sensor-valve combination is installed. When the sensor detects liquid, the upper valve is closed and the lower valve is opened to drain the condensate.
Due to the recycling module, the gas-liquid separators (GLS) are also under pressure. The fractionated liquid is fed to the recycling module via pressure-stable flexible lines. A valve-storage tube-valve combination is available for each of the ten GLS. When a GLS is switched active for fractionation in the software, the upper valve (fraction valve in) opens and the storage tube fills with the fractionated liquid. The software uses the flow rate of the modifier pump to calculate how long the valve can remain open and when it is full. It will close and open the lower valve (fraction valve out) to transfer the fractionated liquid to the collection container.

7 Operation

7.1 System start - up

▶ Switch on the external thermostat. The recommended temperature is 5 °C.

▶ Switch on the chiller.

▶ Push the ***ON/OFF-switch***.

⇨ This powers up the remaining hardware.

▶ Switch on the system with the ***main switch***.

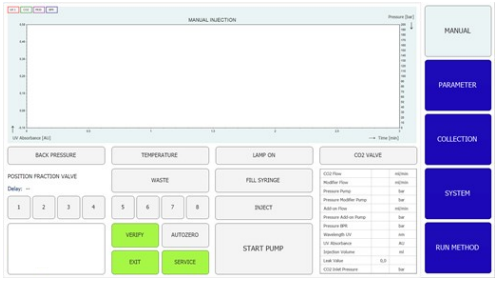
⇨ This also starts the computer and the touchscreen.

▶ Release the emergency switch button.

▶ Press the green button next to the emergency switch.

▶ Tap the *[Prep SFC]* symbol on the touchscreen to start the software.

⇨ Prep SFC control software window opens.



▶ Tap the *[Verify]* button to ensure that all modules are communicating correctly.

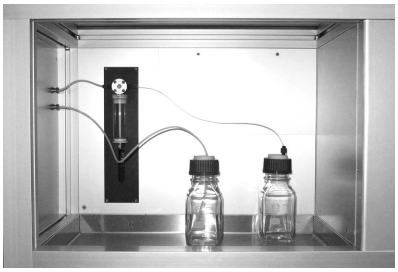
7.2 Positioning of the sample

NOTE

The capillary tubes should descend in a straight line and without tension.

▶ Place the sample in the sample tray.

▶ Feed the capillary tubes through the fittings of the holder until it reach the base of the sample bottle.

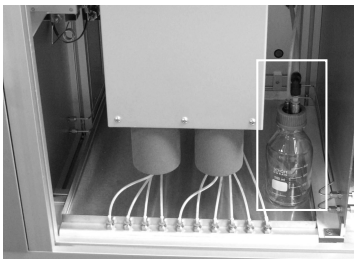


7.3 CO_2 exhaust air control (collection bottle)

In the compartment of the gas-liquid separators (GLS) there is a collection bottle on the right-hand side for solvent that has not been completely separated in the GLS. Normally, complete separation of liquid occurs in the GLS, but under certain circumstances and depending on the operating conditions (flow rate, solvent and temperature of the decompression heating module or temperature of the GLS), small amounts of liquid may be collected in the collection bottle.

▶ Open the collection bottle.
▶ Empty the collection bottle.
▶ Screw the bottle cap tightly onto the bottle.

NOTICE! Make sure the bottle cap is closed tight. Neither gases nor solvents should escape into the GLS compartment.



In the initial phase of a run:

▶ Check the level of the collection bottle from time to time to make sure that the GLS is working correctly.

7.4 Positioning the fractionation bottles

NOTE

Fraction bottles of any size can be placed in the safety tray under the GLS module.

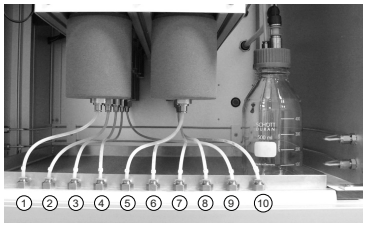
The number of the GLS corresponds to the outlet number of the fractionation valve.

NOTE

Depending on the selected flow rate, either up to 8 fractions (single mode, flow rate max. 180 ml/min) or up to 4 fractions (tandem mode, flow rate above 180 ml/min) can be collected. In tandem mode, GLS 1 and 2, 3 and 4, 5 and 6 as well as 7 and 8 are connected in parallel.

In tandem mode, the fraction tubes that belong together (e.g. GLS 1 and 2 for fraction 1) must be connected to a common fractionation bottle.

- ▶ Place the fraction bottles underneath the unit in a suitable collecting tray.
- ▶ Attach the appropriate capillary tubes of the gas-liquid-separators to the fraction bottle.
- ▶ Attach the tubes 9 and 10 to the waste container.
- ▶ Attach the tubes which are not used for fraction collection to the waste container.



7.5 Method selection

Navigation path:

➔ *PARAMETER*

- ▶ Tap the *[Load]* button to select the method you intend to use.
- ▶ Specify the number of injections which have to take place in sequence.
- ▶ Check if the correct column is in place at the selected column position.
- ▶ Tap the *COLLECTION* menu button.
- ▶ Define the number of fractions to be collected and the fractionation time-scale.
- ▶ Check the system settings in the system window.

See Software, for more detailed information about setting up methods, adjusting fractionation conditions and specifying system settings.

7.6 Starting a run

NOTE

Depending on the selected parameters, an equilibration time of several minutes is required to adjust to the set temperature profile. The shortest recommended time period is 5 minutes. If a stable temperature profile has not yet been established during this time, the equilibration time is automatically extended until either a stable temperature profile has been established or a maximum duration of 15 minutes has been reached. If a stable temperature profile has not been established after 15 minutes, the run is automatically stopped.

Navigation path:

➔ *PARAMETER*

Precondition:

☑ Make sure there is enough CO_2 and solvents to complete the separation run of the sample. The eluent consumption is highly dependent on the used method. To prevent damage to the pumps, it is necessary to avoid running them dry.

☑ Make sure the volume of the fraction bottles and the waste container are large enough for the whole separation to avoid overfilling.

☑ Make sure the collection bottle is empty and the collection bottle cap is tightly screwed.

☑ Make sure instrument is ready for a run.

☑ Make sure the correct method and fraction conditions are selected.

▶ Select the correct column, gradient and detection wavelength.

▶ Tap the *[Pump Start]* button.

⇨ The pumps are starting with the starting conditions of the selected gradient.

▶ Tap the *[Injection]* button.

⇨ The injection can be started when the set temperature conditions are reached.

7.7 Finishing a run

NOTE

After a run is finished, the pumps stop automatically and the CO_2 stop valve is closed automatically. The temperature controllers and the back pressure regulator will remain active.

▶ Tap the *[STOP]* button.

⇨ A new window opens.

▶ Tap the *[Cancel]* button.

⇨ The *RUN METHOD* menu opens but the run continues.

▶ Tap the *[Finish Injection]* button.

⇨ All injections that are already on the column (stack injection) will be finished. Then the system stops. The caption of the automatic start button changes to stopped.

▶ Tap the *[Immediately]* button.

⇨ The run stops immediately.

7.8 Shutting down the system

Navigation path:

➔ *MANUAL*

▶ Tap the *[EXIT]* button to close the Prep SFC control software.

▶ Shut down the windows computer.

▶ Switch off the systems with the ***main switch*** to shut down all the modules.

7.9 Using the emergency switch

▶ Press the ***emergency switch*** button to turn off the pumps, the detector and all temperature-controlled modules.

⇨ This closes the CO_2 stop valve that no more CO_2 can emerge.

⇨ The windows computer is still running. No data gets lost by the emergency switch off.

7.10 Monitoring the system functionality

The built-in electronics of the Prep SFC 660 system includes a “watchdog” functionality. This function is enabled at the initializing of the software. In case of a software or computer crash the watchdog triggers the emergency power switch cutting power to the pumps and the heating elements. The CO_2 inlet valve reverts to its closed state.

If the system is idle the watchdog time is 10 minutes, if the pumps are running this time is reduced to 2 minutes.

During normal shutdown of the software, the heating elements are switched off, but the emergency power circuit is not switched off.

8 Cleaning and servicing

NOTE

- ▶ Carry out only the service and cleaning operations described in this section.
- ▶ Do not carry out any servicing and cleaning operations that involve opening the housing.
- ▶ Use only genuine BUCHI spare parts in order to ensure correct operation and preserve the warranty.
- ▶ Carry out the service and cleaning operations described in this section to extend the lifetime of the instrument.

8.1 Cleaning the UV detector flow cell

Precondition:

☑ The detector baseline starts to get noisy. Possible sample residues are in the flow cell.

- ▶ Remove the columns.
- ▶ Replace the columns by a stainless steel capillary tube.
- ▶ Rinse the stainless steel capillary tube with CO_2 modifier mixture.

For further instructions please consult the manufacturer's manual for the Knauer Detector UVD 2.1S.

8.2 Removing the UV detector flow cell

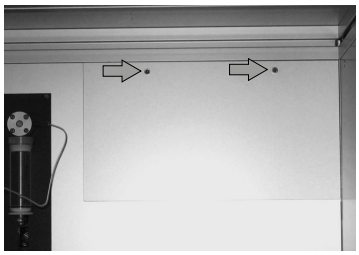
NOTE

The detector is installed in reverse order.

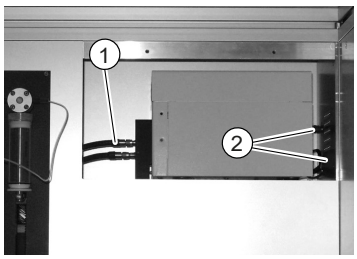
When installing the detector, please ensure that the detector is positioned as centrally as possible in the detector compartment so that sufficient air circulation is guaranteed.

To change the lamp of the detector, it is necessary to remove it from the instrument.

- ▶ Remove the two fixing screws.
- ▶ Take out the detector flap.



- ▶ Remove the screws from the detector housing of the optical fibers.
- ▶ Remove the power supply cable and the network cable.



- ▶ Remove the detector from the detector compartment.

8.3 Back pressure regulator valve

Please contact the BUCHI Customer Service if any problems occur with the back pressure regulator valve.

8.4 Fractionation valve

Please contact the BUCHI Customer Service if any problems occur with the fraction valve.

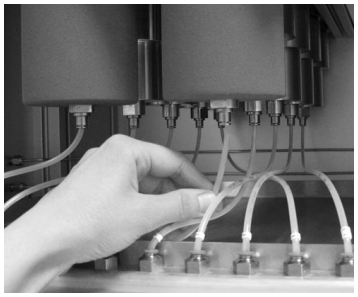
8.5 Gas-liquid-separators (GLS)

NOTE
To avoid contamination in the collected fractions, it is recommended to flush the gas-liquid separators with a CO_2 modifier mixture after each run.

- ▶ Purged each GLS for 30 seconds with a total flow of 150 ml/min and 30% modifier content.
- ⇨ If this is not sufficient clean them manually.
- ▶ Remove the GLS cups and clean them manually with an ultrasound bath using a suitable solvent.

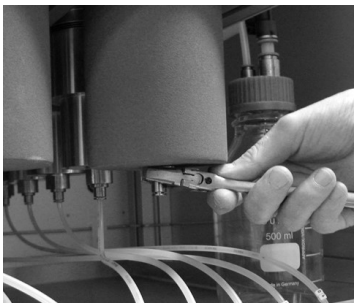
NOTE
Make sure not damage the insulation.

- ▶ Press the clamping ring of the hose connector upwards.
- ▶ Pull the hose out downwards without exerting.
- ⇨ Fraction hoses from the GLS cups are detached.



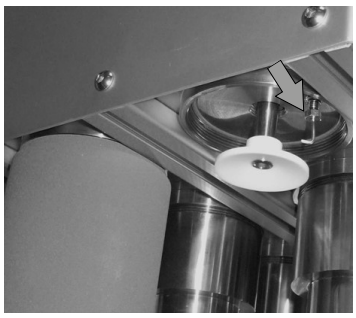
▶ Press the clamping ring of the hose connector upwards.
▶ Pull the hose out downwards without exerting.
⇨ Fraction hoses from the bulkhead connector are detached.

▶ Place a 16 mm wrench on the metal surfaces above the hose connector.
▶ Loosen the GLS cups clockwise.



▶ Unscrew the GLS cup with your hands.
▶ Remove the GLS cup. **CAUTION! A GLS cup weighs approx. 1,2 kg.**

▶ Clean the GLS and GLS cups with a suitable solvent.
▶ Clean the GLS and GLS cups with a lint-free cloth. **CAUTION! Do not use solvents that can attack polyurethane (e.g. acetone). Be careful not to bend the input capillary of the GLS.**



▶ Place the GLS cups.
▶ Screw the GLS cups counter-clockwise with your hands.
▶ Tight the GLS cups hand-tight with a 16 mm wrench.

9 Help with faults

9.1 Troubleshooting

Problem	Possible cause	Action
Computer does not start or the touch-screen remains dark	Mains plug is not connected	▶ Connect the mains plug
	Fuse or FI-circuit breaker are tripped	▶ Switch on fuse or FI switch
	Computer or touchscreen defective	▶ Contact BUCHI Customer Service
Indicator on the cooling unit does not light up although the instrument is switched on	Mains plug is not connected	▶ Connect the mains plug
	Cooling unit is defective	▶ Contact BUCHI Customer Service
Cooling unit does not start	Communication to the instrument is interrupted	▶ Check that the data cable is connected correctly
	Cooling unit is defective	▶ Contact BUCHI Customer Service
Cooling unit does not reach the specified temperature	Cooling water supply interrupted or restricted	▶ Check cooling water supply
Unusually high pressure	Pump defective	▶ Contact BUCHI Customer Service
	Malfunction of valves	
	Valve malfunction	
Low flow rate (no pressure)	Pump suction not operating (because of loose fittings on the suction capillary tube, for example)	▶ Tighten the fittings on the suction capillary, open the purge valve and pump at a high flow rate.
	Leaks in the system	▶ Tighten fittings
	Check valves dirty	▶ Clean or replace check valves. See pump manual.
CO_2 leakage at the pump head	Loose fittings	▶ Tighten fittings
	High-pressure pump seals are worn out	▶ Replace high-pressure pump seals. See pump manual.
Pressure fluctuation	Check valves are dirty or worn out	▶ Clean or replace check valves. See pump manual.
Set temperatures are not reached	Defective heating elements	▶ Contact BUCHI Customer Service
	Defective temperature control	

10 Taking out of service and disposal

10.1 Taking out of service

- Remove all solvents and coolants.
- Switch off the instrument and disconnect it from the mains power supply.
- Clean the instrument.
- Remove all tubing and communication cables from the device.

10.2 Disposal

The operator is responsible for proper disposal of the instrument.

- When disposing the equipment observe the local regulations and statutory requirements regarding waste disposal.
- When disposing, observe the disposal regulations of the materials used. Materials used see Chapter 3.4 "Technical data", page 20.

10.3 Returning the instrument

Before returning the instrument, contact the BÜCHI Labortechnik AG Service Department.
https://www.buchi.com/contact

11 Appendix

11.1 Spare parts and accessories

Use only genuine BUCHI consumables and spare parts in order to ensure correct, safe and reliable operation of the system.

NOTE

Any modifications of spare parts or assemblies are only allowed with the prior written permission of BUCHI.

We are represented by more than 100 distribution partners worldwide.
Find your local representative at:

www.buchi.com

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